

**The use of the blowpipe in the examination of minerals, ores,  
furnace-products, and other metallic combinations / By Professor Plattner  
... Translated from the German, with emendations, by Dr. Seridan Muspratt  
... With a preface by Professor Liebig.**

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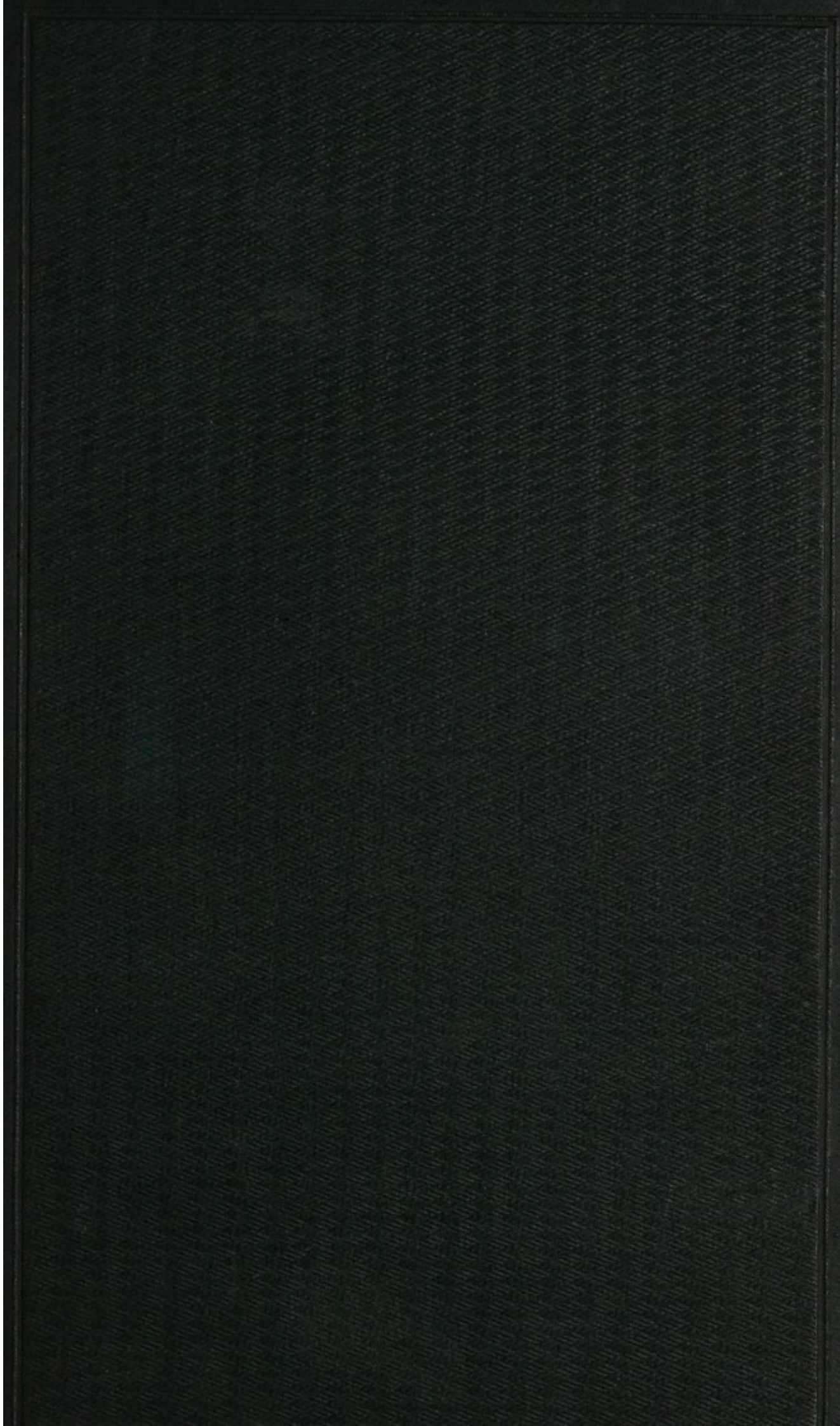
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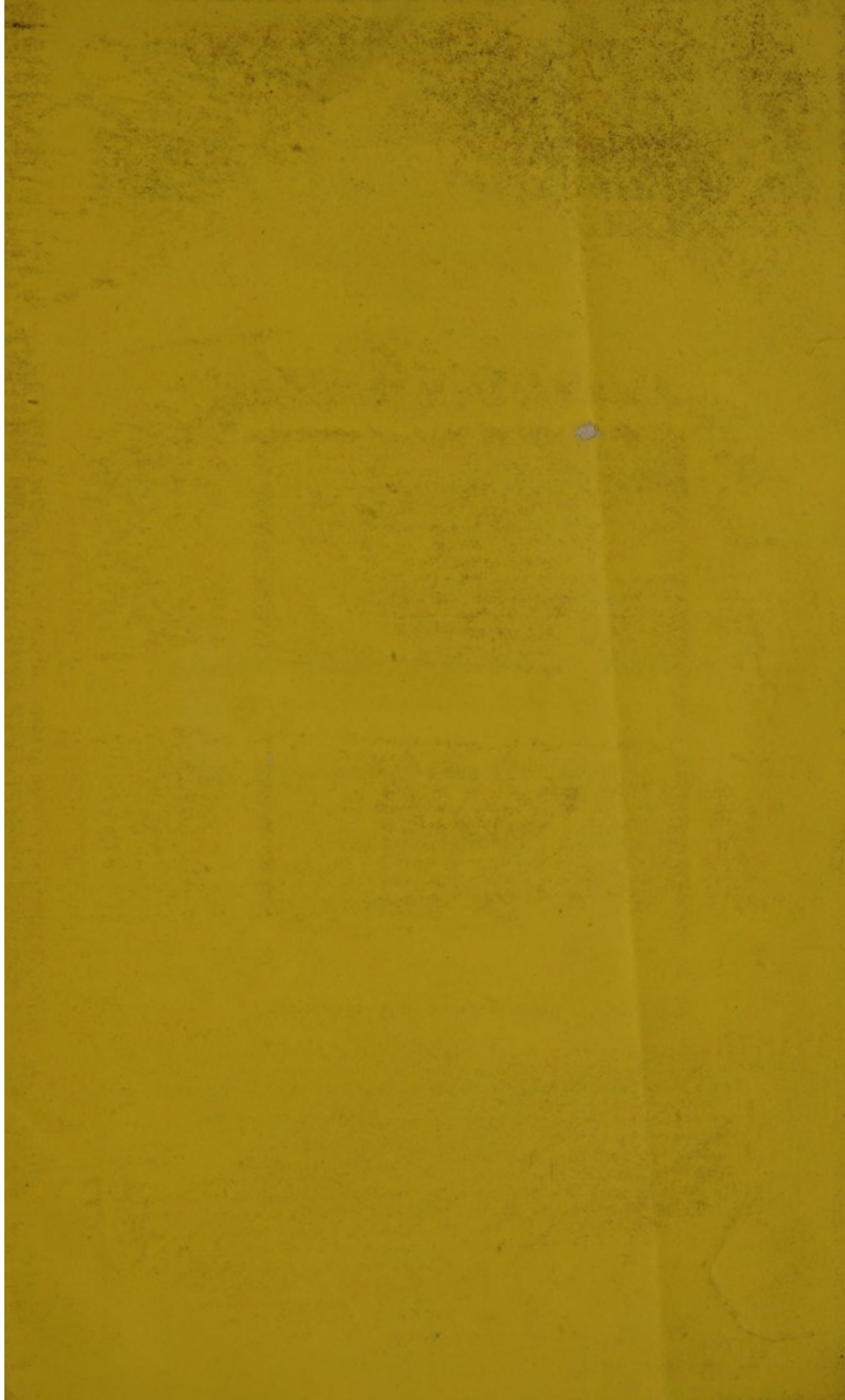


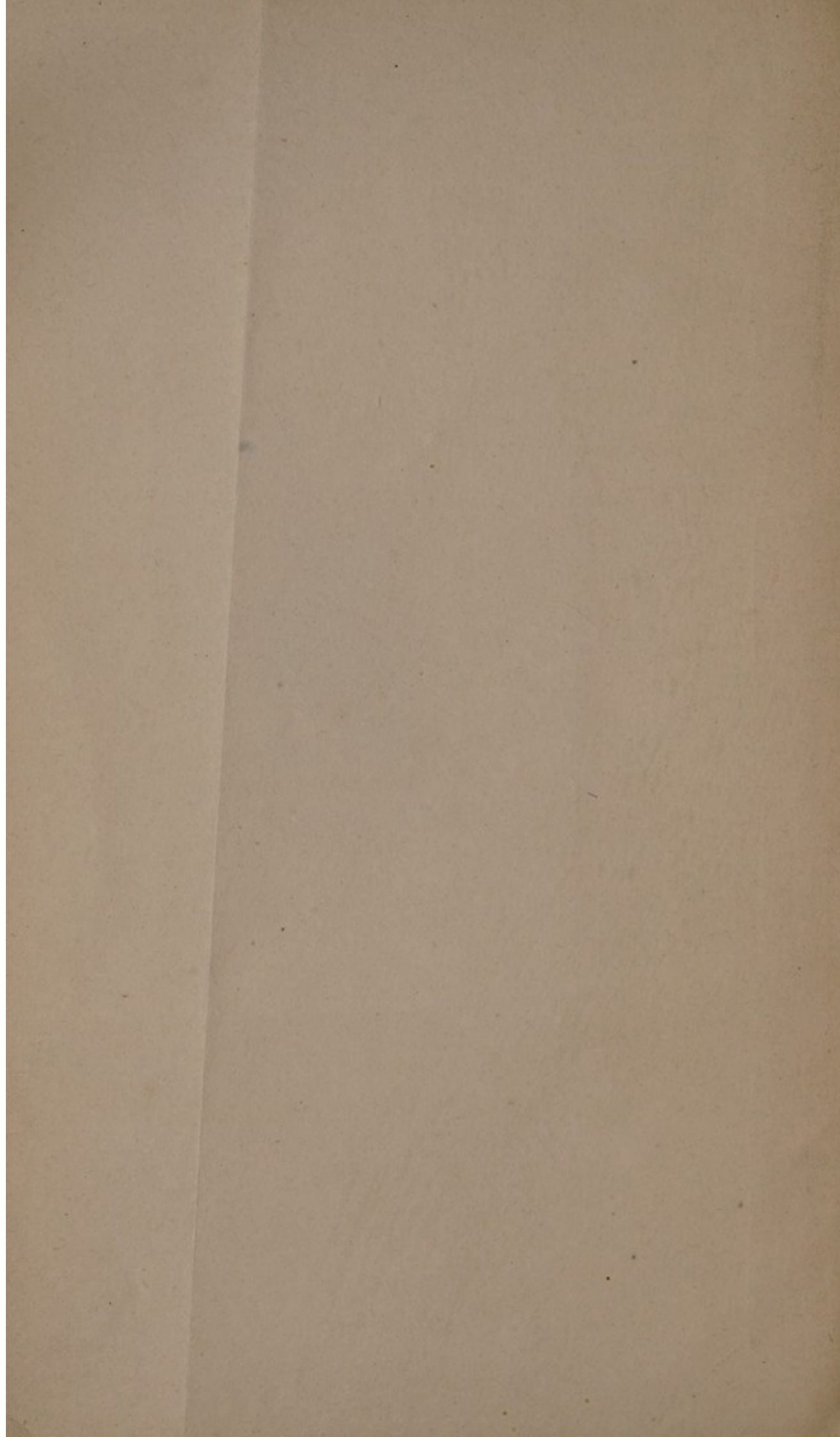


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*Hugh Lee Pattinson.*







THE  
USE OF THE BLOWPIPE,  
IN THE EXAMINATION  
OF  
MINERALS, ORES, FURNACE-PRODUCTS,  
AND OTHER METALLIC COMBINATIONS.

BY  
PROFESSOR CHARLES FREDERICK PLATTNER,  
*Assay-Master at the Royal Freyberg Smelting-Works.*

TRANSLATED FROM THE GERMAN,  
WITH NOTES,  
BY JAMES SHERIDAN MUSPRATT, PH.D.,  
*Of the University College, and Giessen Laboratories.*

WITH A PREFACE BY PROFESSOR LIEBIG.

ILLUSTRATED BY NUMEROUS DIAGRAMS.

"Segnius irritant animos demissa per aurem,  
Quam quæ sunt oculis subjecta fidelibus."

*Horace.*

LONDON:  
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MITCHELL, HEATON, AND MITCHELL, PRINTERS, LIVERPOOL.

# PREFACE

## BY PROFESSOR LIEBIG.

---

DR. SHERIDAN MUSPRATT'S translation of Plattner's excellent Treatise upon the Use and Application of the Blowpipe, has been executed with as much fidelity as ability, and I consider that its publication in England will be of essential service.

This instrument is of the highest advantage to the Chemist, Geologist, and Mineralogist, as a means of ascertaining, with the greatest accuracy, in a few minutes, all the constituents of a Mineral.

M. Plattner's work is the simplest and best adapted for this purpose, as, besides the methods of Gahn, Berzelius, and Gustavus Rose, it embraces the valuable results of his own practical experience.

The translated edition is further enhanced by Dr. Muspratt's annotations.

DR. JUSTUS LIEBIG.

GIESSEN, 23rd March, 1844.



REVIEW  
BY PROFESSOR J. H. JONES

The reviewer's knowledge of the  
author's work in the field of the  
history of the Church in the  
Middle Ages is such that he is  
able to say that the book is  
a valuable addition to the  
literature of the subject.

The reviewer is of the opinion  
that the book is a most  
valuable addition to the  
literature of the subject, and  
that it is a most  
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valuable addition to the  
literature of the subject.

THE REVIEWER

TO  
P. J. MURPHY, Esq. M.D.,  
A.B., L.R.C.S.I., &c.

Liverpool.

To whom can I with more propriety dedicate this Work on Chemistry, than to him whose encomiums on the noble and useful Science, first induced me to devote myself entirely to its study.

You first awoke in me a laudable desire of fame, and the hope of benefiting others by extending the limits of an almost boundless science: this has, if possible, increased the gratitude and affection of,

Your attached Friend,

THE TRANSLATOR.

Giessen, August, 1844.

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1888

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## TRANSLATOR'S PREFACE.

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PLATTNER'S "Probirkunst mit dem Löthrohre" has a very high reputation on the continent. It has been greatly and deservedly eulogized by four of the most eminent chemists of the day,—Liebig, Berzelius, Rose, and Kane, and has been translated into French by Dr. Sobrero. The idea of rendering it into English, for the use of chemical students unacquainted with German, originated in a suggestion of the translator's friend, Mr. William Bastick, who found it extremely useful, when manipulating in the Giessen Laboratory.

Professor Liebig, in his "Chemistry of Agriculture and Physiology," having so clearly established the necessity of a scientific examination of the mineral substances on which the fertility of soils depends; and such analyses being materially facilitated by the Blowpipe, when employed according to the method of Plattner; it is evident that his work should occupy a place on the shelves of every well-informed agriculturist, in company with that of the renowned German chemist.

The Mineralogist or Metallurgist, will also find his labours abridged, and his results accomplished with more accuracy, by the aid of this instrument. But the value of Plattner's work is stamped by the approbation of Liebig,—a name which alone is sufficient to justify a translation.

In the original, the engravings are placed at the end of the work, while here, they occur in the same page with the explanatory text, the advantages of which method are obvious.

Most of the symbols have been altered, with a view to suit the more recent analyses; and when a German technical is retained, it is invariably explained in the appended notes. Portions of the work have been condensed; some have undergone an improved arrangement, while others, deemed immaterial, have been omitted.

The translator has been considerably aided in the execution of the work, by "Phillips' Mineralogy," and the "Handwörterbuch des Chemischen Theils der Mineralogie" of Rammelsberg. His labours have been also occasionally lightened, by the assistance of his brother Frederick, and of his esteemed friend, Professor Hanstein. To convey to the reader the author's meaning, in language concise and clear, has been a principal aim; and in attaining this object, the translator has to acknowledge his obligations to his kind friend Dr. Thomas O'Dwyer.

*Seaforth Hall,*

*December, 1844.*



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SECTION I

## I. THE BLOWPIPE.

As Berzelius has given a full and complete history and description of the Blowpipe, in his "*Anwendung des Lothrohrs in der Chemie und Mineralogie*,"\* they need not be here detailed; the work, as I imagine, being in the hands of every person who makes practical use of this instrument.

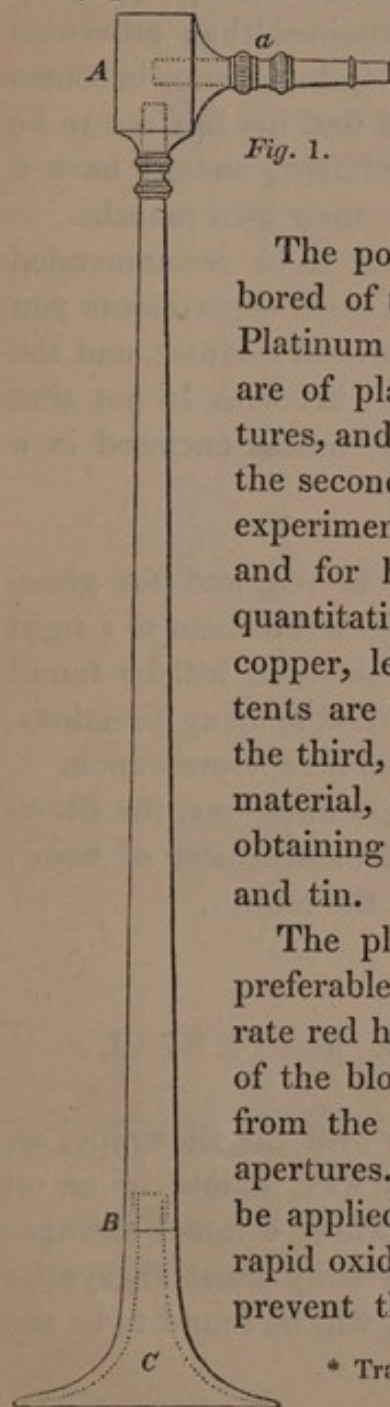


Fig. 1.

My Blowpipe, which I use in all my analyses, is of brass, and has been described by Gahn, and approved of by Berzelius. (Fig. 1.)

The points of this Blowpipe are turned and bored of three different sizes, and are partly of Platinum and partly of Brass. The first, which are of platinum, contain the narrowest apertures, and are employed for qualitative analyses; the second, of brass, I use for such qualitative experiments as require a strong oxidation flame, and for heating silver, gold, and copper, in quantitative assaying, also for the roasting of copper, lead, and tin ores, whose metallic contents are to be determined with precision; and the third, which are manufactured of the same material, contain the widest bore, and are for obtaining the quantitative proportions of lead and tin.

The platinum points, however, are always preferable to those of brass, because by a moderate red heat used on charcoal before the flame of the blow-pipe, they are more easily cleaned from the sooty particles which obstruct their apertures. This method of cleansing cannot be applied to brass points, on account of the rapid oxidation of the metallic composition; to prevent this, the operator must adapt to the

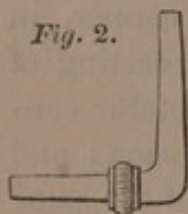
\* Translated by J. G. Children, Esq.



opening a sharp-pointed fragment of horn, or a small needle ground to one-half of its length; by this means the aperture through which the air passes is purified. The black matter adhering to the exterior of the tube can be easily displaced with a small wooden or leaden file, upon which is placed a quantity of fine moistened bone ash.

To defend the lips from injury, I fix to my Blowpipe a mouth-piece of horn, (c). If, during the act of blowing strongly, this mouth-piece be pressed to the lips partially open, a much greater blast can be obtained than otherwise could be without its aid, and when an operator once becomes accustomed to its management, he will find his lips not to be fatigued. All who employ this instrument, should have a mouth-piece manufactured, suitable to their own mouths.

A Blowpipe entirely of silver is not to be recommended for quantitative researches, because at such experiments you must often blow uninterruptedly for several minutes, and the silver being a good conductor of heat, becomes so hot that it cannot be held between the fingers, unless enclosed in a non-conducting substance.



*Fig. 2.*

When the Blowpipe is required for glass-blowing, Berzelius employs a tube bent to a right angle; (see *Fig. 2.*) This tube will be found convenient, as it can be turned in many directions, suitable to the materials to be operated upon.

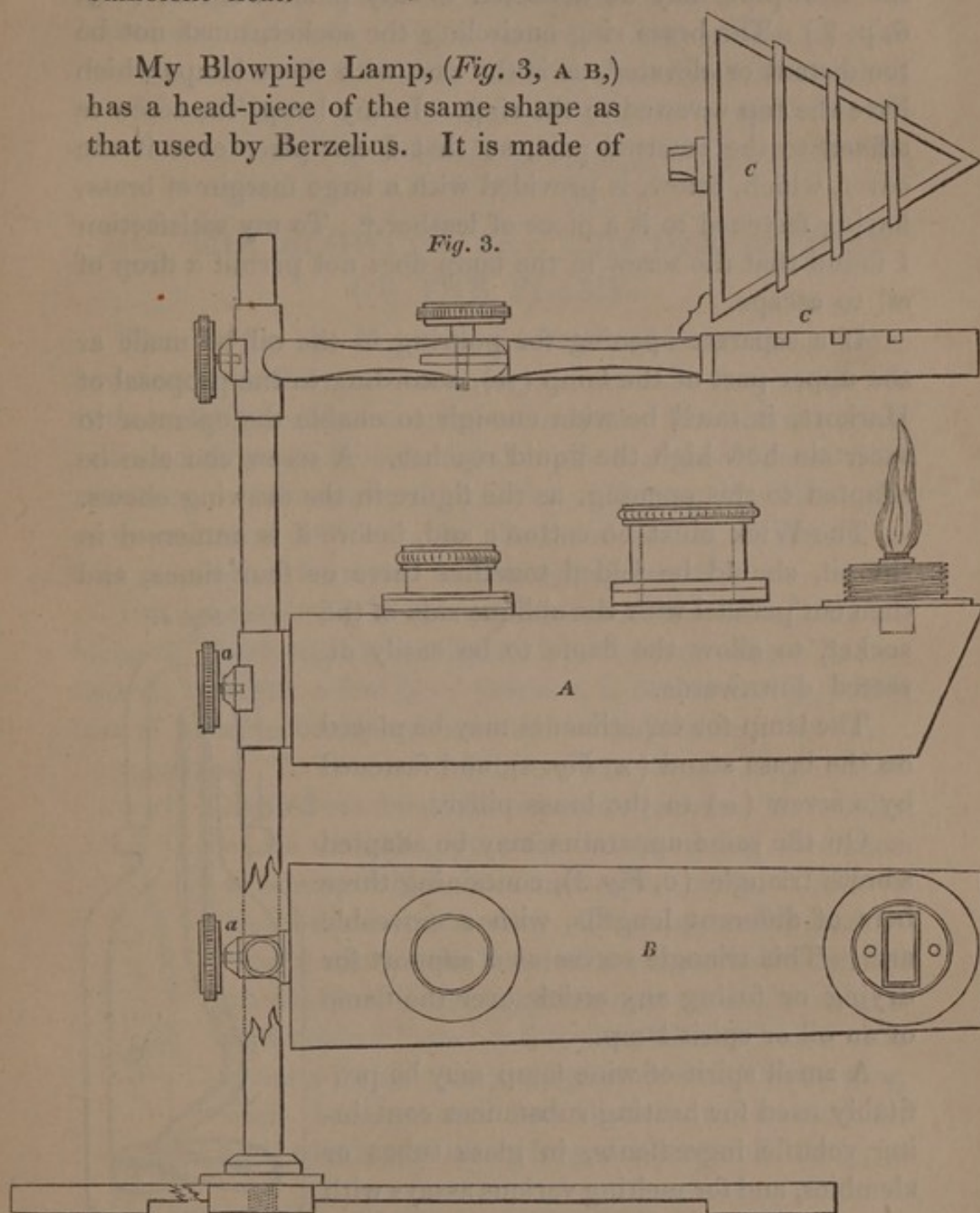
When the hands are required for examinations, the Blowpipe should have affixed a very broad mouth-piece of bone; by this means it can be held safely in the mouth.

## II. THE COMBUSTIBLE MATERIAL.

In many qualitative examinations with the Blowpipe, an operator may employ the flame from wax, tallow, or an oil lamp; but in quantitative analyses, where a much stronger heat is necessary, the flame of an oil lamp must always be used. Olive Oil, or refined Rape Oil, will be found to be the

best for such lamps. Unrefined rape oil must never be employed, as it produces much smoke, and does not afford sufficient heat.

My Blowpipe Lamp, (*Fig. 3*, A B,) has a head-piece of the same shape as that used by Berzelius. It is made of



iron plate, tinned over, and varnished black. As a greater proportion of test material is used in quantitative than in



qualitative analyses, and also a stronger heat required, the socket in my lamp is wider than usual, so that the flame of the Blowpipe may be deflected to any point desired, (*Fig. 6, p. 7.*) The brass ring encircling the socket, must not be too distant or elevated, as is the case with those lamps which have the top screwed to the ring. In my lamp, the screw is affixed to the external part, so that I can place over it the cover, which, below, is provided with a large margin of brass, having fastened to it a piece of leather.\* To my satisfaction I found that the screw in the lamp does not permit a drop of oil to escape.

If a separate opening for pouring in the oil be made at the upper part of the lamp (B), according to the proposal of Harkort, it must be wide enough to enable the operator to ascertain how high the liquid reaches. A screw can also be adapted to this opening, as the figure in the drawing shews.

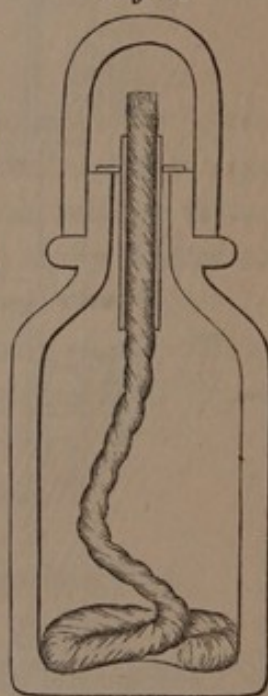
The Wick must be cotton; and, before it is immersed in the oil, should be folded together three or four times, and then cut parallel with the oblique side of the socket, to allow the flame to be easily directed downwards.

The lamp for experiments may be placed on the brass stand (A, *Fig. 3*), and fastened by a screw (a) to the brass pillar.

On the same apparatus may be adapted a brass triangle (c, *Fig. 3*), containing three bars of different lengths, with a moveable arm. This triangle serves as a support for drying or fusing any article over the flame of an oil or spirit lamp.

A small spirit-of-wine lamp may be profitably used for heating substances containing volatile ingredients, in glass tubes or alembics, and for melting various assays with

*Fig. 4.*



\* The leather is fastened in the following manner.—First, that side which is *not* to be cemented to the brass rim, must be covered over with molten wax, and when it hardens, the other side must be varnished with



bisulphate of potash in the platinum spoon, &c. For this purpose Berzelius uses a small phial of strong glass, with a ground top, in which is inserted a tube of silver or iron, for containing the wick. *Fig. 4* shews the intersection of such a lamp, which is about  $1\frac{1}{2}$  inch in diameter. Although a small brass lamp of another form is sometimes used, still, the glass one, if carefully handled, is in every respect preferable.

### III. THE BLOWING, &c.: AND DESCRIPTION OF THE FLAME.

The air for the Blowpipe is obtained from the mouth, and not from the respiratory organs, because they could not without fatigue afford a sufficient stream. The current of air is forced through the tube by the action of the *Musculi Buccinatores*, which are in the cheeks. During the blast, the communication between the chest and mouth is closed, so that respiration is carried on through the nostrils.

Until a person becomes accustomed to the use of this instrument, a strong and uninterrupted stream cannot be obtained; but after a few days' practice, it becomes easy. The fear of blowing being injurious to health, is soon removed.

*Fig. 5.*



To obtain an intense heat by blowing on the lighted combustible, requires a previous knowledge of its various parts; for on examining the flame of a lamp which does not emit smoke, it will be found divided into many. This appearance is more clearly seen in the flame of a wax or tallow candle, where four distinct portions are noticed. *Fig. 5* represents a flame: a fine clear blue colour is visible at *a b*; this azure tinge surrounds the burning wick, but becomes less distinct as it ascends, and, where the flame elongates perpendicularly, is

a strong solution of shell-lac in alcohol; over the brass edge rub also some of the solution, and then keep the leather pressed upon it for some time. When the spirit has evaporated, the shell-lac remains as a durable cement.



wholly diffused. In the centre of the flame is a very dark conical portion ( $c$ ); this part is encircled by  $d$ , the sphere whence nearly all the light emanates: external to this is  $a e b$ , which part affords scarcely any light, but gives out more heat than any other. The hottest part of this exterior flame is at  $f$ ; the heat diminishing towards the summit ( $e$ ), and base ( $a b$ ). When a very fine iron wire is introduced horizontally into the flame, it expands and becomes of a white heat at the outer part ( $a e b$ ), whilst at  $c$  it barely assumes a red one.

The cause of this phenomenon\* is as follows:—The small interstices of the wick, like other porous bodies, imbibe the melted tallow or wax, which is a combination of carbon, hydrogen, and oxygen; these by heat are converted into an empyreumatic oil (two of the compounds of carbon and hydrogen,†) and carbonic oxide, which burn and produce flame. The external part ( $a e b$ ) is the hottest, because it is in contact with the atmospheric air which supplies oxygen. The small blue margin is produced from combustion of carbonic oxide and a trifling proportion of carburetted hydrogen. The inner dark cone is filled with combustibles which are only partially consumed, ‡ on account of the air which passes through the flame being deprived of its oxygen by  $a e b$ .

Of the four different parts of the flame, three are as easily perceived in the flame of a lamp as in the flame of a candle; but the last ( $a e b$ ), if at all seen in the flame of the latter, must be very indistinct, as it only disperses a small quantity of light.

The following are the three parts necessary for researches with the Blowpipe:—the blue ( $a b$ ); the one which produces much light ( $d$ ); and the outer ( $a e b$ ). As the first and second are the most essential, I have described them at length.

\* Berzelius, *Lehrbuch der Chemie*, third edit., vol. 1, p. 345.

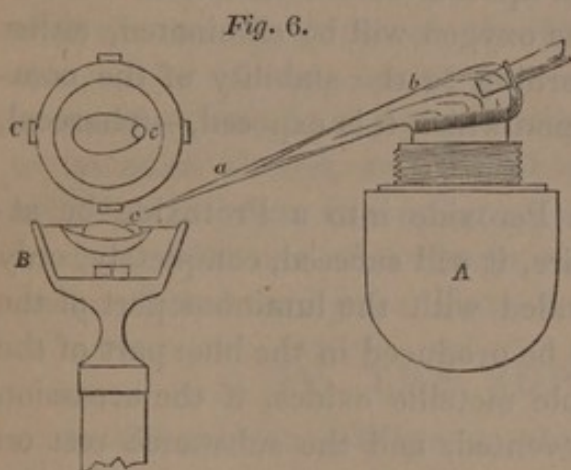
† [Carburetted hydrogen and olefiant gas.]—(Translator.)

‡ [To ascertain this, introduce a glass rod just over the dark part ( $c$ ); it will be blackened, from the deposition of carbonaceous matter.]—*Trans.*



## THE OXIDATION-FLAME.

When an operator with the Blowpipe blows lengthways on the flame of a lamp, so that the air just passes over the wick, (*Fig. 6.*) a long narrow blue flame will be formed,—



which is the same as *a b*, (*Fig. 5.*), excepting in shape; (the flame of *Fig. 6* is conical, that of *Fig. 5*, oval.) The heat is greatest at the extremity of this flame, because it is there the largest quantity of air sent through the Blowpipe is consumed.

In the free flame, the warmest part forms a girdle round the whole, the heat being most intense at *f*. Where the heat is greatest, substances which are not acted upon by an ordinary flame, oxidate, melt, and evaporate. The farther the matter can be kept from the apex of the blue flame, in a temperature sufficiently high, is favourable to oxidation.\* The operator must not blow too strongly, as it would be very deleterious to oxidation, especially if the assay rests on charcoal. The wick must be evenly cut, and kept free from all fibres, otherwise yellow streaks, which have a reducing effect, will be mixed with the blue flame. If these directions be adhered to, no difficulty can be found in ascertaining how far the tube should be introduced to produce the oxidating flame. It will be found most serviceable, not to have the opening too small in the point of the Blowpipe.

## THE REDUCTION FLAME.

By allowing a stream of air to pass over the wick, at a greater distance than in the production of the oxidating flame, the whole ignited mass receives the same direction, and appears as a long narrow luminous cylinder, the end being sur-

\* [Because the atmospheric air will have free access.]—*Trans.*



rounded with that part of the flame which emits only a small degree of light. By forcing the air as described, the particles rising from the wick (perceptible in a free flame,) are consumed, and the heat becomes concentrated in a less space. If such a flame be directed upon a metallic oxide, so that the point covers it totally, the oxygen will be eliminated, either partially or entirely, according to the stability of the compound, or the materials upon which it is exposed,—(charcoal, or platinum wire.)

If the reduction of a Peroxide into a Protoxide be attempted on a platinum wire, it will succeed, completely, only when the assay is surrounded with the luminous part of the flame: this effect can also be produced in the blue part of the flame, with easily-reducible metallic oxides, if the accession of atmospheric air be prevented, and the substance rest on charcoal. When the oxide of lead is mixed with a borax-glass, it can be reduced to the metallic state, upon charcoal, with as much facility in the blue as the bright part of the flame. Even if a piece of tin be liquefied upon charcoal in the blue part of the flame, no oxide is formed, because oxidation cannot take place when atmospheric air is excluded from the surface of a molten mass. The foregoing results avail for some, but not for all reducing tests upon charcoal. Positive results are attainable only in the bright part of the flame. The reducing flame is much more difficult to form than the oxidating; for this reason, the following directions must be studied.

(a) The wick should not be drawn too high out of the holder, nor must it be too short; because in the first case a smoky flame would ensue, impeding reduction, from the deposition of soot upon the assay, and in the second, the substance could not receive sufficient heat, if surrounded by the luminous part of the flame.

(b) To ensure exact results, have the wick evenly cut, and perfectly free from all extraneous fibres, as in the production of the oxidating flame.

(c) To secure success, when the reducing flame is produced, let it be kept uninterrupted.



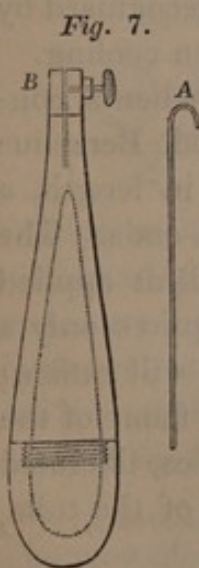
And, lastly,—

(*d*) The operator must use great precision as to the treating of a qualitative or a quantitative analysis. For qualitative examinations, a blowpipe point with an aperture narrower than that for oxidation is employed; but for quantitative researches, (which should be always performed on charcoal,) a blowpipe point with an opening rather wider is necessary. As, in many qualitative analyses, the flame has to be often altered, and a different point of the blowpipe affixed, it is better to employ a point with a narrower orifice, because, with one of too large a calibre, oxidation is more easily effected than reduction.

#### IV. THE SUPPORTS.

1. CHARCOAL.—When a substance requires melting, it should be exposed to the flame of the blowpipe upon a body that will augment the heat, without combining with the assay. The best substance to employ for this purpose is well-burned pine. It should be cut with a saw into long pieces, partly in the shape of a parallelopiped, and partly four-sided like a prism. For qualitative analyses, use the long sides of the charcoal, where the annular rings are on the margin, and for quantitative, use the interior part.

2. PLATINUM WIRE.—A moderately strong wire of platinum, about two inches long, and curved at one end, may be employed in many qualitative examinations with great advantage. (*Fig. 7, A.*) The curve serves as a support in all tests of oxidation and reduction, where alteration of colour only is to be observed. This support can be relied on, for it is totally free from the false variations of colour which are too often perceptible when the assay rests on charcoal. In the treatment of compound metals, or in such tests of reduction where a body easily melted is to be operated upon, charcoal, however, must be used. It is necessary to have at hand several platinum wires, so as to proceed without being obliged to





take off with force the glass adhering, after the extraction of the bead, or to wait for its solution in hydrochloric acid,—which is the better mode.

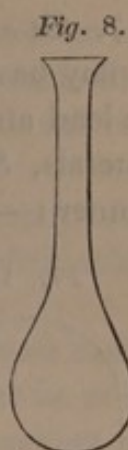
Should it happen that the platinum curve melts with the re-agents, it must be cut away, and a new one formed. A wire can be used for a very long time, and when it becomes too short to be held between the fingers, the straight end may be fastened into a cork, or a hilt containing a box for holding the rest of the platinum wires, until any of them are required. (*Fig. 7, B.*)

3. PLATINUM FOIL.—The use of platinum foil in qualitative examinations is very limited, but in some cases it may be successfully employed. Foil of thin drawn platinum, a few inches long, and half an inch broad, should be selected. This metal is such a bad conductor of heat, that when the assay is exposed to the flame of the blowpipe at one end, the other may still without inconvenience be retained between the fingers. Bodies in the metallic condition which are easily fused or reduced, should seldom be tested upon platinum foil, because, from its combining with them, a hole might be produced in the metal. Should this occur, the hole can be lopped off with scissors, and then, if the platinum be found too short, it can be easily held by forceps. Salts, or substances containing manganese, can in general be melted upon platinum foil; by this means the alkaline and earthy salts are distinguished, and the presence of manganese is recognised by the dull green colour communicated to soda, upon cooling.

4. GLASS TUBES.—When the volatile ingredients contained in ores and minerals are to be ascertained, Berzelius employs a glass tube from three to six inches in length, a quarter of an inch in diameter, and open at the ends. The body to be analyzed is inserted at one end, and heat applied to that part of the tube. If the compound requires only a slight degree of heat, the flame of a spirit-lamp will suffice; on the contrary, if a strong heat is necessary, the flame of the blowpipe must be employed. The volatile bodies, liberated during the roasting, sublime in the upper part of the tube, where they can be examined.



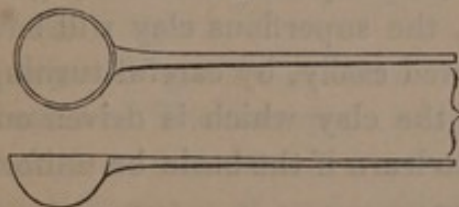
5. GLASS FLASKS.—If the quantity of water, or gaseous ingredient, contained in any substance, be required, or if a body, when heated strongly, decrepitates, a glass flask must be used, of the form of *Fig. 8*. If the small alembic be sufficiently wide for the free circulation of air, the vaporized bodies will be easily obtained. Flasks must not be employed in the sublimation of sulphur, arsenic, &c., lest combustion should ensue.



A supply of glass tubes and small flasks should always be in reserve. After the roasting has been performed in a tube, the part containing the fused substance should be removed with a file, and the remainder of the tube cleansed with the aid of a spirit-lamp and a wire enveloped in soft paper. When the tube becomes too short, one end can be sealed, and it may be used for tests of sublimation only.

6. TWO PLATINUM SPOONS.—The form of these utensils is shewn in the annexed wood-cuts. The diameter of *Fig. 9*

*Fig. 9.*



is 9-16ths, and that of *Fig. 10*, 3-8ths of an inch. The larger spoon has a handle of platinum (joined by rivets of the same metal), which can be encased in wood; the

*Fig. 10.*



small one, when heated, may be held with pincers.

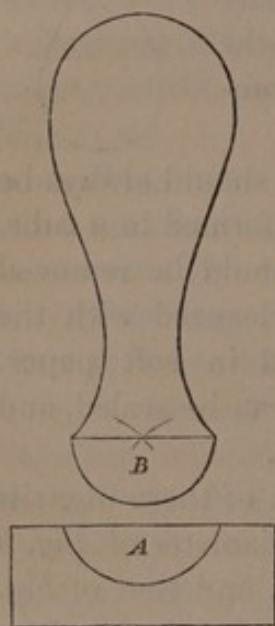
The first I use for melting various substances with bisulphate of potash, and for heating gold obtained in quantitative assays; and the second, for fusing substances with nitrate of potash. In some cases, when bodies containing water are heated with nitrate of potash, the platinum spoon becomes tarnished with the remaining particles of metallic oxides; these may be removed, either by a drop of sulphuric acid, or by a hot solution of bisulphate of potash.\*

\* [I have found that rubbing the platinum spoon with charcoal powder is the most effectual method of removing stains.—*Trans.*]



7. CLAY BASINS.—For quantitative metallic tests, I employ basins for roasting the Ores, and for the reduction of the lead and tin oxides contained in calcined and uncalcined minerals, &c. These dishes are formed in the following manner:—at first a fire-proof clay is kneaded into a stiff

Fig. 11.

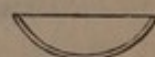


paste with water, and then the forming surfaces of the press, which are of box-wood, (*Fig. 11, A B,*) are rubbed over with a paint-brush dipped in oil. The width of (A), at the upper part, is  $\frac{7}{8}$ ths of an inch, and the depth,  $\frac{5}{16}$ ths; and (B) is constructed of a diameter smaller by the  $\frac{1}{32}$ nd of an inch. Over the middle of the concavity of the press, place a slip of paper, 3 inches in length, and  $\frac{1}{4}$  of an inch in breadth, and upon this a small clay ball about  $\frac{1}{2}$  an inch in diameter. Then take part (B) of the press, and stamp it horizontally upon the clay mass lying in part (A), as far as is required. This being accomplished, the superfluous clay will have

exuded, and (B) can be removed easily, by careful turning. With a small knife, cut away the clay which is driven out, and then examine the margin, to learn if the basin be uniform throughout, and sufficiently thin.

Fig. 12.

*Fig. 12* shews the cross section of one of my basins, which is only  $\frac{1}{32}$ nd of an inch thick.



If the basin be thicker in one place than in another, (B) must be applied with greater force to that part. When the basin is ready to be taken from (A), the convex part of the press must be carefully removed, and then the slip of paper gently pulled, to extract the clay dish. The utensil, with the slip of paper adhering, is now put away on a stone to dry, and the press is ready for the formation of others.

An operator unused to this work will find that sometimes the basins alter in shape after leaving the mould; but this can be remedied by pressing them with the fingers upon the convex part (B).



A few hours after drying, the slip of paper falls from the clay dish. When this occurs, the basin must be heated to redness over a fire, in a potter's vessel, or in a platinum crucible over a large spirit lamp.

In calcination, when these vessels are used, one may often serve as a lid to the other; but, for this purpose, their edges must be ground with a file.

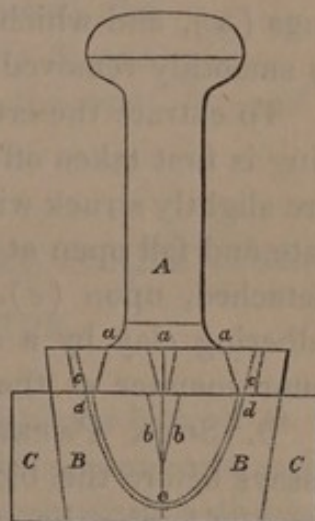
No labour should be spared in manufacturing these basins so as to make them sufficiently thin. The clay must be made neither too stiff nor too moist; the first fault will prevent the vessel assuming a proper form, and the second endangers its falling to pieces when removed from the machine. The true consistence of the clay can be ascertained by two or three experiments.

8. CLAY CRUCIBLES.—These crucibles are employed with advantage in the performance of quantitative lead assays, and in cases where the compound to be examined consists of sulphurets, arseniurets, and seleniurets. They are easily manufactured with an iron instrument, consisting of a mould and a stamp; the mould being composed of two parts, united by a ring.

*Fig. 13* gives the form of this instrument. The Stamp (A) is so bored through at four parts, (a), that the internal diameter of each orifice is little more than the diameter of the side; but the cross section of the outer part is nearly the double. These perforations serve for extracting the superfluous clay that has been placed in the mould for the formation of a crucible. The forming part is  $\frac{3}{4}$  of an inch in diameter at the top, and  $\frac{5}{8}$  of an inch long.

The Mould (B B) consists of two parts, accurately joined, which form at the outside a truncated cone. At the inner side, the distance of each part is 1-32nd of an inch from the

*Fig. 13.*





Stamp; and the edges near (*b*) are flattened, so that small depressions may be produced in the two opposite points of the mould, to prevent the crucible from being injured when the stamp is withdrawn. *c c* is the binding ring into which the mould is fitted, so that when unclasped, the mould falls with it exactly into a plane.

For the formation of these crucibles, a stiff paste is prepared with fire-proof clay; when this paste acquires a certain dryness, it is worked with the fingers into small balls, each containing more clay than is required for the manufacture of one of these vessels. Before introducing one of these balls into the mould, the forming parts and the stamp are well smeared with oil, by means of a fine brush, and then the mould, with the binding ring, is placed on an anvil resting upon a folded cloth to give elasticity. When the machine is thus arranged, the stamp is struck perpendicularly upon the ball with a mallet, until the protruding part (*c c*) rests on the margin (*d*) of the mould. The stamp causes the clay in the mould to spread, and the superfluous portion escapes through the small apertures at (*a*). After the stamp has remained in the mould for about five minutes, it is gently screwed out; by this means the clay contained in the openings (*a*), and which is still in conjunction with the crucible, is smoothly removed.

To extract the crucible requires great care. The binding ring is first taken off the mould, and then the two sides (*В В*) are slightly struck with a hammer; this causes them to separate and fall open at (*b b*), and the crucible remains perfectly detached, upon (*e*). The crucible, when cleansed from any adhering clay by a small knife, is dried and heated in the same manner as the clay basins.

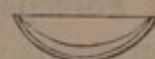
9. SODA PAPER.—In heating the quantitative silver assays before the blowpipe, Harkort found it necessary, instead of laying the ore, mixed with re-agents, upon charcoal, to enclose the mixture in a substance that will resist the first effects of the flame of the blowpipe, and prevent any loss of the particles. After some investigations, he ascertained that



fine letter-paper, saturated with a solution of soda, was the best. This paper, which is made use of advantageously, not only in silver, but also in many more quantitative analyses, is prepared in the following manner:—In a flat basin, dissolve half an ounce of crystallized carbonate of soda (perfectly free from any sulphate), in one ounce of distilled water; then steep a number of strips of fine letter paper in the solution for a few minutes, and allow them to dry in the open air. When dry, cut them into pieces,  $1\frac{3}{8}$  inch long, and  $\frac{7}{8}$  inch broad.

10. A MIXTURE *of seven parts of Charcoal with one part of Clay*.—This mixture is used for lining the clay basins in which Lead and Tin ores are to be calcined. It is best prepared as follows:—Weigh seven parts of fine dry charcoal powder, and one part of finely sifted clay; mix the latter with water, and then add the former; next, knead the mixture into a paste. The tenacious mass thus formed is dried in a warm stove, and reserved for use. When a basin is to be lined with this mixture, a small quantity of it must again be moistened, and strongly rubbed over the interior of the vessel employed for calcination. The thickness of the lining at the bottom of the basin must be about 1-16th of an inch, and at the top rather less. When this operation is finished, the clay dish is placed upon the triangle (c, *Fig. 3*), and dried over a spirit lamp. *Fig. 14* shews the cross section of such a lined basin.

Fig. 14.



## V. INSTRUMENTS, &c., REQUISITE IN ANALYSES WITH THE BLOWPIPE.

1. A BALANCE.\*—The operator may please himself as to the form of the Balance; but he should possess one sufficiently delicate to turn with 0.1 milligramme, when both pans are charged with 200 milligrammes.

\* [The descriptions of the Balance and the other instruments are greatly abridged,—their forms and applications being so well known to all blow-pipe analysts.—*Trans.*]



2. A SET OF WEIGHTS.—Weights, above 100 milligrammes, are seldom required. It is best, however, to have a box containing the Gramme\* and its divisions, viz.:—

One of 1000 milligrammes.

One of 500 ditto.

One of 200 ditto.

Two of 100 ditto.

One of 50 ditto.

One of 20 ditto.

Two of 10 ditto.

One of 5 milligrammes.

One of 2 ditto.

Two of 1 ditto.

One of 0·5 ditto.

One of 0·2 ditto. And

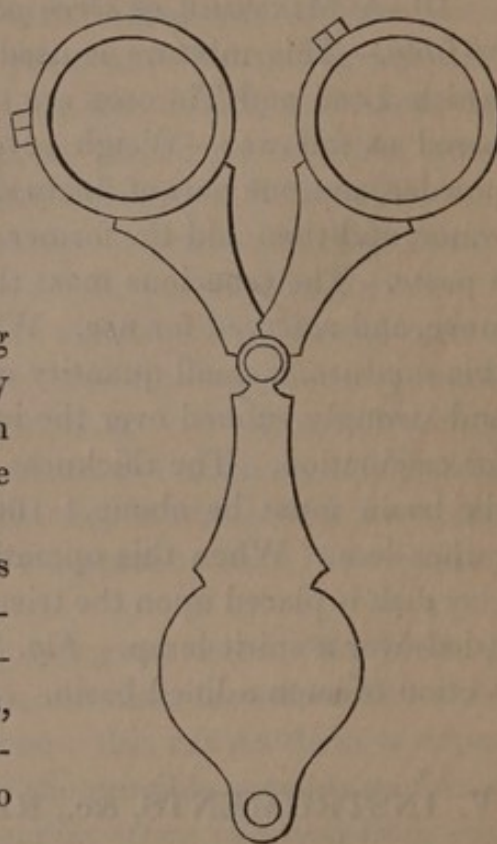
Two of 0·1 ditto.

3. A MICROSCOPE is indispensably necessary, in various reactions, and also to determine accurately the diameters of gold and silver globules upon the Scale.† An instrument formed of two fine plano-convex lenses, (*Fig. 15.*) of equal magnifying power, is very suitable; they must be so arranged that, when required, one can slide over the other, by a moveable arm.

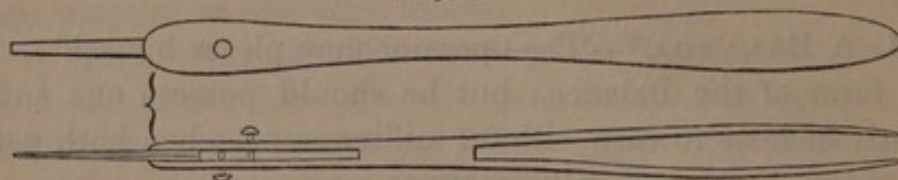
4. FORCEPS.—Various kinds of Forceps are needful for experiments with the blowpipe:—

(a) One with platinum points, to hold a small lamina of a specimen in the flame in order to try its fusibility. (*Fig. 16.*)

*Fig. 15.*



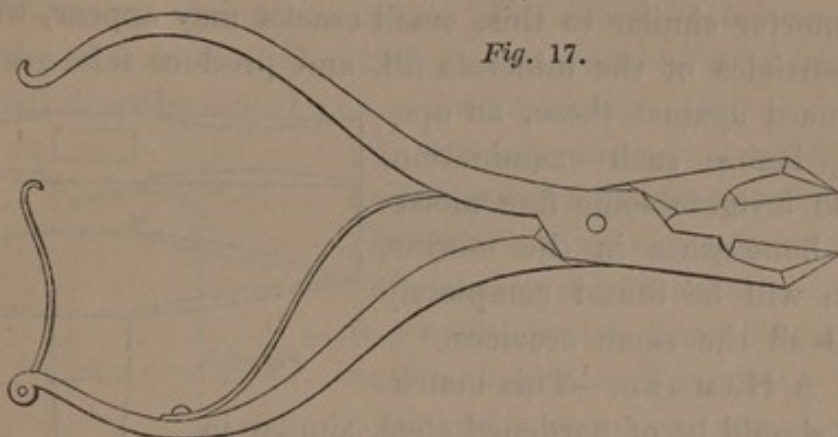
*Fig. 16.*



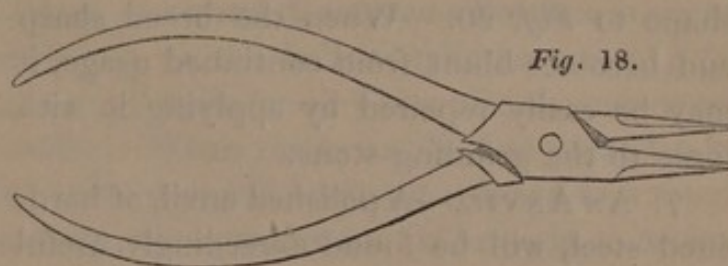
\* [1 Gramme = 10 Decigrammes, = 100 Centigrammes, = 1000 Milligrammes, = 15·438 English (avoirdupois) grains.—*Trans.*]

† [The Scale is described in Part III., immediately after the Gold Assay.—*Trans.*]

(b) One much broader and stronger than a pincers; for detaching small fragments from a specimen, without the formation of much dross. (*Fig. 17.*)

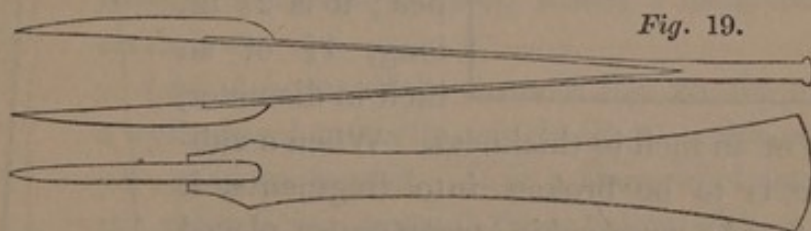


(c) One of steel (*Fig. 18.*), is employed for removing the scoræ in silver and gold



assayings, and in various other operations. Its points must be broad, and its inner surfaces rough.

(d) One of brass, for holding small fragments of minerals, &c., in the flame. (*Fig. 19.*)



(e) One of brass, with points exceedingly fine, for raising the balance, weights, &c., and placing the silver and gold globules upon the graduated scale; and,

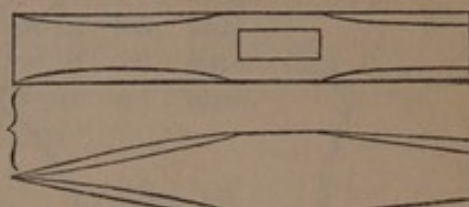
(f) One of iron, for trimming the lamp wicks.

5. A MORTAR AND PESTLE of *Agate*; or of *Calcedony*, which is preferable.—Mine, which I employ for all blowpipe analyses, is  $2\frac{1}{4}$  inches in diameter,  $\frac{3}{4}$  of an inch high, and  $\frac{7}{16}$ ths of an inch deep. It is transparent at the bottom, free from fissures, and is not only applicable to the pulveri-



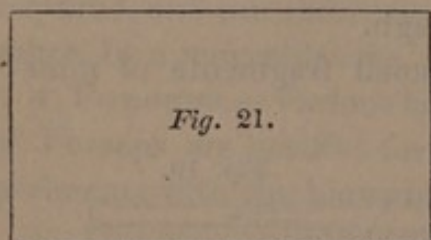
zation of substances that are to be qualitatively examined, but also for purifying the scorïæ and coaly particles of reduced metallic globules. When very hard substances are trituated in a mortar similar to this, small cracks may appear, which the particles of the minerals fill, and produce false results. To guard against these, an operator, before each examination, should levigate some fine moistened bone-ashes in the mortar, which will be found completely to fill all the small crevices.\*

6. A HAMMER.—This instrument should be of hardened steel, similar in shape to *Fig. 20*. When the broad sharp end becomes blunt from continued usage, it may be easily repaired by applying it with force to the grinding-stone.



*Fig. 20.*

7. AN ANVIL.—A polished anvil, of hardened steel, will be found exceedingly useful in a laboratory, for crushing minerals, ores, &c. Mine (*Fig. 21*), is in the form of a parallelopiped; it is  $2\frac{1}{2}$  in. long,  $1\frac{3}{8}$  of an inch in diameter,



*Fig. 21.*

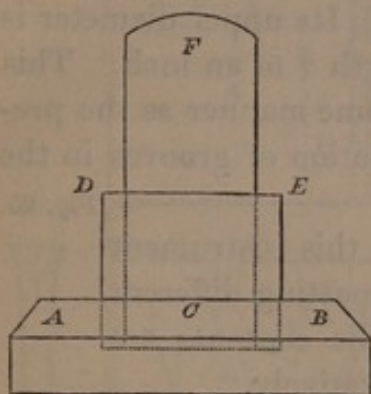
and  $\frac{5}{8}$  of an inch in thickness. When a substance is to be broken into fragments, it must first be wrapped in strong paper, placed upon the instrument, and then struck sharply with the hammer. Should the paper, in this process, be torn, and thrown off the face of the anvil, the malleable particles will be found adhering to the paper, and may be examined separately.

\* [Gahn having once lost the pestle of a mortar similar to the one above described, took a button of calcedony, of suitable diameter, and fastened it with sealing-wax to a cork. This new pestle answered so remarkably well, that it was the only kind he ever used afterwards. I have been obliged



If a mineral is to be heated upon charcoal before the blow-pipe, it can be reduced to small pieces in a machine similar to that described in "*Poggendorff's Annalen der Physick und Chemie. Jahrgang, 1831. 11tes Stück.*" Having often employed it in the quantitative examination of minerals, &c., I shall describe it:—Upon a hardened steel plate (A B, Fig.

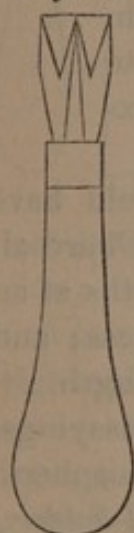
Fig. 22.



22), is a depression (C), containing a hollow cylinder (D E), which is completely filled by a massive cylinder (F), hemispherical at the end. The two cylinders (D E and F), are also hardened steel, well ground. When a mineral is to be pulverized, (F) is removed, and the substance is placed in the hollow cylinder (D E); the cylinder (F) is replaced, and struck forcibly with a mallet. When the two cylinders (F and D E) are separated, the substance will be found reduced to a rough powder. If necessary, this powder can be brought to an impalpable state, in an agate mortar.

8. CHARCOAL BORERS.—For quantitative blowpipe analyses, grooves of various dimensions are bored in the charcoal

Fig. 23.



that serves as a support. I employ three different borers, of hardened steel, which I shall here describe:—

(a) A borer of the form of Fig. 23, for making a cylindrical cavity in which silver, gold, or copper, may be heated. It is a four-sided pyramid, the sides being ground from below with a three-edged file, so that it appears like a double chisel, crossing at a right angle. Its under diameter is  $\frac{1}{3}$  of an inch. This instrument may be held in a wooden case, containing a brass ring.

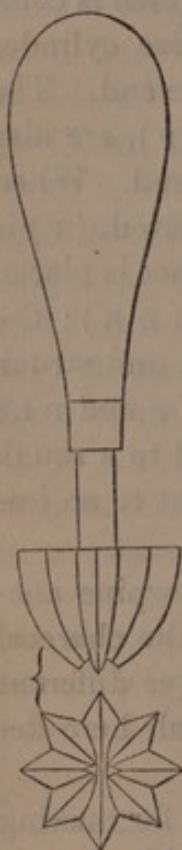
When a groove is to be made with the borer, it is placed at a right angle, pressed upon the char-

to have recourse to the same expedient, and have thought it proper to mention it, for the use of those to whom a similar accident may happen.—BERZELIUS.]—*Translator.*



coal with force, and turned upon its axis until the groove is sufficiently deep. The instrument is then withdrawn, and the charcoal-dust removed from the cavity. A silver assay requires a deeper groove than a copper one, owing to the former metal requiring, when operated upon, to be mixed with lead.

Fig. 24.



(*b*) A second Charcoal borer, for forming larger grooves. (*Fig. 24.*) Its upper diameter is  $\frac{7}{8}$  of an inch, and its length  $\frac{3}{4}$  of an inch. This borer is handled in the same manner as the preceding one, for the formation of grooves in the charcoal.

Fig. 25.

The cavity formed by this instrument serves as a support in roasting different substances whose metallic contents are to be determined quantitatively.

(*c*) A third Charcoal borer, of the form of *Fig. 25.* This borer has one end of the same form as that first described, but the double chisel is only 3-16ths of an inch in diameter. I employ it for making incisions in the charcoal, to contain quantitative lead and tin assays. The other end has a diameter of  $\frac{3}{8}$  of an inch, it is sharp like a spatula, and serves to make small grooves for qualitative experiments.

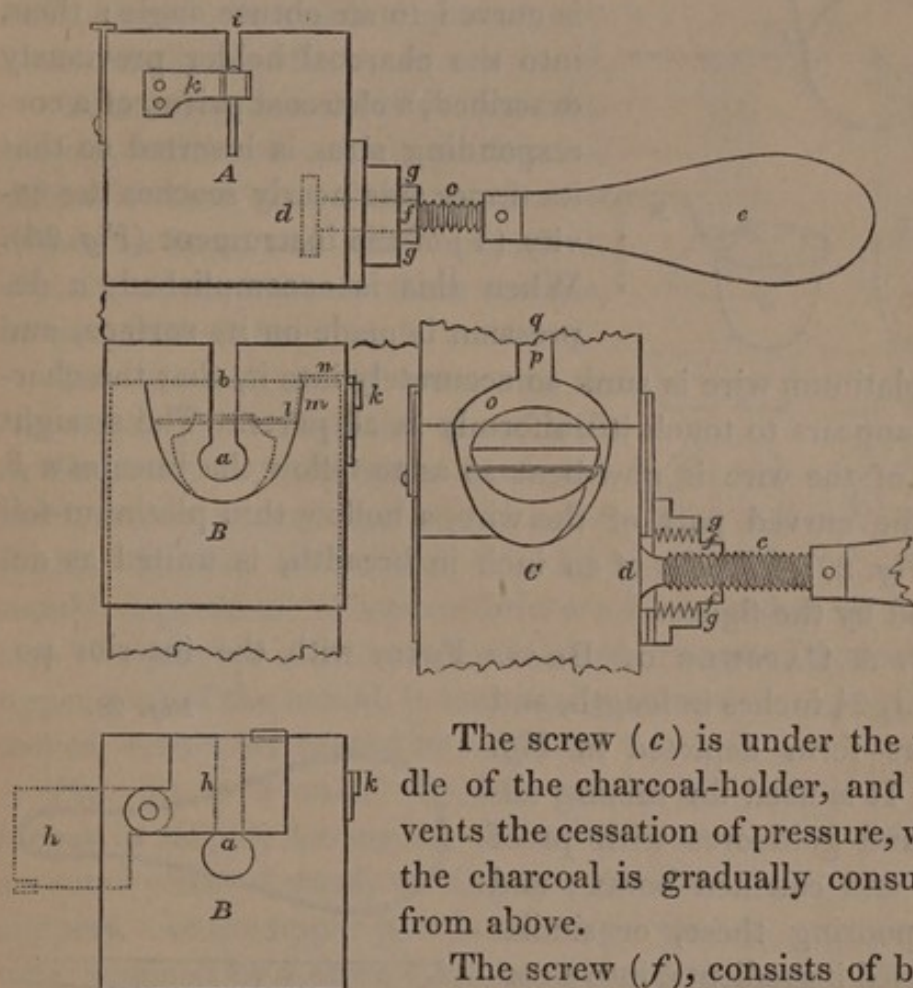


9. A CHARCOAL SAW.—This instrument should have very fine teeth, for cutting with ease long pieces of Charcoal. The one I employ is five inches in length, three-eighths of an inch in breadth, one sixteenth of an inch in thickness; and is provided with a wooden handle, three inches in length.

10. A CHARCOAL HOLDER.—In quantitative assayings, which are to be melted without the accession of atmospheric air, the charcoal must be encased in iron foil (*charcoal-holder.*) *Fig. 26* presents several sections of this instrument. Its sides are  $1\frac{3}{8}$  inch in breadth, and  $1\frac{1}{2}$  inch in height. At the anterior side (*B*) it is provided with a round opening (*a*), termi-

nating at *b*; and at the reverse, is a small iron screw (*c*), provided with a wooden case (*e*), on whose inner end is an iron disk (*d*), which revolves round its axis.

Fig. 26.



The screw (*c*) is under the middle of the charcoal-holder, and prevents the cessation of pressure, when the charcoal is gradually consumed from above.

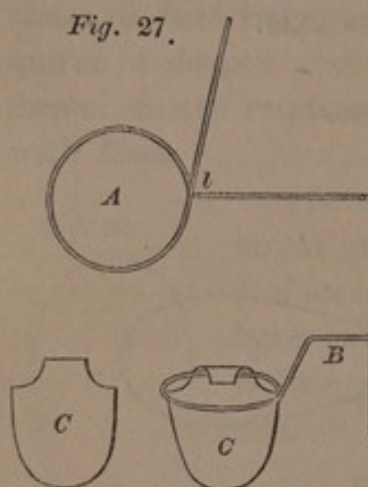
The screw (*f*), consists of brass, and can readily be removed, because it is inserted at *g*. A small iron plate (*h*) is fastened, by a rivet, to the face of the charcoal-holder, so that the incision (*b*) can be opened or closed, as the figure shows. On the right side (*A*) of the instrument is a small opening (*i*), a quarter of an inch in length, for the reception of the platinum wire, to be described hereafter; and under this aperture is a small brass case (*k*), for holding its end.

11. A PLATINUM WIRE AND FOIL.—When an ore is to be roasted, or an assay melted, in a clay basin, the latter must be supported by some substance placed in the cavity of the



charcoal. For this purpose, a strong platinum wire  $3\frac{1}{2}$  inches in length, will be found applicable. On the end of the platinum wire, a ring (Fig. 27, A) is

Fig. 27.

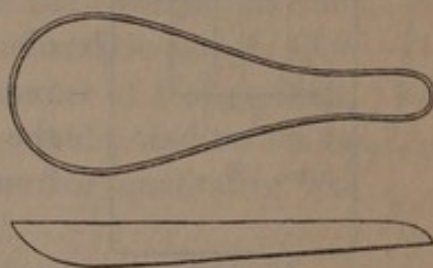


made, by means of the small forceps (Fig. 18), and the touching point (l) is curved to an obtuse angle: then, into the charcoal holder previously described, a charcoal prism, of a corresponding size, is inserted so that its upper side nearly reaches the cavity (i) of the instrument (Fig. 26). When this is accomplished, a depression is made on its surface, and

the platinum wire is sunk so accurately into it, that the charcoal appears to touch it uniformly in all parts. The straight part of the wire is now bent so as to follow the lines *m n k*. To the curved part of the wire, a hollow thin platinum foil (c, Fig. 27), 9-16ths of an inch in breadth, is united as depicted by the figure.

12. A CAPSULE OF BRASS FOIL, with the interior polished,  $2\frac{1}{2}$  inches in length, and of the form depicted in Fig. 28. It is used for mixing silver and gold ores with proof lead and calcined borax; and for pouring these, or similar mingled ingredients, into a cornet of soda paper.

Fig. 28.



13.—A SMALL SCISSORS, sufficiently strong for cutting metallic laminae, as well as soft materials.

14. A KNIFE.—For many researches, a steel knife, well sharpened and magnetized, is requisite. I employ a small English one for mixing the various fluxes in quantitative assays, and also for determining the degree of hardness of many metallic bodies.

15. Some FILES, of different sizes and fineness, three-edged, flat, half-round, and round, which it is unnecessary here to describe, are required in many examinations.



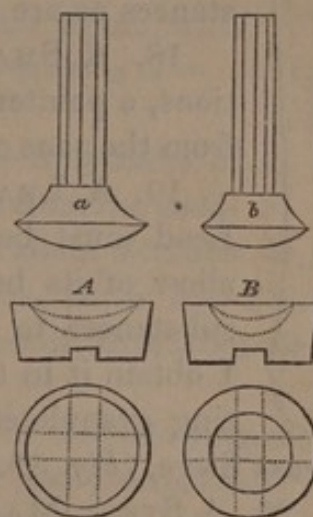
16. AN IRON INSTRUMENT *for the formation of CUPELS*;\* with a Bolt and Stand.—Small Cupels, of bone-ashes, are necessary for refining plumbiferous alloys (obtained in quantitative assayings), containing Silver or Gold. The best method for the preparation of these Cupels is to cast them in a metallic mould; in which they are to remain till the cupellation is completed.

Generally, two Moulds are employed, with concave deepenings of different sizes, for the formation of cupels in large and small operations. *Fig. 29, A, B*, presents these moulds, as well as their bolts, *a, b*.

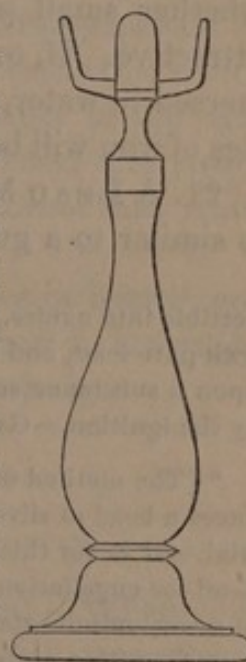
The Moulds are of iron, and in the upper part, 13-16ths of an inch in diameter; the bolts are of hardened steel, partly ground and of a circular form. In the under part of each mould, depressions of a cross form are filed, so that when one arm of a pincers is inserted, and the other pressed against the upper part of the mould, it may easily be removed, even when heated to redness.

*Fig. 30* is a small wooden stand,  $3\frac{1}{4}$  inches in height, having on the under part a round piece of wood, serving as a durable support. In the upper part is a strong brass wire, fastened by a screw; and at the end of this wire is a brass cross, the arms elevated to an angle of  $85^{\circ}$ , so as to allow the mould to be conveniently placed between them. When the transverse incisions in the bottom of the iron instrument are not brought into contact with the arms of the brass cross, an operator is enabled to raise or depress the instrument continually, so as to afford the required heat.

*Fig. 29.*



*Fig. 30.*



\* [*Cupellation*.—This operation is resorted to for the purpose of separating what are called noble metals, from those which are more readily con-



*Fig. 31.* 17. AN IVORY SPOON, well polished and ground, 5-16ths of an inch in breadth, and of the form given in *Fig. 31*. The handle of this spoon, which is in the form of a spatula, is for mixing in the capsule, ores with pure lead and calcined borax; and the spoon part is necessary in the weighing of Ores and such substances as are to be fused.



18. A SMALL BRUSH.—For qualitative examinations, a painter's brush is indispensable, for detaching from the pans of the Balance any remaining powder, &c.

19. A LEAD SIEVE.—In Blowpipe analyses, the Lead must be in a very minute state of division, to allow of its being intimately mixed with the various substances to be operated upon. I obtain it in this state, by agitating granulated Lead in a small Brass Sieve, (*Fig. 32.*) \*

*Fig. 32.*



20. A STEEL MAGNET.—A strongly magnetized steel blade, or a steel magnet about  $3\frac{1}{2}$  inches long, and  $\frac{1}{8}$  of an inch in diameter, will be useful in ascertaining whether small particles of the ores under examination are attractive. If, to a mixture of finely-divided iron and tin, immersed in water, the end of the magnet be applied, the particles of iron will be found adhering, when it is withdrawn.†

21. A LEAD MEASURE.—The instrument, (*Fig. 33.*) which is similar to a gunpowder-measure, is for avoiding the weigh-

vertible into oxides. The operation consists in fusing the alloy on charcoal with pure lead, and then heating the resulting bead in the oxidating flame, upon a substance sufficiently porous to absorb the fused oxides produced by the ignition.—GRIFFIN ON THE BLOWPIPE.]—*Translator.*

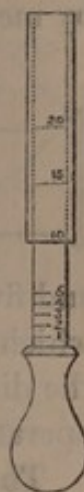
\* [The method of assaying is so very delicate, that it almost always produces a bead of silver, when the common lead of commerce is submitted to trial.—It is for this reason that so much care must be taken in preparing Lead for cupellation. The best method for obtaining the Lead pure, and in a very minute state of division, is to precipitate it from a solution of the acetate, with a plate of zinc.]—*Trans.*

† [This experiment always succeeds, if, in a mixture thus immersed, small particles of iron are present.]—*Trans.*

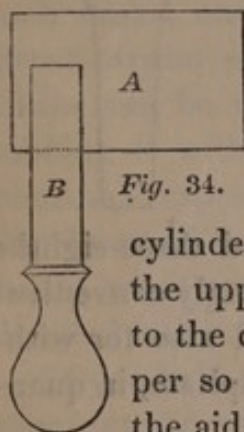


ing of a certain quantity of lead, in quantitative assay-*Fig. 33.* It consists of a glass tube, open at the extremities, containing a small wooden cylinder. This glass tube is divided into parts of equal degrees, 10, 15, 20, each of which are capable of holding 100 milligrammes of granulated lead. For quantities less than 100 milligrammes, the piston is graduated as in the figure, with

1, 2, 3, 4, 5, . . . . corresponding to —  
10, 20, 30, 40, 50, milligrammes.



22. A SMALL WOODEN CYLINDER.—A cylinder of wood is employed to make small cornets from the soda-paper described at page 14. It should be about 9-32nds of an inch in thickness, as in *Fig. 34, B*; and, for convenience, should have a handle at one end.

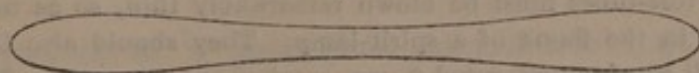


*Fig. 34.*

The cornet is made in the following manner. A slip of paper (A) is so placed under the cylinder, that one of its long sides projects beyond the upper end of (B), as in the figure, a space equal to the diameter of the cylinder. The part of the paper so protruding is then folded back on (B), with the aid of a small spoon; the part of the paper to the left of (B) is next folded over the cylinder, turned in such a manner that the remaining portion of the paper to the right of (B) is folded over the latter, and the closed end struck upon a hard even surface, in order that the cornet may retain its shape, after the removal of (B).

23. A POLISHED IRON SPATULA, 4 inches in length, and of the form given in *Fig. 35*. It is employed in levigating such alligations as are to be roasted quantitatively.

*Fig. 35.*

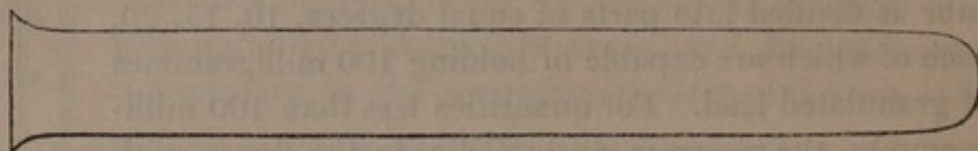


24. TEST-TUBES, with a Funnel and Stand.—When compounds which contain several heterogeneous ingredients, cannot be accurately recognized in the Blowpipe-flame, an operator must resort to liquid testing, to ascertain each con-



stituent, either qualitatively or quantitatively. This is effected by means of test-tubes, (*Fig. 36*), five and a half inches long,

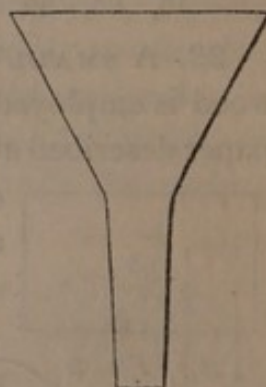
*Fig. 36.*



and five-eighths of an inch in diameter,\*—which are very well adapted for containing the dissolved compound, when it is to be operated upon by re-agents.

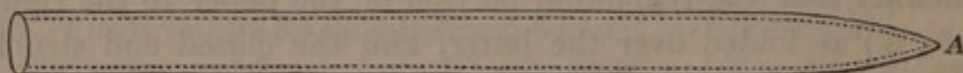
The Funnel, (*Fig. 37*), is for the reception of a Paper Filter, into which the turbid liquid is poured from a test-tube, to separate the precipitate produced by a re-agent, from the clear solution.

*Fig. 37.*



25. A small Glass PIPETTE.—This instrument should be five inches in length, and three-eighths of an inch in diameter, (*Fig. 38*.) It is exceedingly convenient for affusing precipitates with hot water; and also for withdrawing the supernatant liquor from the precipitate, in quantitative Gold and Tin analyses.†

*Fig. 38.*



26. AN EDULCORATOR.‡—The Edulcorator (*Fig. 39, A*), is a bottle, capable of holding about a pint, and the mouth of which is closed by a cork, through which a short piece of strong glass tube is passed; but so as not to project far

\* [The test-tubes must be blown remarkably thin, so as not to crack when held in the flame of a spirit-lamp. They should also be provided with a small wooden perforated frame, serving as a support.]—*Trans.*

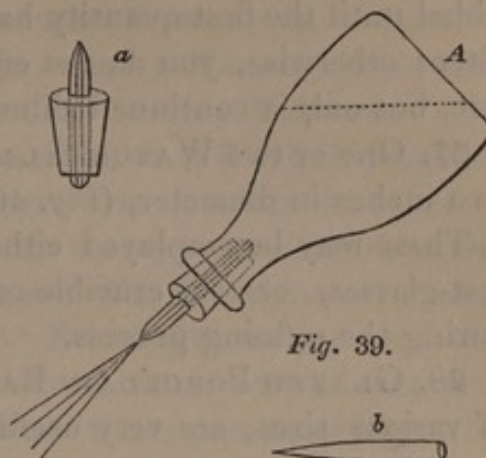
† [The point only should be slightly contracted, and not drawn out into a capillary tube.]—*Trans.*

‡ [The above description of this very useful instrument is taken from "Griffin's Experimental Chemistry," as it is much better than the one given by my author.]—*Trans.*



beyond the cork. This adjustment of the cork and tube is represented in *Fig. 39 (a)*.

Another, and an improved form of the *tube*, is shewn in *Fig. 39 (b)*. The cork must fit very tightly in the neck of the bottle; and the external orifice of the glass tube must be very small,—never exceeding 1-40th of an inch in diameter. It is a convenience to have two



*Fig. 39.*

such flasks, one with a tube wider than the other, to give a larger stream of water, when required. The orifice of such a tube can be widened by grinding it upon a sandstone, or rubbing it with a file which has been anointed with camphorized turpentine; or it can be narrowed, by holding it in the flame of the spirit-lamp, or before the blowpipe. The bottle, when corked, should be rather more than half full of distilled water.

If you hold the edulcorator with the cork downwards, and then put the point of the tube into your mouth, and blow air into the bottle, the water, upon your removing the point from your mouth, will, for a few moments, be expelled from the edulcorator with considerable force. It passes out in a fine stream, which can be directed upon the precipitate in the filter, so as to stir it up and wash it, with great ease and effect. Near the end of the edulcoration, the jet of water should be directed towards the edges of the filter, and not upon the precipitate itself, by which means the precipitate is washed to the bottom of the filter, and brought into a small compass.

Precipitates of a gelatinous consistence require much washing. The jet of water is sometimes deficient of force sufficient to stir them up. In this case, you may agitate them with the round end of a small glass rod; but you must be exceedingly careful not to force the rod through the filter, otherwise the filtration will have to be repeated. It is to be



observed that, after every addition of water, no more is to be added until the first quantity has completely run through the filter; otherwise, you do not effect a washing of the precipitate, but only a continued dilution of the solution.

27. One or two WATCH-GLASSES two inches in diameter, (*Fig. 40 a*).

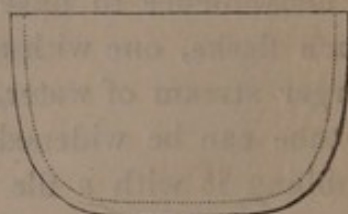
—These may be employed either as test-glasses, or as crucible-covers during the refining process.

28. GLAZED PORCELAIN BASINS of various sizes, are very useful for the precipitation or evaporation, &c. of liquids, and also for heating in the dry state, earths and metallic oxides with re-agents. The form of two such vessels is given in *Figs. 40 and 41*.

*Fig. 40.*



*Fig. 41.*



29. A cylindrical Bottle, of lacquered sheet iron, provided with a screw top, for containing lamp oil.\*

## VI. BLOWPIPE RE-AGENTS.

The three principal re-agents, indispensable in examinations with the Blowpipe, are—

*Carbonate of Soda;*

*Biborate of Soda, or Borax;*

*Phosphate of Soda and Ammonia, or Microcosmic Salt.*

These tests are to be provided in a state of purity; I shall therefore describe each separately.

1. *Carbonate of Soda*.—When a current of carbonic acid gas is passed through a saturated solution of the apothecaries' soda in water, bicarbonate of soda is precipitated in crystalline grains, which must be well washed with cold water, and

\* [The rest of the chapter describes a box for charcoal, test-tubes, funnels, tinder-boxes, re-agent boxes, balance case, &c., particular description of which is unnecessary.]—*Translator*.



dried. In this state it may be employed for qualitative analyses; but, when required in quantitative analyses, it must be calcined in a flat porcelain matrass, to liberate the second atom of carbonic acid, and its water of crystallization.

Winkler obtains the bicarbonate of soda free from sulphate of soda, in the following manner.—He dissolves 4 ounces of crystallized carbonate of soda in 8 ounces of distilled water, and adds to the solution  $1\frac{1}{2}$  ounce of carbonate of ammonia; then heats the mixture to  $133^{\circ}$  Fahrenheit, in a water-bath. After some time, a large quantity of bicarbonate of soda precipitates, which is separated from the supernatant liquor by filtration.

To ascertain whether the carbonate of soda contains any sulphate, mix two parts with one of pure silica, and fuse this mass upon charcoal, in the reduction flame; if the smallest quantity of sulphate of soda is present, the resulting bead will be of a deep yellow or red colour, owing to the formation of sulphuret of sodium.

A second method for ascertaining the presence of sulphate of soda in the carbonate is, to heat a small quantity of carbonate of soda upon charcoal, in the reduction flame, until it is entirely absorbed. Cut out, with a small knife, this part of the charcoal, and digest it in water; to the solution add a small piece of bright metallic silver; if sulphuric acid had been present, the surface of the silver would become first yellow, then brown, and finally black.

The Carbonate of Soda, in qualitative examinations with the blowpipe, serves to determine—first, whether a body fuses or is rendered soluble:—second, the presence of silicic acid in combination:—and third, to reduce metallic oxides.

In quantitative analyses, it serves as the means of rendering soluble, silicic acid, tungstic acid, and titanitic acid combinations; and also, for quickening the reduction of different metallic oxides.\*

\* [The best method of preparing a large quantity of bicarbonate of soda is, to pass a stream of carbonic acid gas over an atomic mixture of crystals of soda and the dry carbonate. Great heat is evolved during the process, owing to the rapidity with which the gas is absorbed.]—*Trans.*



2. *Biborate of Soda*.—This salt, which is met with in commerce, in large masses, must be re-crystallized, to free it from foreign matters. If the crystals yielded are pure, they will give a transparent glass when heated upon the ring of a platinum wire. In quantitative analyses, owing to the crystals occupying so much space when heated, on account of their intumescence, the borax must be employed in a calcined state. According to Berzelius, almost all substances dissolve when fused with borax, and form, with its ingredients, acid and basic combinations; for this reason it is such a valuable flux in blowpipe experiments.\*

3. *Phosphate of Soda and Ammonia*.—This salt is best obtained by heating together, in distilled water, 100 parts of crystallized phosphate of soda, with 16 parts of sal-ammoniac. Chloride of sodium separates; and the liquid, when filtered and evaporated, affords the salt in fine crystals. When this salt is heated upon charcoal, or platinum wire, it loses its water and ammonia, and is converted into metaphosphate of soda, which, in consequence of its excess of acid, has the power of fusing almost every chemical compound.

This salt, in the liquid state, detects magnesia and the protoxide of manganese.

Besides the three re-agents which I have described, there are many others necessary to facilitate the fusion, or to effect the decomposition of certain substances heated before the blowpipe, and also to aid in the detection of particular elements; these are:—

4. *Nitrate of Potash* (Saltpetre).—This salt is obtained in small prisms, when the saltpetre of commerce is dissolved in hot water, and the liquid allowed to cool gradually.

(a) The production of colours, in beads formed with borax or microcosmic salt upon the platinum wire, can sometimes be facilitated by the addition of saltpetre. A small crystal of this salt is supported near the blowpipe-lamp, upon a porcelain capsule, and when the bead is removed from the flame,

\* [Graham remarks that the anhydrous salt is very fusible by heat, and forms a glass. This glass possesses the property of dissolving most metallic oxides, the smallest portions of which colour it.]—*Trans*.



it is instantly pressed upon the saltpetre. The globule immediately tumefies, and the oxidized metal exhibits its colour on the exterior. If the blowpipe-flame is at all altered during this examination, the re-action will be destroyed. By this expedient, a portion of manganese, so minute as otherwise to pass unnoticed, can be readily discerned, by a beautiful emerald green colour being produced.

(b) Saltpetre also has the power of oxidizing arsenic and chromium; converting them into acids which combine with its base.

5. *Neutral Sulphate of Potash*.—This salt is obtained in a state of purity from apothecaries.\* In the liquid state, it is useful for ascertaining the presence of zirconia and oxide of cerium, should an unsatisfactory result be produced before the blowpipe-flame.

6. *Bisulphate of Potash*.†—This salt generally presents itself when the neutral sulphate is strongly heated with oil of vitriol. If the bisulphate obtained, contains too much acid, (which must, if possible, be avoided,) it emits thick white vapours, when heated only slightly. Bisulphate of potash, in solution, indicates lithia, boracic acid, nitric acid, hydrofluoric acid, bromine, and iodine; and separates barytes and strontian from other earths and metallic oxides.

7. *Vitrified Boracic Acid*.‡—This acid serves, in qualitative examinations, to detect the presence of phosphoric acid in minerals, and small portions of copper in lead alloys. In

\* [It precipitates in oblique four-sided prisms, when oil of vitriol is added, drop by drop, to a concentrated solution of caustic potash.]—*Trans.*

† [The fusible salt remaining when nitrate of potash is decomposed in a retort by two equivalents of oil of vitriol. The excess of acid in this salt, acts upon metals and alkaline bases, very much as if it were free.—*GRAHAM.*]—*Trans.*

‡ [“This acid is generally prepared by dissolving the borax in four times its weight of water, at 212°; the solution is filtered while hot, and a quantity of oil of vitriol, equal to one fourth of the weight of the borax, immediately added. The sulphuric acid unites with the soda, and forms sulphate of soda, which remains in solution; while the boracic acid, on cooling, separates in thin shining crystalline plates. These plates are drained, and being sparingly soluble, may be washed with cold water, and, afterwards, re-dissolved in boiling water, and re-crystallized. The boracic acid still



quantitative analyses, I generally use it to ascertain the quantity of copper contained in a lead ore, and also the quantity of copper united with various metals.

8. *Chloride of Ammonium* (Sal-ammoniac).—This salt can be obtained in a sufficiently pure state for analysis, by recrystallizing the sal-ammoniac of commerce.

It serves, in qualitative examinations, for precipitating alumina from a potash solution; and also, for separating magnesia, and the protoxide of manganese, from earths and metallic oxides.

9. *Carbonate of Ammonia*.—Take two parts of marble (carbonate of lime), and one part of chloride of ammonium. Pulverize them separately, and mix the whole intimately together. Put the mixture into a spacious retort, and insert the neck into a receiver. Heat the retort by means of a lamp, and keep the receiver immersed in cold water. Carbonate of ammonia will sublime and condense in the solid state: remove it by a spatula, and keep it in a well-stoppered phial. A solution of this salt is useful for separating glucina from alumina, and the oxide of uranium from peroxide of iron.

10. *Oxalate of Ammonia*.—When a hot solution of carbonate of ammonia is treated with oxalic acid, this salt is gradually deposited, in needle crystals, as the liquor cools. It is an excellent test for lime.

11.—*Nitrate of Silver*.—Dissolve pure silver in nitric acid, and filter the solution, to separate from it the small particles of gold: then set aside the filtered solution, in a dark place, to crystallize.

The nitrate crystallizes in colourless tables (anhydrous), which become purple when exposed to the sun's rays.\*

The silver can be obtained from the mother liquor of the crystals, by adding a solution of chloride of sodium (common salt). Chloride of silver is precipitated, in a bulky and curdy

retains a small quantity of sulphuric acid, probably in a state of chemical combination, and if required of absolute purity, must be fused at a red heat in a platinum crucible, then re-dissolved and crystallized. The density of the vitrified acid is 1.83."—GRAHAM.]—*Trans.*

\* [From the reduction of a portion of the oxide of silver to the metallic state.]—*Trans.*



state. By heating this Chloride upon charcoal, after being filtered and well washed with carbonate of soda and borax, a bead of metallic silver is produced.\*

Nitrate of silver is an excellent re-agent in examinations with the blowpipe, for detecting traces of phosphoric, arsenic, or hydrochloric acids.

12. *Nitrate of Cobalt in solution.*—The pure metal must be dissolved in diluted nitric acid, and the solution evaporated to dryness, then re-dissolved and filtered, to separate any foreign matter. †

There are a few substances not easily distinguished by simple ignition, which acquire marked characters on being moistened with a solution of nitrate of cobalt, and then heated strongly in the oxidating flame. These are,—

Alumina, acquiring a beautiful *pale blue* colour,

Magnesia, a *rose-red* tint, and

Zinc, a *bright green*.

Fig. 42.

A few drops of a solution of cobalt may be placed upon the substance to be operated upon, by means of platinum wire, or a small dropping-tube, passed through the cork of a bottle in which the solution is preserved.—See Fig. 42.



13. *Chloride of Platinum and Sodium* in solution. This double salt crystallizes in transparent prisms of a bright yellow colour, when a mixed watery solution of chloride of platinum and chloride of

\* In this process, Chloride of Potassium is formed, and oxygen and carbonic acid escape with effervescence.

† [Great care must be taken to have the Cobalt perfectly free from Iron, Nickel, and other metals with which it is generally contaminated.]

“To separate Cobalt from Nickel, with which it is almost always associated, the mixed oxalates of the two metals are dissolved in ammonia; the liquid is then diluted, and exposed to the air in a shallow basin for several days. The Ammonia evaporates; the salt of Nickel precipitates as a green powder, and the salt of Cobalt remains in solution. The liquid is then decanted, and if there is no additional precipitate in twenty-four hours, it is free from nickel, and may be evaporated to dryness, and then decomposed by ignition in a covered crucible. The oxide is reduced by the carbon of the acid, which goes off as carbonic acid, while the metallic Cobalt remains as a black powder.” ]—*Trans.*



sodium is slowly evaporated. The crystals thus obtained must be well washed, and then dissolved in absolute alcohol.\*

This solution determines the presence of potash, even when in combination with soda or lithia.

14. *Sulphate of Copper* (Blue Vitriol.)†—This salt may be employed with advantage in many cases, to determine the acid of chlorine, when contained in complex combinations.

15. *Acetate of Lead* (Sugar of Lead.)‡—This re-agent is only required as a confirmatory test for chromium, in such combinations as do not give a striking result with the blowpipe.

16. *Oxalate of Nickel*, perfectly free from iron and cobalt. It is obtained perfectly pure in the following manner. Heat copper-nickel (arsenic-nickel) to redness with a quantity of borax in a clay crucible, and when in a state of fusion remove the crucible from the muffle, and expose it to a free current of air. If the ore had contained iron, cobalt, lead, antimony, or sulphur, they would have been oxidized and dissolved in the borax, while the nickel would remain in combination with the arsenic. Separate the arsenite of nickel from the dross, and examine a portion of it before the blowpipe for cobalt. If it be not free from this metal, repeat the preceding operation with borax. When the mixture is entirely free from cobalt, heat it strongly with a quantity of carbonate of soda and nitrate of potash; during this process the arsenic acid combines with the soda or potash, and the oxide of nickel remains in an uncombined state. Dissolve the soluble part of

\* [Chloride of Platinum will answer the purpose; but no ammoniacal salts must be present when an operator is testing for Potash.]—*Trans.*

† [“Sulphate of Copper may be formed by dissolving copper in sulphuric acid, diluted with half its bulk of water, by ebullition, when the metal is oxidated, with formation of sulphurous acid. But the sulphate of copper is more generally prepared on the large scale, by the roasting and oxidation of sulphuret of copper. It forms large rhomboidal crystals, of a sapphire blue, containing five equivalents of water, which lose their transparency in dry air.”—GRAHAM.]—*Trans.*

‡ [This salt is generally prepared by dissolving the protoxide of lead in the acid procured by the distillation of wood. It effloresces in the air, and is partially decomposed; for this reason it should be kept in a bottle fitted with a well ground stopper.]—*Trans.*



the residue in boiling water, and filter. The oxide of nickel which remains, after being well affused with water, must be dissolved in muriatic acid, with the aid of heat; and the nickel precipitated, as an oxalate.

If the arsenic-nickel should contain copper, it must be precipitated from an acid solution of it, by sulphuretted hydrogen gas, previously to the heating with borax.\*

The oxalate of nickel is used in qualitative examinations for the detection of potash, in a salt which also contains soda or lithia.

17. *Black Oxide of Copper*.—It may be obtained artificially, by calcining metallic copper; through precipitation from the salts of copper by the addition of pure potash; or by heating nitrate of copper to redness.

It is useful for ascertaining the presence of small quantities of chlorine, when in combination, &c.

18. *Silica, or Silicic Acid*.—This compound is obtained in a state of purity as follows:—Heat to redness in a platinum crucible, powdered rock crystal (quartz), with a quantity of carbonate of soda or potash; dissolve the fused compound in water; add to the solution a quantity of hydrochloric acid, and evaporate to dryness. A fine gritty powder remains, which, being well affused with water, and then ignited, gives pure silica.

Silica, with soda, is an excellent test for the presence of sulphuric acid; and, when in combination with borax or soda, separates tin from copper.

\* [Professor Liebig has lately described a beautiful method for separating Nickel and Cobalt, by means of Cyanide of Potassium. When cyanide of potassium is added to a solution of nickel and cobalt, a precipitate of cyanide of nickel is formed, which re-dissolves on adding an excess of the re-agent,—a double compound of cyanide of nickel and potassium being produced, which is soluble in water. This double cyanide is decomposed by dilute sulphuric acid, with precipitation of cyanide of nickel.

“The double compound of percyanide of cobalt and cyanide of potassium in aqueous solution, is not at all affected by boiling with hydrochloric, sulphuric, or nitric acid.”—GMELIN.

A full description of this reaction is given in Parnell's Elements of Chemical Analysis, page 162.]—*Trans.*



19. *Fluor Spar*,\* which must be freed from its water of crystallization. It serves, when mixed with bisulphate of potash, to detect lithia, and boracic acid, in their various combinations. It is also a re-agent for gypsum.

20. *Gypsum* (Sulphate of Lime).—It must be kept for analyses in an anhydrous state.†

Anhydrous gypsum, in blowpipe analyses, serves as a re-agent for fluor spar. If small quantities of these two substances be placed slightly apart upon charcoal, and heated in the oxidating flame, both melt and run into each other, producing a colourless bead, which becomes opaque upon cooling. If an excess of gypsum is present, the bead will be colourless also. According to Berzelius, the clear glass appears to be a compound of hydrofluoric and sulphuric acids with lime, which, if exposed too long to the oxidating, or too short a time to the reducing flame, is decomposed, sulphurous acid being eliminated.

Gypsum, sulphate of barytes, and sulphate of strontian, fuse with fluor spar; also fluorides of barium and strontium, with gypsum, as well as with fluor spar.

21. *Bone Ashes*.—They are employed in the cupellation of gold and silver. Harkort reduces them to many states of minute division by the processes of *sifting* and *washing*. The bones are burnt until they become perfectly white, and then freed from any carbonaceous matter that may have adhered to them. This being done, they are pulverized in a mortar, and the finer portions separated by a sieve. The remaining powder is then thrown upon a filter, and treated with boiling water, which extracts any soluble matter. The washing, which is then resorted to, is for procuring the bone-ashes of a more uniform degree of fineness. The mass from the filter is mixed with water in any cylindrical glass, allowed to settle

\* [Derived from *fluo*, to flow. It is an excellent flux for ores.]—*Trans.*

† [It is easily obtained free from water, by heating to redness a quantity of it, in a powdered state, in a platinum or porcelain crucible. It occurs abundantly in Derbyshire, Yorkshire, and Saxony; and in various forms. It can be prepared artificially, by adding sulphuric acid to any soluble salt of lime. Its formula is  $\text{CaO}, \text{SO}^3 + 2\text{H O}$ .]—*Trans.*



for a few moments, and then decanted; the coarser powder is deposited at the bottom of the vessel, and the finer passes over suspended in the water. By repeated decantations in this way, sediments are obtained of different degrees of fineness, the last, or that which remains longest floating through the liquid, being the finest. The resulting powders must be kept in separate phials. The coarser ashes are used for the cupellation of rich silver ores, and the finer, for assaying ores in which only a minute quantity of gold or silver is present.

22. *Proof Lead*.—It is employed either in a finely-divided state, or else in small fragments; but it must be entirely free from traces of gold, silver, or copper, as it is used in the quantitative analyses of these metals. The lead from the silver-smelting furnaces will answer both purposes, if properly sifted. If such lead is not easily obtained, the operator can prepare his proof lead in the following manner:—Dissolve sugar of lead (acetate of lead) in a small quantity of hot water, filter, and then add plates of zinc to the solution until the metallic lead is wholly deposited. Dry the resulting lead between folds of blotting-paper, and keep it at hand in a stoppered phial. When this lead is employed in silver and gold cupellations, it need not be accurately weighed, but quantities of it taken (to save time and trouble) according to the standard given in the Lead Measure, previously described, (page 25).

The method just described is the best for obtaining lead perfectly pure, and in a minute state of division.

23. *Common Salt* (Chloride of Sodium).—It is taken either in a powdered or melted state; but its use is very limited. It is principally employed for glazing the earthen crucibles in which quantitative lead assays are performed.

24. *Charcoal Powder*.—This can easily be obtained by triturating pieces of charcoal in a mortar. It is generally employed for roasting or smelting, in quantitative tin or lead examinations.

25. *Graphite* (Plumbago, or Black Lead).—It is used quantitatively, when free from impurities, for roasting earths, minerals, and various products, upon copper. When a good



kind of graphite cannot be obtained, pure anthracite may be substituted. The graphite which is generally met with is so impure, that an operator, to get the best pieces, must pick from a mass of it, those fragments which are unctuous to the touch, and in scales. These parts are then heated in a crucible, and afterwards well pulverized. If this graphite contains over ten per cent. of foreign matter, it must, previously to being used, be digested in nitro-muriatic acid (aqua regia), to free it from iron and other impurities.\*

26. *Tin*.—Tin foil is generally used cut into little slips, half an inch broad, and tightly rolled.

Tin serves for the highest degree of reduction in glass fluxes, where small quantities of oxides of the metals are present, which are capable of being reduced to a lower state of oxide, in which state the results are more convincing. The operator brings the globule, heated in the reducing flame, in contact with the free end of a rolled rod of tin. By this means a small portion of melted tin is deposited upon the flux. The whole is then melted perfectly in the reducing flame. When the tin has been added to the bead (which contains the substance for examination), an operator must not direct the flame upon the glass for too long a time, partly because the tin would become in such a state as to prevent metallic oxides which should only be reduced to the state of protoxide, from manifesting their presence by the peculiar colour imparted to the bead (or pearl), and partly because so much tin would be dissolved, (particularly if phosphates were present,) that the globule, upon cooling, would be quite opaque, thus rendering the analysis of no value.

27. *Iron*.—It is generally kept in the shape of wires, about the size of a strong knitting-needle. It is employed to reduce the phosphates to the state of phosphurets. In this process a white brittle metallic bead is formed, which will be mentioned under Phosphoric Acid. It is employed also in quantitative cupellations.

\* [Plumbago is extensively used for the manufacture of crucibles, especially those required for the purposes of the Mint, as they are very durable, and sustain a violent heat. The composition of the plumbago of Cornwall, according to Saussure, is 96.0 carbon, and 4.0 iron.]—*Trans*.



28. *Silver Wire*.—A small silver wire is useful for ascertaining the presence of hepar (sulphuret of antimony), or soluble sulphurets. Silver which is obtained from the reduction of hornsilver, (chloride of silver,) and beaten into wires, is also necessary in many quantitative gold examinations.

29. *Nitric Acid*, chemically pure.—It is very useful in converting the protoxide of iron, when in solution, into the state of peroxide, and also in the separation of silver from gold.

30. *Hydrochloric Acid*, chemically pure.—It is an excellent test for the presence of ammonia. When ammonia is liberated from any of its combinations, by being heated with soda or potash, it can be detected easily by holding over it a glass rod, dipped in muriatic acid,—dense white fumes of sal ammoniac being produced. It also serves for detecting carbonic acid in its soluble combinations; and lastly, it is useful for dissolving earthy salts, and those silicates melted with soda and borax, whose bases cannot be found with certainty before the blowpipe.

31. *Rectified Sulphuric Acid*.—In blowpipe examinations it serves to increase the action of phosphoric acid in those substances which contain it. It also serves as a re-agent for lime, when this earth is in combination with phosphoric acid, and shews no decided reaction in the blowpipe flame. It may also be employed instead of the bisulphate of potash, to separate barytes and strontian from other earths.

32. *Acetic Acid*, free from Sulphuric Acid.—This is ascertained by its giving no precipitate with acetate of barytes. Acetic acid is not often employed in blowpipe analyses. It is used only for the examination of compound substances, containing traces of chromium and phosphoric acid, as will be seen hereafter, under Chromium.\*

\* [Acetic acid may be easily prepared for chemical purposes, by distilling the acetate of soda, which is made on a large scale in purifying the rough wood vinegar, with sulphuric acid. The acid which distils over, from its secondary action on the sulphuric acid, contains traces of sulphurous acid; in order to get rid of this, it is rectified over peroxide of lead, with which the sulphuric acid unites, forming a sulphate of lead. The formula of Acetic Acid, according to Berzelius, is  $C^4H^4O^4$ .]—*Trans.*



33. *Tartaric Acid*, in a crystallized state.—It is useful with sulphuret of ammonium, for separating yttria and zirconia from iron. It also detects small quantities of arsenic in metallic combinations; and it is also employed in quantitative cupellations.

34. *Caustic Potash*, in solution.—An operator, when travelling, may carry it in a solid state, and dissolve it when it is required. It must, when dissolved, be colourless, scentless, and highly caustic to the taste. It is used in such blowpipe experiments as only produce a proper result when tested in the humid way; as for instance, separating alumina and glucina from the protoxide of manganese, chromium, and the peroxide of iron.

35. *Caustic Ammonia*, free from carbonic acid.—If carbonic acid is not present, there is no precipitate on the addition of chloride of calcium. It is employed with the blowpipe for detecting the earths in combination, and for separating alumina, glucina, yttria, peroxide of iron, and oxide of chromium, &c., from lime, magnesia, and the protoxide of manganese, in a state of solution.

36. *Sulphuret of Ammonium*, (Hydrosulphate of Ammonia.) This re-agent is prepared by passing a stream of sulphuretted hydrogen through caustic ammonia, diluted with an equal volume of water, until no precipitate is formed on the addition of a salt of magnesia. This compound only serves for separating the protoxides of manganese and cobalt from magnesia, and the oxide of iron from yttria.

37. *Absolute Alcohol*.—Its use with the blowpipe is limited. It is however not to be rejected, as it serves for detecting potash when combined with silicic acid, which before the blowpipe alone, gives the same reaction as soda or lithia. It also enables an operator to separate and distinguish barytes from strontian; and, lastly, it is a proof that arsenic, or arsenic acid, may exist in such combinations as do not give decided results in the dry way.\*

\* [Absolute alcohol is obtained by distilling rectified spirit with chloride of calcium at a moderate heat in a retort. The water of the spirit combines with the chloride of calcium, and pure alcohol comes over. This process should be conducted in a water-bath.]—*Trans.*



38. *Re-agent Papers*:—Blue litmus; reddened litmus; Brazil wood; and turmeric papers.

(a) *Blue litmus paper*.—This is prepared by immersing slips of fine unsized paper in a decoction of litmus, for some time. When complete, these slips are suspended upon a fine thread to dry. Blue litmus serves for detecting free acids in solution; its blue colour being changed into red.

(b) *Reddened litmus paper*.—A decoction of blue litmus is treated with sulphuric acid, until its colour has assumed a distinct red tint. The slips of paper which are then passed through this mixture should be distinctly red when dry. It serves for the detection of free alkalies, its colour being restored to a blue.

(c) *Brazil wood paper*.—If brazil wood be boiled in water for a sufficient time, it communicates a fine red colour to the fluid. It must then be filtered, and slips of paper allowed to digest in it, in a manner similar to what has been described. It is an excellent re-agent for hydro-fluoric acid, being tinged straw-yellow, when immersed in a very dilute solution.

(d) *Turmeric Paper*.\*

The alkaline and acid re-agents, &c., must be kept in glass bottles, with good ground stoppers, and the test papers, which should be cut into fine slips, kept in small paper boxes.†

\*[Turmeric root should be reduced to powder, and then treated with boiling alcohol; the decoction must be filtered, and the paper treated in the manner just described to imbibe the colour. When the turmeric paper is of a bright yellow colour, the change which is produced by the alkalies is very characteristic;—it becomes dark brown.]—*Trans*.

†[*Distilled Water* must be used in the preparation of the re-agents; in fact, it ought always to be employed in chemical manipulations, to prevent inaccuracy.]—*Trans*.



## DESCRIPTION OF A CONVENIENT AND USEFUL BLOWING APPARATUS,

*For Quantitative Analyses with the Blowpipe.*

From the many analyses I have made with the aid of the Blowpipe, to estimate the quantity of metals contained in minerals and ores, I am convinced that a number of successive quantitative examinations with it would weary even an experienced operator. This evil may however be easily remedied, provided the operator is always employed at the same place, with a conveniently-arranged blowing apparatus.

Such an apparatus should possess the following qualifications:—

1. It should be perfectly air-tight, under the greatest pressure used in blowpipe experiments.
2. It should possess an orifice fine enough to afford an equal, uninterrupted, and sufficiently sharp stream of air.
3. That the pressure may be increased or diminished at pleasure.
4. That a long flexible tube be employed for conducting the stream of air, which is not in connection with those parts of the apparatus which move up and down.
5. That it occupies as little room as possible.
6. And that, during the blowing, it is not required to fill it with air by means of a treadle, or other contrivance requiring physical exertion.

If such an apparatus be considered essentially necessary, the chief object of the application of the blowpipe is lost; on this account it should be premised, in the first instance, that this apparatus should be employed only by such as are well acquainted with the use of the mouth-blowpipe; and even then only as an auxiliary, or to preserve health in case inconvenience to the chest should arise from blowing;—because,

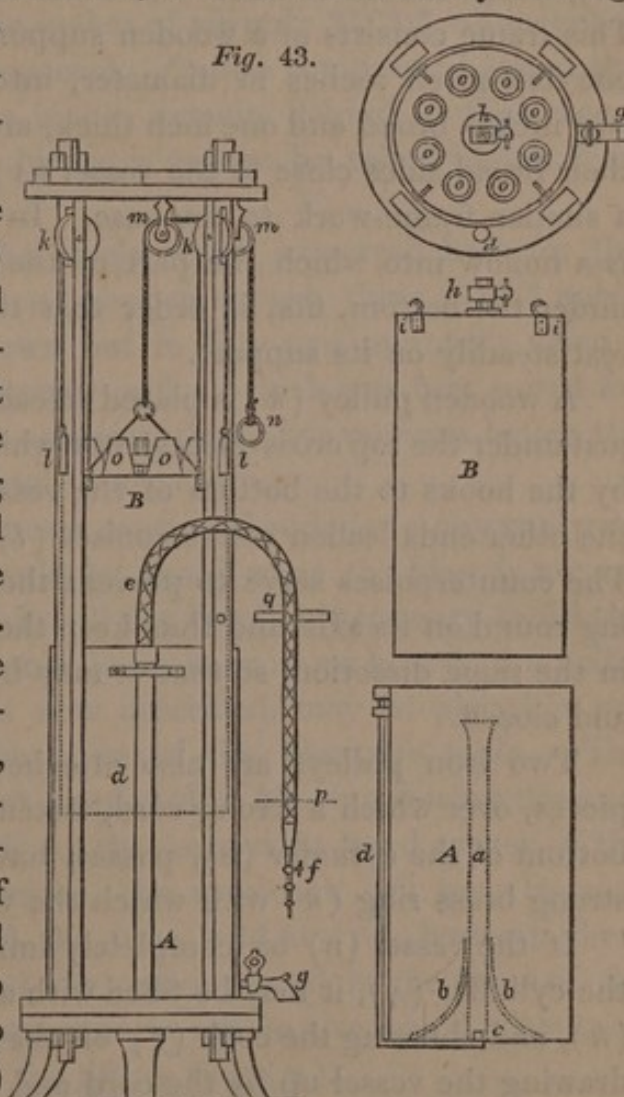


if a person unaccustomed to the use of the ordinary blowpipe should be sometimes obliged to employ it when travelling, his results would always be uncertain.

My Blowing Apparatus, (which was constructed according to a model of a gasometer which Lampadius the mining commissioner had the kindness to lend me out of the Royal Laboratory, and which, with some slight changes, is similar to that proposed by Harkort,) possesses all the above qualifications.

It consists of two cylindrical vessels of strong sheet zinc, (*Fig. 43*, A and B). (A) is 2 ft. 2 in. high, and 1 ft. 2 in. in diameter; the inner (B) is 2 ft. 1 in. high, and 1 ft. 1 in. in diameter. Instead of zinc, copper, or tinned iron, may be employed. The last, however, does not last nearly so long as zinc, as it is liable to oxidize unless well protected by paint or varnish.

Through the middle of (A) a brass tube (*a*) with a funnel-shaped mouth, rises perpendicularly 1 ft. 11 in.; it is secured in its place by two slips of strong tin plate (*b b*) soldered to its sides and to the bottom of the vessel. To the end of this tube another, of a less diameter (*c*) is attached, which passes externally underneath the bottom and up the side of the cylinder (A), and is fastened at the top with a slip of zinc. To the top of the tube (*c*)





a strong caoutchouc tube (*e*), three feet long, and half-an-inch broad, is fastened, having at its extremity a brass stop-cock (*f*); it is to this that the various blowpipe-nozles are affixed. A brass stop-cock (*g*) is also placed near the bottom of the vessel (*A*), for the purpose of drawing off the water.

To the bottom of (*B*), which should be strengthened internally with a double cross of tinned iron, is attached a brass stop-cock (*h*), having an orifice of half-an-inch in diameter for admitting air. Four hooks are also attached to the bottom, to which the counterpoises for steadying the apparatus are fastened.

Both vessels are placed in a strong frame of hard wood, six feet high; the smaller (*B*) being inverted in the greater (*A*), which should be filled with water to about sixteen inches. This frame consists of a wooden support, six inches high and one foot seven inches in diameter, into which four uprights, two inches broad and one inch thick, are mortised, lying with their broad sides close to the vessel (*A*), and joined above to a similar frame-work to the base. In the base of the frame is a hollow into which the part of the tube (*c*), that passes under the bottom, fits, in order that the cylinder (*A*) might rest steadily on its support.

A wooden pulley (*k*) is placed in each of the four uprights, just under the top cross-pieces, over which cords pass attached by the hooks to the bottom of the vessel (*B*), and having on the other ends leaden counterpoises (*l*) each a pound weight. The counterpoises serve to prevent the vessel (*B*) from turning round on its axis, and thus keep the stop-cock (*h*) always in the same direction, so that it may be conveniently opened and closed.

Two iron pulleys are also attached to the upper cross-pieces, over which a strong cord, fastened to the centre of the bottom of the cylinder (*B*), passes, having on its other end a strong brass ring (*n*) with which the vessel (*B*) is drawn up.

If the vessel (*B*) be completely immersed in the water of the cylinder (*A*), it may be filled with air by opening the cock (*h*), and shutting the cock (*f*) on the caoutchouc tube, then drawing the vessel up by the cord and ring above-mentioned,



until it nearly reaches the surface of the water, and shutting the cock (*h*) through which the air entered.

The air enclosed in this manner, which measures 3317.5 cubic inches, can be compressed by means of eight leaden weights (*o*), of four pounds weight each, in the form of the frustrum of a cone, so as to produce, through a blowpipe-nozle placed on the stop-cock (*f*), an equal and sufficiently strong stream of air for all blowpipe experiments. The nozzle must, however, have a larger diameter than those employed with the ordinary blowpipe. The proper diameter cannot be well described here; but the operator soon discovers the most convenient to use.

If the vessel (*A*) be filled with water to sixteen inches, the vessel (*B*) raised nearly to the surface, and the weights laid on, of the 3317.5 cubic inches of air only 2123.3 cubic inches escape through the stop-cock (*f*) in a compressed state; the remaining 1194 cubic inches remain behind in their usual state, as the level of the water cannot be raised higher than sixteen inches.

When I employed a nozzle which answered best for the examination of silver and copper assays, these 2123.3 cubic inches of air were blown out in fifty-two minutes; when I took a nozzle with a larger orifice, which was best suited for lead and tin assays, it occupied thirty-five minutes before the vessel (*B*) had completely sunk down.

As the blowing is never continued so long in any analyses, the stop-cock (*f*) should be closed when the blast is not required. In this way, it will not be often necessary to fill the apparatus more than once in an hour or an hour-and-a-half.

The apparatus, as now described, may be placed in the laboratory at the right side of the blowpipe table. The flexible tube, when not employed in blowing during the analysis, may be laid aside at the near right hand corner of the table; when it is required, the stop-cock (*f*) may be conveniently opened, and the tube held at (*p*), between three fingers, like a pen, and the orifice placed in the flame of the lamp. When the apparatus is not in use, the flexible tube may be suspended to the lifting ring (*n*).



This apparatus would also serve to form such small glass tubes as may be useful in the examination of minerals, &c., before the blowpipe, by using a lamp with a thick wick. The compression of the air being also diminished by the removal of some of the weights, it would be also necessary to employ a nozzle with a larger orifice than that used in analytical operations. In this case both hands may be left disengaged, by fixing the stop-cock (*f*) on a small support, in such a manner that the current of air can be directed on the flame in a proper manner.

In conclusion, I will just state in what manner the vessel (*A*) is filled with water. The vessel (*B*) being deprived of its weights, is drawn up as far as the frame admits, or till the stop-cock (*h*) comes in contact with the central iron pulley (*m*); it is kept in this position by two wooden pins, which are inserted into two corresponding holes in the front uprights; the funnel-shaped orifice of the upright tube (*A*) is then covered with a glass, and water poured into (*A*) until it stands at sixteen inches; it should not be filled higher than this, as the pressure of the air in the upper vessel (*B*) would cause the excess to flow over.

When the vessel is full, the glass is removed, the wooden pins withdrawn, the vessel (*B*) allowed to descend, and the weight placed upon it. If the vessel is made of well-japanned sheet-iron, cold water may be used to fill it; but if of zinc or copper, or unglazed iron, hot water should be employed; as otherwise, in a warm room, aqueous vapour would be condensed on its external surface, producing oxidation. Although this oxidation in itself would matter nothing, the vessels would lose much in external appearance,—the zinc becoming white, and the copper, green. By adding one-fourth of boiling water, this unseemly appearance may be obviated.

# QUALITATIVE ANALYSIS

## WITH THE ELEMENTS

### SECTION II.



THE HISTORY OF THE  
CITY OF BOSTON  
FROM THE FIRST SETTLEMENT  
TO THE PRESENT TIME  
IN TWO VOLUMES  
BY NATHANIEL BENTLEY  
OF THE BARRISTER AT LAW  
IN THE SUPREME COURT OF JUDICATURE  
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## QUALITATIVE ANALYSES

### WITH THE BLOWPIPE.

It is well known that the miner frequently encounters substances, which, from their exterior appearances, he may have considered to belong to some certain class of minerals, but which, by subsequent chemical analysis, are proved to be of an entirely different constitution. It happens that in the earthy gangues of the more frequently occurring ores, minerals of great scarcity and value exist, but in such a minutely disseminated state as to be scarcely discernible with the aid of the microscope. Again, it is very difficult for the smelter to know the nature of the ingredients given him to assay. The mixed mass is given to him ready for furnacing, and except it be from previous experience, he is quite unable to judge, by the appearance of the mass, the degree of heat necessary for its fusion; and difficulties may again stand in his way, with respect to the further working of his furnace products, from the presence of substances which he either does not know to be contained therein at all, or which he may consider to exist only to a very small extent. A means is wanted by which the miner and smelter can obtain the necessary information with respect to the contents of minerals, ores, and furnace products, and as it seldom happens that there is a laboratory within such a distance as to be available upon every emergency, the blowpipe affords the only means whereby this knowledge may be obtained with certainty.

By means of the blowpipe, the substances which comprize a mixed mass can in all cases be determined. It is therefore my intention now to treat of the Blowpipe as the agent for Qualitative Analyses. I will describe the qualitative examin-



ation of alkalies, earths, metals, their oxides, and the non-metallic bodies, so far as they have come under my own observation; and I shall attach to each the name, some instruction as to its nature, whence derived, and the mineralogical or chemical formula; and I will also give general rules, in as short and concise terms as possible, with respect to the behaviour of earths, metallic oxides, &c., before the blowpipe, alone, or with borax, microcosmic salt, soda, and a solution of cobalt; and also a description of the different appearances presented by the sublimates of some metals, when heated in the blowpipe-flame upon charcoal.

## I. GENERAL RULES FOR QUALITATIVE BLOWPIPE EXAMINATIONS.

¶ *A General Rules by which the ingredients of Minerals, &c. may, for the most part, be detected, when heated alone or with re-agents in the Blowpipe flame.*

It is always advisable, before commencing an analysis, to spread a sheet of clean white paper upon the table, in the middle of which the blowpipe-lamp is placed; by this precaution, any substance falling from the charcoal or wire can be re-obtained, and the investigation continued. If it is of any consequence that the table be preserved from the hot falling assays, three or four sheets must be employed. The presence of foreign matter upon the paper must be particularly guarded against, otherwise on a bead falling and coming in contact with it, a false result would necessarily follow. Clean paper should be employed for each analysis.

With respect to the quantity of the substance which should be employed, when seeking its behaviour alone with the re-agents before the blowpipe, it is always advisable that too much be not taken. If, for instance, an analyst wishes to examine the solubility of an earth, mineral, &c., with borax or microcosmic salt, on the platinum wire, a piece about the size of a mustard seed is taken. In cases where metallic



oxides are present, and the colour only of the bead or glass is sought, considerably more is used, and in a pulverized state; but if it is the reduction of a metal by means of soda upon charcoal, a still larger quantity is employed, and also in a powdered state. Experience, however, soon teaches the proportions to be used in the different occurring cases. If too much substance be employed, it inevitably follows that uncertain results are the consequence.

In examining the relation of a substance by itself, or with fluxes before the blowpipe, the operator proceeds as follows:—

( $\alpha$ ) A part of the substance to be examined is gradually heated to redness, in a small glass retort, over the spirit-lamp. During this process, all phenomena must be observed,—if decrepitation takes place; if water or any other volatile body be eliminated, and its odour. The water condensed in the cooler parts of the retort, should be tested by litmus or brazil wood paper, to ascertain if it gives an acid or alkaline reaction.

( $\beta$ ) Another portion is heated gently upon charcoal before the blowpipe-flame: the odour will indicate volatilized acids, arsenic, selenium, or sulphur. The difference of smell should be noticed when the sample is heated in the oxidating as well as in the reducing flame, since selenium and sulphur are best detected in the former, and arsenic in the latter. It should be noticed if the charcoal has become coated with a sublimate, and if near or at a distance from the assay; of what colour it is, and if it remains the same when cold as when hot. If the substance under examination be an earth, it must be heated strongly for some time, and then removed from the charcoal to litmus paper, and moistened with a few drops of distilled water; if an alkaline reaction occurs, the substance contains a carbonate of one of the alkalies; for these occur in nature, *Witherite*, *Strontianite*, *Calcareous spar*, *Magnesite*, *Bitter-spar*, &c., and become caustic with strong heating, and exhibit alkaline properties.



( $\gamma$ ) If the substance contains volatile metals, metallic oxides, sulphur, &c., it is examined in a tube which is open at both ends. The substance is placed within the tube, at a short distance from one end, which is then heated with the spirit-lamp, and afterwards with the blowpipe flame. The tube is held in an inclined position. The more perpendicular the position, the stronger the current of air. It is very easy to regulate the rapidity of the draught according to the rate of oxidation that may be required.

In this treatment, many substances are sublimed, which were not, when heated in the retort, because the access of air was comparatively small. The oxygen of the air is absorbed, forming volatile acids or metallic oxides; some escape as gases, and may easily be recognized by their smell,—sulphur, for instance, exhaling sulphurous acid. Others are deposited in the upper and cooler parts of the tube, at greater or less distances from the heated body, according to their degrees of volatility; in the cases of arsenic and antimony, for example, the former is given off as arsenious acid, and the latter as oxide of antimony.

( $\delta$ ) In examining a substance with regard to its *fusibility*, a part is placed in a small cavity, formed in a piece of charcoal, and heated for a length of time in a strong oxidating flame. The more easily-reduced metals and metallic oxides are acted upon in this experiment.

If the substance be an earth or silicate, small pieces of it are broken off with a hammer, and a piece chosen with a sharp point or side. This is held between the platinum points of a pair of forceps, and a strong blowpipe flame allowed to play on the sharp extremity for some time. It will at once be seen if the substance is fusible or not. If infusible, the sharp point or edge will remain unaltered; if fusible, it becomes rounded; and if entirely fusible, it will melt into a round bead. These appearances should be examined through a strong magnifying glass. When a splinter is heated in the inner flame, the outer may become coloured. For instance, if the mineral contains potash or soda; with potash, the outer



flame will be violet, with soda, yellow. If lithia, without soda, it will be tinged red. If the three are present, the yellow flame predominates.

Berzelius treats those minerals which are found to be very infusible, in the following manner. The substance is levigated in an agate mortar, with water. The menstruum, with the finely-divided particles floating through it, is dropped on the charcoal, and heated by the oxidating flame, to evaporate the water. This operation is repeated until the charcoal becomes covered with a coating of the fine particles, which are carefully collected and held in the points of the forceps, and then subjected to a powerful oxidating flame. For the most part, some change takes place, owing to the extreme divisibility of the mineral, showing that the substance is not altogether infusible. In this case, the Microscope will be in great requisition, in order to discern the action which may have been produced.

When the substance occurs in a granulated state (such as sand), one of the grains should be placed on the charcoal; but as this is so apt to be lost, the better way is to knead the substance with water into a paste, which is then placed on the charcoal, dried by the oxidating flame, and examined as to its fusibility. This manner should always be resorted to, when treating substances that are in a minute state of division.

The heat obtained by blowing through a blowpipe (that is, the small hand blowpipe,) is limited, so that there are some bodies which are never fused before it; silica and alumina, for instance, in however finely-divided a state. There are many minerals which melt and tumefy, and thereby give a blistered glass, which, on account of the air-bubbles, appears opaque. The intumescence and blistering appears at a certain degree of heat, that is, when all the water is expelled. According to Berzelius, the "cauliflower" appearance which sometimes takes place on heating, proceeds from a change in the combination of the ingredients and their relative positions; but the tumefaction and blistering which occurs in igneous fusion, he considers to arise only from the escape of some ingredients in the form of a gas, although it often hap-



pens that this ingredient is not discriminated in an analysis of the body. This occurs for the most part with silicate of lime or alkalies with alumina.

The examination of the comparative fusibility of minerals is of essential importance, as many which consist principally of earths, and contain very little of the true metallic oxides, can readily be distinguished by this means. Hence it is that the list given by Henry Rose in all the editions of his *Manual of Analytical Chemistry*,\* of a considerable number of minerals arranged according to their different degrees of fusibility, is of great interest.

Of the minerals which occur most frequently, the following are, when heated between the platinum points of a forceps in a strong blowpipe-flame, perfectly infusible:—*Quartz, Corundum, Spinel, Zeylanite (Pleonaste), Automolite (Gahnite), Olivine, Cerite, Zircon, Disthène (Cyanite), Leucite, Talc, Gehlenite, Anthophyllite, Staurolite, Allophane, Kymophane, Gadolinite*. Those that phosphoresce on being heated: *Rutile, Titaneisen* (or Titaniferous Oxydulated Iron), *Tantalite, Turquoise (Calaite), Chondrodite, Topaz*. Very difficult, or only on the edges fusible, are particularly the following:—*Adularite, Tetartine (Albite), Petalite, Labradorite, Anorthite, Tabular Spar, Meerschaum, Speckstein, Serpentine, Epidote*. Those which tumefy on the first application of heat:—*Dichroite* (some varieties moderately fusible), *Beryl (Emerald), Euclase, Titanite, Sodalite, Schwerstein (Tungstate of Lime), Heavy spar (Sulphate of Barytes), Celestine, Gypsum (Sulphate of Lime), Apatite, Fluor-spar*. The fusible are the *Zeolites*, the most of which present intumescence when heat is first applied, *Oligoklas (Soda Spodumene), Spodumene*, (which also tumefies), *Meionite, Elaolite, Nepheline, Amphibolite*, the greater part of which effervesce during fusion. The *Pyroxenes* (of which those containing large quantities of magnesia, are with difficulty fusible), *Vesuvian, Idocrase* (which tumefies on melting), *Granite, Orthite* (which boils while fusing), *Wolfram, Boracite, Datholite, Botryolite, Tourmaline* and *Axinite* (which swell up when melted). *Amblygonite, Lazurstein, Hauyn, Nosin, Eudyalite, and Pyrosmalite*.

\* Translated by Mr. John Griffin.



( $\epsilon$ ) The behaviour of the substances with fluxes should be examined. The fluxes are BORAX, MICROCOSMIC SALT, and SODA.

§ 1. TREATMENT OF THE SUBSTANCE WITH BORAX.

This can be performed either on a platinum wire or on charcoal. If the substance contains only earths and metallic oxides, it should be first examined on the wire. The following is the method of procedure: the loop of the platinum wire is first heated to redness in the blowpipe flame, then rapidly dipped into the borax, and the adhering particles fused in the oxidating flame. As it seldom happens that a sufficient quantity of Borax adheres the first time, the process should be repeated once or twice, until a bead is formed large enough to fill the loop completely. The bead generally remains so firmly fixed in the loop, that it cannot be removed without force. If the wire is quite clean the bead will be perfectly colourless, both when hot and cold; if it should happen to be slightly tinged, which is easily perceived by holding it between the eye and the light, it must be removed from the platinum wire. The separation of the glass bead from the wire is easily effected by heating the bead strongly, and then quickly removing it from the flame, placing it over a porcelain basin, and striking the wire against the rim; it immediately falls into the basin and solidifies. The quicker this operation is performed, and the firmer the wire is held, the more perfectly is the separation of the bead effected.

The borax bead melted on the platinum loop, must, when cold, be moistened on one side with the tongue, which side should then be brought in contact with the substance to be tested, in the state of fine grains or of very fine powder, and the adhering particles melted together with the borax in the oxidating flame. In general, earthy substances are better examined when a single particle is at first dissolved, because when the substance is employed in the state of powder, the undecomposed part cannot be well distinguished from the separated and insoluble portions which it may contain. With borax this is not so often the case as with microcosmic salt, as, with the latter silicic acid, is separated.



When the oxidating flame is playing upon the assay, the operator must observe if it dissolve quickly or slowly; whether any gas is generated; if, when entirely dissolved, the glass when held between the eye and the light is coloured; and whether the colour changes or remains the same on cooling.

There are some bodies which, with borax in certain proportions, give a clear glass when hot or cold, but in the reduction, or more particularly in a quick intermittent flame, become opaque, milk-white, or opalescent, and, in some cases, even coloured. This re-action is generally observed in such bodies as form clear glasses while melting, but are enamelled on cooling. Such is the case with the alkaline earths, with yttria, glucina, zirconia, oxide of cerium, tantalic and titan-ic acids, and with some others, as silica, alumina, peroxide of iron, oxide of manganese, &c. The presence of silica hinders the phenomenon of the glass becoming enamelled on cooling. The same occurs with those silicates which give a transparent glass only when the assay is perfectly saturated,—over-saturated, the mass is, on cooling, opaque. When a borax bead, containing a body in certain proportions, becomes opaque in a quick intermitting flame, it is said to become *turbidly streaked*.

Where metallic oxides are present, and the glass may be coloured, it is necessary to observe that too much of the sample be not taken; otherwise, the colour will be so deep as to be indistinguishable. When a glass is so dark that the colour cannot be decided upon, it must be pressed while hot between the points of a forceps, or drawn out into long thin threads; the colour can then be more readily recognized. Many judge of the colour best with the naked eye; others with a magnifying glass: but in the latter case it must be particularly noticed, that the glasses of the magnifier are not tinged, else false determinations will be the result.

When the colour which a substance gives with borax before the oxidating flame has been remarked, and all the phenomena carefully observed, the glass must be operated upon in the reducing flame, and in such a manner that no



carbon be deposited on its surface. If there appear to be metallic oxides dissolved in the glass, which are not reduced by the borax, as for example, oxides of cerium, manganese, cobalt, iron, uranium, chromium, titanite and tungstic acids, &c., the operation must be effected on the platinum wire: but if metallic oxides are present, which are readily reduced to the metallic state, as the oxides of zinc, nickel, cadmium, lead, bismuth, copper, silver, antimony, &c., the reduction must be produced on charcoal; otherwise, the metal would amalgamate with the platinum.

When a trial is made upon the platinum wire, in the two flames, the change of colour will be readily recognized, when the glass is either hot or cold, but if the assay be heated on charcoal, the glass must be removed from the wire as previously mentioned.

The bead is placed in a small cavity formed in the charcoal, and then exposed to the reducing flame. After continuing the flame for two minutes or so, the glass is quickly removed and pressed between the broad ends of the forceps, in order that the colour may be more accurately determined. Pressing the glass in this manner also possesses this advantage, that the protoxides, if there be any present, are not so liable to become peroxidized, as when left to cool gradually upon the charcoal.

If the glass contains an easily-reducible metallic oxide, it often occurs that the charcoal becomes coated with a sublimate at a certain distance from the assay; but this appearance takes place only when the oxides of selenium, tellurium, arsenic, antimony, bismuth, cadmium, zinc, tin, and lead, are in excess.

When no colour is given with the reducing flame on the wire, an examination should be made with borax on charcoal, in the following manner. The hot glass is suddenly shook on the charcoal, and heated with a small quantity of pure metallic tin, and the mixture then strongly smelted in the reducing flame. The tin, having a great affinity for oxygen, abstracts part from the metallic oxide contained in the assay, and dissolves without tinging the glass, while the metallic



oxide, which is converted into a protoxide, gives a characteristic hue to the glass on cooling. This method is generally resorted to when examining for traces of copper or iron.

When the behaviour of a substance containing a sulphuret or arseniuret is to be ascertained by heating with borax, it is best to sublime, first, most of the sulphur or arsenic, in an open tube, and then to heat on charcoal, with the necessary proportion of borax (and tin if requisite) in both flames, and observe all the striking characteristics.\*

## § 2. TREATMENT OF THE SUBSTANCE WITH MICROCOSMIC SALT.

As with Borax, it can be treated partly on the platinum wire, and partly on charcoal. If the substance contains earthy ingredients, or only difficultly reducible metallic oxides, the operation is to be conducted on platinum wire, in exactly the same manner as with borax; but if it likewise comprises sulphurets or arseniurets, it must (as with borax) be treated on charcoal. As microcosmic salt gives a very fluid bead when heated, a few additional bends should be made on the platinum wire, when this salt is used, to prevent the melted drop from falling; on charcoal, however, enough of the salt can be taken at once, as there are no obstacles in the way. This flux is better for many metallic oxides than borax, as the colours are often better ascertained when a sufficient quantity of the substance is dissolved. It also separates the acids;—the volatile are driven off, and the fixed remain, and divide the bases either with the phosphoric acid, or remain uncombined, and float about in the molten mass. With the silicates, this is particularly the case: the silicic acid is separated, and flows through the glass as a gelatinous particle. Silicates, when containing bases that, by themselves, produce in vitreous fluxes an opaque glass on cooling, give, in combination with borax, a clear glass; but an opalescent, with microcosmic salt.

\* [When sublimation-tubes are employed, they should be free from *lead*, for arsenic, if heated in a tube containing this metal, is readily decomposed.

The best kinds of glass tubes for blowpipe experiments are obtained from Germany.]—*Translator*.



## § 3. TREATMENT OF THE SUBSTANCE WITH SODA.

When assaying a substance with soda, it is necessary to learn if it consists of earthy ingredients only, or of acids, free or in combination; whether it is soluble; whether it contains metallic oxides which give a reduced metal; or if sulphurets or arseniurets are present, &c. The numerous examinations will be fully treated of, together with the pyrognostic effects produced upon metallic oxides, when heated in the blowpipe-flame with Soda.

*( a. ) Fusibility of a Substance with Soda.*

A great number of bodies possess the property of uniting with soda, and forming combinations partly fusible and partly infusible. To the fusible, but few, such as Silica, and the metallic oxides of titanic, tungstic, and molybdic acids, &c., belong; they are, however, more or less absorbed by the charcoal, if not in combination with silica.

In testing the fusibility of a substance with soda, the following is the mode of procedure:—

The substance, when in powder, is kneaded into a paste with soda, a moistened spatula being used for the purpose; but when in a granular state, one of the grains is taken, and covered with the soda paste; in both cases a portion is placed on charcoal, and heated till all water is expelled; the mass is then strongly ignited in the oxidating flame. The soda will first be absorbed by the charcoal; but when a sufficient quantity of the assay dissolves, it exudes, effervescence takes place, and a bead is formed. The treated substance, if insoluble in the soda, will, if decomposed, tumefy gradually, and change its appearance, without forming a bead. The operator should add fresh supplies of soda, and renew the blast until he arrives at a satisfactory conclusion. When, to a substance soluble in the soda, too little of the flux has been added, a part will remain opaque, surrounded by a clear glass; if too much soda has been supplied, the glass will be quite opaque; it is therefore always advisable that the soda be supplied in small doses at first, and more added as the different changes ensue. If the glass assumes a yellow, red, or brown colour, sulphur or sulphuric acid has been present; the smallest trace of these



substances gives a decided re-action. When the substance has been examined on charcoal, it should be treated on the platinum wire in the oxidating flame; if it expands and gives a bluish-green glass in cooling, manganate of soda has been formed, and therefore some oxide of manganese has been present. When the substance contains silica and oxide of cobalt, silicate of soda is formed, which assumes the characteristic cobalt-blue colour. When the previous re-action has been decided upon, and an operator wishes to ascertain the presence of an alkali or earth, the substance must be treated upon platinum foil, with soda. If the glass during the fusion is clear and limpid, only an alkali is contained therein, but if something is deposited, an earth is present. The alkali will also tinge the flame, which must be remarked.

( b. ) *Reduction of Metallic Oxides by means of Soda.*

In this process, metallic oxides, existing in only very minute quantities in minerals, &c., may be more readily found than by the moist way.

There are some metals which, without the addition of soda, are reduced on charcoal in the de-oxydizing flame, and thus determined; but if they are mixed or in combination with other substances irreducible, it is not only difficult, but, in many cases, altogether impossible, to accomplish the reduction sufficiently, in order to arrive at satisfactory results. For instance, when a quantity of native or artificial oxide of tin is treated in the reducing flame upon charcoal, it is possible, with great exertion and fatigue, to obtain a metallic globule; but when soda is used as an auxiliary, the reduction takes place readily.

When a metallic oxide is in combination with a non-reducible body, which prevents its giving a characteristic re-action with soda, it must be treated as Berzelius (according to Gahn), directs; which is as follows:—

Knead the powdered sample with moist soda, into a paste, then place the mass on charcoal, and direct upon it a strong reducing flame. The soda will at first be absorbed; but more must be added, and the heat continued as long as any portion of the assay remains on the surface of the charcoal. The first portion of soda serves for collecting the me-



tallic contents, while the latter is for completing the reduction of the remaining unreduced metallic oxide. A few drops of water are now added to extinguish the ignited parts, and then the whole of the charcoal upon which the assay rested, is cut out with a knife, and put into an agate mortar, care being taken to lose nothing in the transfer. The mass is mixed with water, and ground to a fine powder. The metal therefore, from its greater specific gravity, falls immediately to the bottom. Repeated levigations and decantations are then resorted to, till the whole of the pulverized charcoal is removed. When the charcoal is entirely washed away, the metal, if any had been in the assay, will be found at the bottom of the mortar. If it is an infusible or a brittle metal, it appears in the form of a metallic powder; if a malleable one, it produces flat shining plates.

By this method, one-half per cent. of *Tin*, and even a much smaller quantity of *Copper*, may be very easily detected. If, however, a number of metallic bodies are contained in the same substance, they will be reduced, together, to a metallic alloy. Some, notwithstanding, are obtained singly, as copper and iron, which give reguli distinct from other metals.

The metals which can be reduced by this means, besides the noble ones, are,—Molybdenum, Tungsten, Antimony, Tellurium, Lead, Zinc, Tin, Nickel, Copper, Bismuth, Cobalt, and Iron. However, there are a few of these, which are partly or altogether volatile, and coat the charcoal with sublimated oxides; namely, Antimony, Tellurium, Bismuth, Lead, and Zinc. Some other metals, as Arsenic, Cadmium, and Mercury, are also reducible, but being extremely volatile, must be heated in glass tubes, in order to obtain their sublimates.

If, in these experiments, a metallic button, containing a number of metals, is obtained, the alloy must be examined on charcoal, with borax or microcosmic salt, as will be hereafter adverted to when treating of different qualitative metallic assayings.

When the substance under examination is a sulphuret or arseniuret, or contains either sulphur or arsenic, these bodies



must in the first place be driven off, and the metal oxidized, before the reduction-test can be made. The process is conducted as follows: the sample is ground to an impalpable powder, and pressed into a shallow cavity in the charcoal with the spatula, then acted upon by the blowpipe flame. The oxidating flame is to play upon the assay first, by which means part of the sulphur is given off as sulphurous acid, and the metal oxidized, which combines with the sulphuric and arsenic acids, also formed in the process. When no more sulphurous acid is evolved, which is readily recognized by its characteristic odour, the reducing flame must be brought into action, whereby all sulphuric and arsenic acid is reduced, and the greater part of the metallic arsenic is expelled. When the smell of arsenic is no longer discernible, the oxidating flame is again used, &c., until decomposition is so far accomplished as to satisfy the operator. The assay is now turned over on the charcoal, and the other side treated in the same manner. The mass is then removed, again powdered, and once more treated on the charcoal with the oxidating and reducing flames. The sulphur is by this means entirely got rid of, but arsenic often remains as arsenic acid, in combination with such oxides as those of cobalt or nickel, and can only be removed with great difficulty. When the substance contains a large proportion of arsenic, which might be dangerous if it were allowed to escape in the room, previously to roasting, it should be treated in a tube, whereby the greater part of the arsenic sublimes, and condenses in the upper part.

If the substance contains neither cobalt or nickel, but another metal with arsenic; the metal can be obtained by the reduction of the roasted sample with soda, perfectly free from the arsenic; but if cobalt or nickel be present, the reduced metal will always be contaminated with arsenic, and must therefore be further treated *with borax* on charcoal. In this reduction experiment, an operator must allow an uninterrupted strong flame to play upon the assay. In separating the charcoal which has become impregnated with reduced metal, great care must be exercised in the removal, and also in the levigation and decantation; and, lastly, the metal



obtained in grains, plates, or fine powder, must not only be inspected with the naked eye, but also through a magnifying glass.

When the substance is an earth, containing no colouring metallic oxides, it is to be treated with a solution of cobalt. A piece is selected, of sufficient porosity to allow the solution of cobalt to permeate, and a few drops added. It is then subjected, in the points of the forceps, to a moderate oxidating flame; during this process, the colour will assume a characteristic aspect:—if blue, pure alumina is indicated; if rose-red, magnesia. If the latter colour is discernible, the assay must be fused, because, when magnesia is present, the rose-red tint is better brought out in a strong flame. Minerals containing lime, or an alkali, give a blue tinge, when *strongly* heated with cobalt; but alumina gives it by a moderate heat, and can therefore be readily distinguished.

Those crystalline rocks which will not admit of the solution permeating, must be reduced to a fine powder in an agate mortar; then kneaded into a paste with water, and spread on the charcoal. The solution of cobalt is now added, and the mass subjected to an incipient red heat. If decomposition does not take place, the colour imparted, (blue, red, or black,) proceeds from the cobalt solution only, and not from any of the ingredients. If the mass separates from the charcoal, after the moderate red heat, as a thin pellicle, it must be submitted to an intense oxidating flame, in the points of the forceps. The colour now imparted must be observed on cooling, by daylight; because, when seen at candle-light, a blue often appears dirty violet, or even red, by transmitted light.

When a metallic oxide is present in a mineral, which may give it a white colour, this will not interfere with the alumina or magnesia re-action, unless a large quantity is present, and then the mass will generally become grey or black. In treating some minerals *per se* on charcoal, or in the reduction-test with soda, the metal often sublimes, becomes oxidized immediately, and is deposited upon the charcoal. Many such oxides are easily expelled by an oxidating flame, others with greater difficulty, and some not at all; so that it is often all



but impossible to separate the pellicle of oxide from the charcoal. If such a film rest upon the support after the experiment, a few drops of a solution of cobalt is to be added to it, and the assay submitted to a pretty strong oxidating flame. If zinc be present, a distinct bright green colour will be imparted to the substance, which should be viewed through the microscope.

The quantity of the cobalt solution to be employed, depends greatly upon its strength: experience, however, very soon teaches the proper quantities to be taken, in order to obtain a decided reaction.

Minerals, &c., by examining them according to the foregoing rules, in flasks, open tubes, and with fluxes, in the different flames produced by the blowpipe, give numerous phenomena from which results must be gathered; and attention must be given to the slightest occurrences that ensue, for such often lead to the discovery of substances, never supposed to exist in the assay under examination.

The results, however, obtained in the examination of an unknown body before the blowpipe, can be fully appreciated only by those who are acquainted with chemistry, and therefore, no rules can be laid down sufficient to detail all the minutiae, as they can be arrived at by industry and practice only.

¶B *General Rules for Qualitative Blowpipe Analyses, by means of which, partly with the assistance of the moist way, the single ingredients of compound bodies may be detected.*

If, by means of the blowpipe, alone, or with glass fluxes, the constituents of a compound body are not satisfactorily determined, the moist way must be resorted to, in order to ascertain as nearly as possible, the collective ingredients. By such an association, an operator can, with accuracy and facility, examine the generality of bodies composed of different earths and metallic oxides, native silicates, slags, &c.

For the manipulation, 100 to 150 milligrammes of a substance are quite sufficient to take, in order to ascertain all the



ingredients, and should the operator be an expert blowpipe analyst, he will find that less will serve—only a small portion of a liquid re-agent is necessary. When its pyrognostic qualities have been remarked, or the class to which it belongs has been determined, and whether it is an alkali, earth, metallic salt, silicate, aluminate, compound metallic oxide, metallic oxide with an earth, metallic sulphuret, or a combination of different metals, including arsenic and selenium, the single ingredients may readily be recognized. The procedure will be particularly adverted to in the qualitative analysis of alkalies, earths, metallic and non-metallic bodies, and such substances as are met with in nature. To prevent a repetition of many manipulations, I shall previously describe the methods for melting and dissolving, &c., and afterwards give them separately.

(21) SMELTING OF THE SUBSTANCE WITH BORAX AND SODA.

A compound in which the earths cannot be detected by a single reaction, must be triturated in an agate mortar, to an impalpable powder, and from 75 to 100 milligrammes employed for the analysis. If it contains sulphurets or arseniurets, it must be roasted twice with charcoal, in a manner similar to the quantitative assaying of copper, to expel all or the greater portion of the sulphur or arsenic, and to oxidize the metals which are generally combined with traces of formed sulphuric or arsenic acids. The powder is now triturated in a mortar with soda and borax, the proportions of these re-agents being rectified according to the degree of solubility of the assay; the mixture is then pressed in a cornet of soda paper, (similar to a quantitative copper assay,) and operated upon in a cylindrical cavity made in a piece of charcoal, by an intense blowpipe-flame. In general the melting is more readily effected in the oxidating than in the reducing flame. If reducible metallic oxides or sulphuric acid are not present, the oxidating flame is always preferable, but if otherwise, the reducing flame, in which the metallic oxides sublime, and the sulphuric acid is reduced to sulphur, which combines with the sodium, &c.



When the metallic oxides are in such small quantities as to be difficultly reducible to separate beads, 200 or 300 milligrammes of lead are added, or else a globule of silver, weighing 80 to 100 milligrammes, and the process is carried on like a quantitative silver examination. By this method, the earthy materials, and difficultly-reducible oxides, undergo the igneous fusion, and form a light liquid pearl. The sulphuric acid, acids of arsenic, and easily-tested oxides, are reduced, if present; the sulphur combines with the sodium and some of the other metals, the metallic arsenic partly sublimes and is partly absorbed by the alloy, and the non-sublimable metals unite, particularly when lead or silver has been added, forming a globule. The metallic globule attaches itself to the glass, especially if lead is predominant, a part of which volatilizes. The metallic oxides dissolved in the glass are principally in the state of protoxides. An intense oxidation or reduction must be employed for the preceding analysis, because otherwise fluidity or the reduction of the different ingredients would not ensue. The smelted assay must be obtained limpid, and be as free as possible from bubbles and metallic particles. When much magnesia is present, it is difficult to obtain a translucent glass, therefore more borax is to be added. If, after blowing for some time, metallic laminae are still perceived, it indicates reduction of the oxides, or solution of the non-reducible parts; therefore the heat must be continued.

When an analysis is completed, the assay must immediately be removed from the charcoal, by tapping it upon an anvil (described at p. 18), or on paper, in an agate mortar, and pulverized. This precaution is necessary, because the ignited mass readily absorbs moisture, becomes unctuous, and is therefore powdered with difficulty.

When the assay has been fused in the reduction flame, whereby metallic globules are obtained (compound alloys, containing the lead or silver added with other metals), these must be separated from the molten mass, by allowing it to flow gently from side to side upon the charcoal, while covered by the reducing flame. The whole is then removed with a spatula,



and the carbonaceous particles either burnt or scraped off with a knife. The glass is then divided upon the anvil from the attached metallic globule. If the necessary reduction heat is not attended to, part of the metallic oxide may remain, or a trace be oxidized through the medium of the lead and dissolved in the glass, which may have such an influence on further manipulation as to give entirely false results.

The metallic oxides which are easily reducible with soda and borax in the reducing flame, and which can readily be separated from certain earths and other irreducible metals, are as follows:—Arsenious, Arsenic, Antimonious, and Antimonic Acids, Oxides of Antimony, Silver, Mercury, Copper, Bismuth, Tin, Lead, Cadmium, Zinc, and Nickel. Selenium, Tellurium, Osmium, Gold, Platinum, Iridium, Rhodium, and Palladium, are never met with in nature but in the metallic state; they are therefore readily separated from certain earths and metallic oxides by cupellation with lead or silver. Those metals which are volatile, evolve, either partly or entirely, a vapour by such fusion, which condenses in the charcoal. The metallic oxides which are not reduced when melted with soda and borax, are—Oxide of Chromium, Molybdic, Tungstic, Tantallic, and Titanic Acids, Oxides of Uranium, Cobalt, Iron, Manganese, and Cerium. These can, however, be readily separated from the earths, and recognized by their pyrognostic effects, as will be adverted to under the different heads.

(12) METHOD OF TREATING WITH HYDROCHLORIC ACID THE SUBSTANCE  
MELTED WITH SODA AND BORAX.

The powdered glass is removed to a porcelain basin, and then affused with an excess of hydrochloric acid. The contents of the basin are now to be stirred with a glass rod, and the whole heated upon the triangle, over the flame of the lamp. By this treatment soluble and insoluble parts are separated, the sulphuret of sodium is decomposed, the sulphur combines with the hydrogen of the acid, and is eliminated as sulphuretted hydrogen (hydrosulphuric acid), and the other substances, except the silicic acid, form metallic chlorides. But as siliceous combinations only are generally separated by



fusion with soda and borax, consequently, in these cases the operator very seldom encounters Molybdic, Tungstic, Tantallic, and Titanic acids.

So soon as the solution is completed, the whole menstruum is to be evaporated slowly to dryness, under a chimney, in order to prevent the metallic chlorides (principally composed of chloride of sodium) from being lost by decrepitation.

The evaporation serves two purposes,—to expel the excess of acid, and to separate the silicic acid (silica) as gelatinous hydrate. When evaporated to a consistency, distilled water is added, and the whole mixture again heated; whereby the metallic chlorides are dissolved and separated from the insoluble residue. If the residue contains silica, it can readily be recognized, by filtering, washing, and treating what remains with microcosmic salt, or soda, before the blowpipe.\*

If the substance contains peroxide of iron, it becomes reduced when smelted to the state of protoxide; and is not, on the addition of hydrochloric acid, again converted into the peroxide. As it is, however, absolutely necessary, in order to detect accurately the different constituents, that the iron should be in the state of peroxide, a few drops of nitric acid are to be added to the filtrate (from the silicic acid mixed with the first water used in the edulcoration), and heated, in order to accomplish the purpose. The different constituents intended for examination are then separately investigated, according to the methods given under their respective heads.

(13) SMELTING OF THE SUBSTANCE WITH SALTPETRE (NITRATE OF POTASH), OR BISULPHATE OF POTASH.

When only one of the constituents of the substance under examination is sought for, it is sometimes necessary to fuse it with saltpetre, in order to convert that constituent into a higher oxide; or, in case it be an acid, to combine it with the potash of the saltpetre, from which it can be easier separated and recognized. In the same manner a substance is often

\* [If the reader refers to Dr. R. FRESENIUS's work, edited by BULLOCK, he will find an excellent article, p. 146, upon the Silicates and their treatment.]—*Translator*.



fused with bisulphate of potash, and the melted mass dissolved in water, in order either to free it immediately from certain constituents, or to convert the whole into sulphates, and thus be able, after the solution in water, to effect the separation of its different components.

The fusion with saltpetre may sometimes be performed in the loop of the platinum wire, but it would be better to employ a platinum spoon.\* Pulverulent and pulverizable substances should be mixed in a finely triturated state, in a mortar, with the necessary quantity of saltpetre. Metallic alloys, which do not admit of being pulverized, must be reduced to a finely divided state with the aid of the hammer or file. The quantity of saltpetre to be used can be judged of from the facility with which the substance undergoes oxidation; from three to four times the bulk of the substance may generally be employed, provided it is not of great specific gravity. When only one constituent is sought for, the fusion may take place on the platinum wire; the mixture must in this case be moistened with a little water, and in a soft or doughy state placed on the loop of the wire. The oxidating flame only is used in the fusion, and when the mass has ceased to vesiculate, another portion of the moistened mixture is to be added to the fused mass, and both melted together: this is to be continued until the fused mass, owing to its size, can no longer remain attached to the wire. During the fusion the wire must be held obliquely, and in such a manner that the loop is directed downwards; as, otherwise, the saltpetre would be apt to flow along the wire.

If it is conjectured that the substance contains only an extremely small quantity of the body sought for, or if it be intended to oxidize other constituents, in order to be able, in this state, to subject them to further treatment,—or if a metallic alloy, or an unpulverizable substance be under examination,—a larger quantity must be taken for the experiment, and the fusion performed in a platinum spoon. The

\* [The surface of platinum is oxidized slightly by saltpetre; but this is of no moment, as it produces no disadvantageous effects on the experiment, and as a person need not fear that the spoon will be destroyed.]—*Trans.*



whole mixture should not, however, be put into the spoon at one time, but only small portions; because, during the fusion, gas and vapour are given off, which would easily cause the melting mass to flow over the top of the spoon. The spoon should be first treated externally on the bottom with the oxidating flame, the flame should then be directed into the spoon, and the whole fused until it becomes perfectly fluid, the remainder should then be added to it in small portions, and the mass perfectly fused after each addition. During the fusion, the position of the spoon before the oxidating flame, should be so changed, that every part of the mass to be fused be subjected to its action, and the spoon itself raised to a red heat.

Such metallic alloys only can be treated with saltpetre in a platinum spoon as are readily oxidized, and do not combine with the platinum at a temperature capable of being produced by the blowpipe. The fusion of a metallic alloy with saltpetre, is thus confined to the detection of traces of arsenic, in such metals as can with difficulty be separated from it, and those which are exceedingly difficult of fusion, as for example, nickel.

The fusion of a substance with bisulphate of potash, may also be performed in the large platinum spoon, either in the same manner as with saltpetre, or still better in the flame of the spirit lamp; the substance must, however, be perfectly dry and in a finely divided state. When the fusion is made with the blowpipe, some of the sulphates formed by such a fusion, may be again deprived of their acid by the strong heat. If, however, the fusion is performed in the flame of the spirit lamp, the spoon in the commencement being held only over the top of the flame, until the greater part of the gases escape, and then sunk deeper in the flame, so that the heat may act equally from all sides on the bottom of the spoon, the melting mass will be raised to an incipient red heat only, and the salts so formed will not be decomposed. In some cases where a large quantity of the salt must be employed, the spoon becomes filled before the whole of the mixture has been added. In this case the fluid mass should be poured out on the anvil, and the remainder of the mixture



then melted. It is also sometimes advisable to pour out the melted mixture, particularly in the examination for titanitic acid, where the water necessary for its solution cannot be raised to the boiling point. The quantity of bisulphate of potash which should be employed, depends upon the different constituents of the substance; thus, for example, for protoxide of iron, three to six times; for lime, four to five times; for magnesia, six times; and for alumina, seven to eight times the weight of bisulphate is required to convert them into sulphates. The operator always proceeds more cautiously by employing a slight excess of the acid salt, as the oxides of iron and alumina, by continued high temperature, lose with great facility a part of their combined sulphuric acid.

(24) SOLUTION IN WATER OF THE SUBSTANCE FUSED WITH SALTPETRE  
OR BISULPHATE OF POTASH.

The solid mass obtained by fusing a substance with saltpetre or bisulphate of potash, whether on the platinum wire or in the platinum spoon, cannot, unless it has been poured out when in a state of fusion, be well pulverized, and in this state dissolved in water, because in the removal of such a mass, the platinum is liable to be tarnished; it is therefore necessary to place the wire or the spoon with the fused mass in a porcelain basin or cup, proportionably to the volume of the mass, then pour over it the quantity of water necessary for its solution, and place the vessel on the triangle over the flame of the lamp. As the water becomes warm, the mass generally separates from the platinum, and can then be crushed with the pestle of the agate mortar. In most cases the water can be raised to the boiling point, and the mass thus easily dissolved; but if a titaniferous mineral be melted with bisulphate of potash in order to render the titanitic acid soluble, the water must not be raised to the boiling point, as otherwise the titanitic would not be dissolved, and even that part which was dissolved at a low temperature, would be again precipitated. The manner of treating the solution, and the residue which could not be converted into sulphates, will be more fully entered upon hereafter, when we come to speak of the qualitative examination of earths and metals.



(15) PRECIPITATION (BY CRYSTALLIZED OR PULVERULENT RE-AGENTS) ~  
OF SINGLE CONSTITUENTS DISSOLVED IN FLUIDS.

If it be necessary to decompose a clear solution with a dry salt, the acid or base of which is capable of combining with one of the constituents of the dissolved salt, and as a compound insoluble in this fluid is precipitated, the fluid must in most cases, after the addition of the salt, be slightly heated, in order that it might easily dissolve, and thus serve as a re-agent. In some cases it is also better to warm the liquid before the addition of the salt, as in this case the precipitate is instantly produced. Thus, for example, barytes and strontian can be precipitated by pulverized bisulphate of potash, lime by crystallized oxalate of ammonia; magnesia by microcosmic salt in crystalline grains; phosphoric acid by a crystal of nitrate of silver; chromic acid by acetate of lead, &c. The quantity of the re-agent used each time cannot be accurately estimated, as it entirely depends upon the quantity of the substance to be precipitated.

## (16) DECANTATION, FILTRATION, AND EDULCORATION.

In cases where only one substance is to be sought for, in the clear liquid which covers a residue or precipitate, it is only necessary to pour off carefully the greater part of the liquid. But if the liquid, or the residue, or precipitate, is to be examined for a number of constituents, the whole should be filtered, and the residue on the filter edulcorated with water.

A very thin, porous, or unsized paper should be employed, which should be cut into filters, of an inch or an inch and a half radius. For exceedingly small precipitates, where it may be necessary to remove a portion of the paper with the precipitate, filters should be made of Swedish paper, which is prepared with distilled water, or from paper freed from its earthy constituents, by means of hydrochloric acid, then well washed with distilled water, in order to remove the adhering acid and salts, and then dried.

The operator should proceed in the following manner with the filtration and edulcoration. The filter, placed in the funnel, should be saturated with distilled water, in order that



as little as possible of the dissolved substance might remain behind in it, the liquid and precipitate, or residue, should then be poured by degrees on it; the glass which contained the mass washed with a little water thrown on the filter, and the whole liquid allowed to percolate. If the filtrate contains a number of substances, which it is intended to examine, the filter should be filled with water, which dissolves the greater part of the soluble matter still adhering to the filter, and precipitates; this water, on passing through, should be added to the first liquid. No more of the water used in the edulcoration should be added to the filtered liquid, as otherwise it becomes too dilute, and the constituents are very difficult to be separated from one another. The funnel, with the filter, should then be placed upon another vessel, larger, (as for example, a flask with a narrow neck,) and the residue well edulcorated with more water. The washing-bottle described at page 26 can be used in edulcoration, in order the better to bring a small precipitate to the bottom of the filter. It seldom happens that boiling water is necessary to wash a precipitate; cases however occur where boiling water may be used with advantage, particularly with argillaceous precipitates, which are to be further treated. In this case, the operation may be performed in the following manner: distilled water should be poured into a porcelain vessel, and boiled over the spirit lamp; a portion should then be sucked up with the glass pipette, described at page 26, and ejected on the precipitate; when this portion has filtered through, the operation is to be repeated until the edulcoration is complete.

#### § 7. DESICCATION OF THE FILTERED RESIDUES AND PRECIPITATES.

Sometimes it is not necessary to dry the residue or precipitate, as it may be subjected, while still moist, to further investigation. Cases, however, occur where it must be perfectly dried, and in this state further treated with fluxes on charcoal, or in the platinum spoon.

If the substance to be dried is in sufficient quantity, the filter should be opened out on two folds of bibulous paper, the mass be spread out with a spatula, and introduced into a



perfectly smooth porcelain basin, and dried over the flame of the lamp. But if the residue is small, the unfolded filter should be held to the light, and the part of the paper on which none of the precipitate is seen, cut off with scissors, and the remainder, containing the filtered mass, dried in a small porcelain basin. When the paper is dry, it is to be folded double, and inflamed at one end, while firmly held at the other with the forceps, and thus allowed to burn over a clean dry

¶ *Behaviour of Alkalies, Earths, and Metallic Oxides,*  
I. ALKALIES

ALKALIES.	Upon Platinum Wire.	
1. Potash. K O.	When covered with the point of the blue flame, the outer flame is coloured violet. A small addition of Soda prevents this reaction.	
2. Soda. Na O.	The flame is coloured yellow, although Potash is present.	
3. Lithia. L O.	Flame is reddened, if no Soda is present.	
4. Ammonia. N H <sup>3</sup> .	0	
EARTHS.	Upon Charcoal, and with Borax, on the Platinum Wire.	
5. Barytes. Ba O.	Caustic; infusible. The Hydrate and Carbonate melt, and are absorbed by the Charcoal.	The Carbonate decrepitates, and forms a bead, which is transparent when cold.
6. Strontian. Sr O.	Hydrate, fusible: the Carbonate, only partially so. Reduction flame reddened.	Ditto. [ With moist soluble salts, the flame is intensely crimson. ]— <i>Translator</i> .
7. Lime. Ca O.	Caustic; not changed. The Carbonate becomes caustic, and emits a strong white light.	Readily dissolved. The Carbonate deflagrates, and forms, when cold, a transparent crystalline glass.
8. Magnesia. Mg O.	The Carbonate is decomposed, and the residue has an alkaline reaction.	Like Lime; but the bead formed is not so crystalline.
9. Alumina. Al <sup>2</sup> O <sup>3</sup> .	Not changed.	Fuses to a colourless glass, which remains so when cold.
10. Glucina. Be <sup>2</sup> O <sup>3</sup> .	Ditto.	Fuses to a colourless glass, which becomes slightly opaque when cold.
11. Yttria. Y O.	Ditto.	Ditto.
12. Zirconia. Zr <sup>2</sup> O <sup>3</sup> .	Infusible, but produces a very glaring white light.	Ditto.
13. Thoria. Th O.	Not changed.	With a minute quantity, forms a clear glass.
14. Silica, (or Silicic Acid.) Si O <sup>2</sup> .	Ditto.	Gives a transparent and colourless bead, which is soluble.

[The only way that Potash, if contained in a mixture of Potash and Soda, can be detected by the Blowpipe, is the Mixture. If Potash be present, the brown colour of the Nickel bead



porcelain basin, into which the filtered substance gradually falls, mixed with a little carbon. This carbon is easily burned off in the platinum spoon; but it is unnecessary, when the dry mass is to be further treated with fluxes, as it is by this means destroyed.

It would be superfluous to mention here every trifling appliance which so often contributes to the success of a fusion, a solution, &c., as every person brings to his aid his own peculiar advantages and manipulation.

*alone, and with Re-agents, in the Blowpipe Flame.*

AND EARTHS.

Upon Platinum Foil.		Remarks :—
0		When in solution, they tinge reddened Litmus Paper, blue.
0		
Fuses, with the formation of a dark yellow coating on the surface.		Recognized by its pungent odour. It affects Litmus Paper in the same manner as the preceding alkalies.
0		
With Microcosmic Salt, upon Platinum Wire.	With Carbonate of Soda, on Charcoal.	With a Solution of Cobalt, in the Oxidating Flame.
As with Borax.	Dissolves, and is absorbed by the charcoal.	Fuses to a yellowish-red globule, which is colourless when cold. Becomes greyish by exposure.
Ditto.	Caustic: insoluble. The Carbonate dissolves, and is absorbed by the charcoal.	Assumes a dark greyish colour.
A colourless bead, which, if saturated, becomes opaque when cold.	Not dissolved. The re-agent is absorbed by the charcoal.	Ditto.
Ditto.	No action.	Assumes, on cooling, a beautiful flesh-red colour.
Fuses to a transparent glass, which becomes slightly opaque, if an excess is present.	Intumescent, with the formation of an insoluble compound. The excess of Carbonate of Soda is absorbed.	Gives a fine transparent blue glass upon cooling.
As with Borax.	No action.	Infusible, but becomes of a greyish black colour.
Ditto.	Ditto.	Ditto.
Fuses very slowly; and is scarcely transparent when cold.	Ditto.	Ditto.
As with Borax.	Ditto.	0
Ditto.	Ditto.	With a very small quantity of the Cobalt Solution, a slight blue colour is produced, which changes to a dark grey, when an excess is added.

to fuse a clear bead of Borax with a small quantity of Oxalate of Nickel, upon the platinum wire, and to add is changed to blue: a salt of Soda effects no such change.]—*Translator.*



TABLE II.—BEHAVIOUR OF

METALLIC OXIDES.	Per Se, on Charcoal.		With Borax on a Platinum Wire.	
	In the Oxidating flame	In the Reducing flame	In the Oxidating flame.	In the Reducing flame.
1. Oxide of Cerium. $\text{Ce}^2\text{O}^3$ .	The Protoxide is converted into the Peroxide. In the Reducing flame, this reaction remains unchanged.		Soluble, forming a red or dark yellow glass (like oxide of iron). When cool, the glass is only yellow.	The glass containing oxide becomes pale, so that a yellow oxide becomes colourless. When a large quantity of the oxide is present, the glass is on cooling of an enamel white crystalline texture.
2. Sesquioxide of Manganese. $\text{Mn}^2\text{O}^3$ .	Infusible. On being slightly heated, it becomes of a brown colour. [The Per-, Sesqui-, and Protoxides, produce similar effects.]— <i>Translator</i> .		Intensely coloured:—violet (amethyst) while hot; violet with a tinge of red, when cold. If a large quantity is present, the globule will be black.	The coloured bead becomes colorless. Should the colour be very intense, the reduction will succeed better upon charcoal with the addition of a small quantity of Tin.
3. Oxide of Zinc. $\text{Zn O}$ .	Yellow bead, while hot;—white, when cold. It does not fuse, but, when very hot, gives out an intense light.	It is (gradually) dissipated over the charcoal, with white flakes of oxide of zinc.	Very soluble, forming a clear mass, which, if perfectly saturated, remains colourless when cold. By a strong heat, it is reduced, acting on the platinum.	The saturated mass will become enamel white upon platinum wire. On charcoal the oxide is reduced, and the zinc volatilized. The charcoal also acquires a coating.
4. Oxide of Cobalt. $\text{Co O}$ .	<i>No change.</i>		Deep blue, while hot, and when cold. Should an excess be present, the glass will appear of a blackish hue.	Same as in the Oxidating flame.
5. Oxide of Nickel. $\text{Ni O}$ .	<i>No change.</i>		Strongly coloured.—When a small quantity is present, the glass is violet when hot, and pale reddish brown when cold.	The glass containing the oxide becomes dull or quite opaque, from the presence of finely divided metallic nickel. By long blowing, the particles of Nickel adhere to one another, forming a globule or bead, and the flux becomes colourless.
6. Peroxide of Iron. $\text{Fe}^2\text{O}^3$ .	<i>No change.</i>	Becomes black and magnetic.	When a small quantity is present, the glass is yellow, while hot, but colourless, when cold. If a large quantity is present, it is red, when hot, yellow, on cooling. When an excess is present, it is dark red, when hot, dark yellow, after cooling.	On Platinum wire the glass becomes bottle green. On charcoal with tin, the glass containing the oxide of iron becomes first bottle green, and afterwards vitriol green. On the platinum wire, the glass heated with tin immediately appears copperas green.
7. Oxide of Cadmium. $\text{Cd O}$ .	No change, upon Platinum Foil. Disappears in a short time, covering the charcoal all over with a dark yellow powder, which is only clearly discerned when the mass cools.		When large quantities are present, the Borax melts to a clear yellowish glass; this colour nearly disappears on cooling. When the borax is nearly saturated, the glass formed is sometimes, here and there, milk-white, and, when perfectly saturated, it becomes enamel-white, if slowly cooled.	On Charcoal the glass containing the oxide of Cadmium boils; the cadmium is reduced and volatilizes, and the charcoal becomes coated with dark brown oxide.



With Microcosmic Salt on Platinum Wire.		With Soda.	With a Solution of Cobalt, in the oxidating flame.
In the Oxidating flame.	In the Reducing flame.		
As with Borax; but the colour disappears entirely when cold.	The glass is colorless when hot and cold; by this reaction it is distinguished from iron. The glass remains also clear when over-saturated.	Not fused; soda absorbed by the charcoal, the metal remaining upon the surface of the support, in the state of a white or lightish grey protoxide.	No re-action.
When a large quantity is added, the glass appears violet, but on the addition of more, it acquires a black tinge. If the salt contains much oxide, on being heated strongly, gas is evolved. Saltpetre assists in bringing out the colour, when added.	The glass becomes instantly colourless, both on platinum wire and charcoal. After reduction, the glass becomes perfectly fluid.	In the oxidating flame, upon platinum wire or platinum foil, it fuses to a soluble transparent green mass, becoming opaque and bluish green, on cooling. On charcoal it cannot be reduced.	0
Like Borax, but with this difference, that the saturated glass becomes enamel white upon cooling.	Same as with Borax.	Insoluble. Is reduced on charcoal, covering it with a sublimate of oxide. By a strong flame, the metal may be inflamed.	Gives a beautiful green colour.
As with Borax, but the colour, however, is not so intense. [By transmitted light, the glass appears reddish.]— <i>Tr.</i>	Same as in the Oxidating flame. [If a bright iron wire is inserted into the mass when in fusion, Cobalt is deposited.]— <i>Trans.</i>	On Platinum Wire, in small quantities, it affords a transparent slightly red soluble mass, which is grey when cold. It is reduced on charcoal, giving a grey magnetic powder.	0
Melts to a reddish glass, which is yellow on cooling. If more is added, the glass assumes a brownish red when hot, and a reddish yellow, when cold.	Upon platinum wire, not altered. Treated on charcoal with tin, the glass is, at the commencement, translucent and grey, but by continued blowing, the Nickel deposits, leaving the glass colourless.	Insoluble. It is easily reduced upon charcoal, giving small white shining metallic particles, which, on being triturated, are readily attracted by the magnet.	0
The glass containing a certain quantity becomes yellowish red. When cooling it appears first yellow, then greenish, and lastly colourless. If a very large portion is present the colour will be dark red while hot, and brownish red on cooling, changing to dirty green, and, when perfectly cold, brownish red. The colours disappear on cooling, quicker than with borax.	The glass is not changed from the presence of a minute portion; but if a large quantity is present, it is red when hot, yellow during refrigeration, then greenish, and when quite cold, reddish. With the addition of Tin, the glass is first green, and then the colour is destroyed.	Insoluble. On charcoal it is reduced; and gives, on being pulverized, a grey metallic magnetic powder.	0
With large quantities, it fuses to a transparent glass; if saturated, it becomes milk-white on cooling.	Upon Charcoal, Cadmium is slowly and imperfectly reduced; the metal is volatilized, and a very small quantity of a dark red oxide coats the support. [This colour, when cold, is a beautiful orange red.]— <i>Tr.</i>	Insoluble. On charcoal it is immediately reduced; the metal is converted into vapour, and the charcoal becomes coated with a dark yellow oxide.	0



TABLE II.—BEHAVIOUR OF

METALLIC OXIDES.	Per Se, on Charcoal.		With Borax, upon the Platinum Wire.	
	In the Oxidating flame	In the Reducing flame	In the Oxidating flame.	In the Reducing flame.
8. Oxide of Lead. $Pb\ O.$	Minium (red lead) when slightly heated, becomes black; but, upon increase of temperature, it is converted into a yellow oxide.	It is immediately reduced to a metallic globule, which gradually sublimates, leaving a yellow residue.	Readily fuses to a clear yellow glass, colourless when cold. If a large quantity is present, the globule will be here and there opaque. With an excess, it is enamel-yellow when cold.	The glass diffuses itself over the charcoal; becomes turbid, and boils. The lead is reduced, and the glass becomes again clear. It is very difficult to obtain, in the process, a bead of lead.
9. Oxide of Bismuth. $Bi\ O.$	On platinum foil, it fuses readily to a dark brown mass, which becomes pale yellow, on cooling.	On charcoal it is quickly reduced to grains of Bismuth, which, with long blowing, vaporize, leaving the charcoal covered with yellow oxide.	Readily fusible to a limpid glass, which when a certain quantity is present, is yellow, when hot, colourless when cold. A large quantity gives a yellowish-red glass when hot, while cooling, it is yellow, and when perfectly cold, opalescent.	On Charcoal the glass becomes at the commencement grey and turbid;—boils, the oxide of bismuth being reduced, it again becomes perfectly clear. With Tin added, it is first grey, and then the whole of the bismuth separates, and the glass appears clear and limpid.
10. Peroxide of Uranium. $U^2\ O^3.$	It is converted into the protoxide; it blackens, but does not fuse.		As with Oxide of Iron, but the colour is somewhat clearer. When the glass is strongly saturated, it is streaked with enamel yellow.	Gives the same colour as oxide of iron. The green glass, when saturated to a certain point, is blackened by an intermitting flame. It is either crystalline or enamel.
11. Oxide of Copper. $Cu\ O.$	Forms a black globule, which flows over the charcoal, and the under-surface is reduced.	By a temperature at which the copper does not fuse, the oxide is reduced, presenting the fine metallic lustre of copper. When the blowing ceases, the surface of the metal is re-oxidized, and becomes black or brown. By increasing the heat, a globule of copper is obtained.	The glass is not very strongly colored. A small proportion of the Oxide gives a grass-green when hot, becoming blue on cooling. By a larger dose of the oxide, the glass is intensely green, appearing opaque; on cooling, it becomes opaque, and greenish blue.	At a certain point of saturation, the glass becomes colourless; but in a few moments it becomes red and opaque. By long blowing, the copper is precipitated in the metallic state upon charcoal, and the glass becomes colourless. When treated with Tin, the glass is opaque, and brownish-red on cooling.
12. Oxide of Silver. $Ag\ O.$	Reduced very rapidly.		Partly reduced, and partly dissolved. On cooling, the glass becomes milk-white or opaline, owing to unequal distribution of the dissolved oxide. Metallic silver melts with borax, giving the same re-action.	On charcoal the glass containing the oxide becomes grey, on account of the reduced silver. Later, it becomes limpid and colourless, from the precipitation of the silver. This metal can be obtained as a bead.
13. Oxide of Mercury. $Hg\ O.$	Instantly reduced and volatilized. [Should the whole not sublime, foreign matters will have been present, so that by this means adulteration is detected.]		"All the compounds of Mercury are volatile: mixed with Tin or Iron Filings, and heated in a glass tube, metallic Mercury distils over."	
14. Peroxide of Platinum. $Pt\ O^2.$ 15. Peroxide of Palladium. $Pd\ O^2.$ 16. Peroxide of Rhodium. $R^2\ O^3.$ 17. Binoxide of Iridium. $Ir\ O^2.$	Easily reduced: the metallic particles will not adhere to form a globule.		Reduced, but not soluble. The metallic particles alone upon charcoal do not form globules.	As in the oxidating flame. ("These Metals have no action upon the Fluxes,—which can only serve to detect the foreign metals they may be combined with. "They are best examined by cupellation with lead").



With Microcosmic Salt on Platinum Wire.		With Soda.	With a Solution of Cobalt, in the oxidating flame.
In the Oxidating flame.	In the Reducing flame.		
As with Borax. It is necessary to add a large quantity of the Oxide of Lead, to obtain a decided colour when hot.	On Charcoal the glass becomes grey and turbid. If an excess be present, the charcoal will be coated with a yellow oxide of lead. When Tin is added, the glass never becomes opaque, but a little more turbid, and of a darker grey.	In the oxidating flame upon platinum wire, it fuses readily to a limpid glass, which, upon cooling become yellow and opaque. On charcoal the oxide is readily reduced.	0
A small quantity fuses to a transparent glass. If a large quantity is present, the glass will be yellow when hot, but colourless when cold. If a certain quantity is present, an enamel white here and there is often produced. When an excess is present, enamel white on cooling.	On Charcoal, particularly when Tin is added, the glass remains colourless and limpid while hot; but on cooling becomes opaque and greyish-black.	Easily reduced upon charcoal. [When volatilized, it gives no colour to the flame, by which means it is distinguished from Antimony, Tellurium, &c.]— <i>Tr.</i>	0
Dissolves, giving a clear yellow glass, which becomes green on cooling.	The glass containing the oxide partakes of a beautiful green colour, which on cooling becomes finer and clearer.	Insoluble. With a small addition of soda it gives indications of melting; with a greater quantity of soda it becomes yellowish-brown; and by a still greater addition, the oxide is absorbed by the charcoal unaltered.	0
The glass containing the same quantity of oxide as with Borax, is not so highly coloured. A small quantity of oxide gives a green glass when hot; blue, when cold. A greater quantity gives a beautiful green, when, hot; blue, when cold. An excess of oxide gives an opaque glass, when hot; a greenish blue, when cold.	When nearly saturated, the glass is of an intensely dark green while hot, and on cooling appears opaque, and of a reddish brown. When the glass containing a very small quantity is treated with Tin, it is opaque and reddish-brown when cooled.	It dissolves upon the platinum wire to a limpid green glass, which, by refrigeration, loses its colour, becoming opaque. It is readily reduced upon charcoal, and gives one or more beads of copper.	0
Metallic Silver, as well as the oxide, gives a yellowish glass. If a large quantity is present, the glass becomes opaline, and appears yellowish by daylight, red by candle-light.	As with Borax.	It is instantly reduced.	0
"All the compounds of Mercury are volatile: mixed with Tin or Iron Filings, and heated in a glass tube, metallic Mercury distils over."		When mixed with soda, and heated to redness in a closed tube, it is reduced and vaporized. The sublimate condenses in the coldest part of the tube, as a grey coating, which, when agitated, unites, forming a globule.	0
As with Borax.	As in the Oxidating flame.	As with Borax; the soda is absorbed by the charcoal.	0



TABLE II.—BEHAVIOUR OF

METALLIC OXIDES, AND ACIDS.	On Charcoal, without Re-agents.		With Borax upon Platinum Wire.	
	In the Oxidating flame	In the Reducing flame	In the Oxidating flame.	In the Reducing flame.
18. Peroxide of Gold. $\text{Au}^2 \text{O}^3$ .	The instant the Peroxide becomes red-hot, it is reduced.		It is not soluble, but is reduced, and can by fusion be obtained as a metallic globule.	As in the Oxidating Flame.  ("Gold has no tendency to combine with Oxygen or Sulphur; and hence retains its brilliancy in the open air for any length of time. It melts at $2016^\circ$ ; its density is 19.5; it is not acted on by any single acid, but is dissolved by nitro-muriatic acid, and by a mixture of nitric and hydrofluoric acids."—Kane.)
19. Peroxide of Tin. $\text{Sn O}^2$ .	The Protoxide inflames like touch-wood, and is converted into the Peroxide. The peroxide burns brilliantly, but it remains unchanged.	The Peroxide of Tin is reduced by long heating.	In small quantities it dissolves but very slowly, forming a glass which is colourless and limpid, whether hot or cold:—it may, perhaps, become opaque. A glass perfectly saturated, when heated, becomes opaque, loses its globular form, and appears a confused crystalline mass.	A glass containing a small quantity of the oxide, is not altered. If more be added, the oxide is partly reduced upon charcoal.
20. Oxide of Antimony. $\text{Sb O}^3$ .	It is displaced, without change, and deposited upon another part of the charcoal.	It is reduced, and sublimes. The charcoal becomes coated with a white oxide, and a greenish-blue colour is imparted to the flame. [Antimony, when heated in a tube open at both ends, gives off large quantities of white vapours.]— <i>Trans.</i>	It dissolves in large quantities, and gives a limpid glass, which in the flame appears yellow, and on cooling becomes almost colourless. On charcoal, the dissolved oxide becomes paler: an addition of Tin does not alter this effect.	The glass, when treated only for a short time in the oxidating flame, becomes grey and cloudy, from particles of the reduced antimony; these are quickly volatilized, and the glass becomes lucid. When treated with Tin, the glass becomes ashy-grey or black, whether there is a large or a small proportion of the oxide present.
21. Oxide of Chromium. $\text{Cr}^2 \text{O}^3$ .	Unchanged.		It colours the Borax highly, but it dissolves slowly. If an equal quantity is present, the glass will be yellow when hot, yellowish-green, when cold. If a larger quantity is present, it is dark red, when hot; on cooling, yellow; and when cold, emerald-green, with a trace of yellow.	The slightly-saturated glass is of a beautiful green, when hot or cold. When a greater quantity is added, the colour is more strongly marked, becoming emerald green. The addition of tin does not produce any change.
22. Tellurous Acid. $\text{Te O}^2$ .	Melts, and is reduced with effervescence. The reduced metal volatilizes, and covers the charcoal with a coating of white oxide.		Dissolves, forming a limpid colourless glass; heated upon charcoal, its colour is grey, becoming opaque by reduction, from the metallic particles disseminated through the mass.	As in the Oxidating flame.
23. Molybdic Acid. $\text{Mo O}^3$ .	It melts, and is absorbed by the charcoal. In a strong reduction-flame, particles of the metal appear, which separate in the form of a grey metallic powder.		Soluble, forming a limpid glass, which appears yellow upon cooling, but is colourless when cold. When a large proportion of acid is present, the glass is dark yellow, when hot; opaline, when cold. With an excess the glass is dark red, when hot, becoming on cooling of an opalescent bluish grey.	The glass, treated in the oxidating flame with a very small quantity of acid, is coloured brown. If a little more be added, the glass becomes wholly opaque.



With Microcosmic Salt on Platinum Wire.		With Soda.	With a Solution of Cobalt, in the oxidating flame.
In the Oxidating flame.	In the Reducing flame.		
As with Borax.	As in the Oxidating Flame.	As with Borax. The soda is absorbed by the charcoal.	0
It dissolves in small quantities, very slowly, giving a limpid and colourless glass, which is not changed on cooling.	The glass containing the oxide is not altered, either upon platinum wire or charcoal.	Upon the platinum wire, Peroxide of Tin (stannic acid) effervesces, combining with the soda, forming an unmeltable tumacious mass. Upon charcoal it is converted into the metallic state.	Assumes a bluish green colour.
Dissolves with effervescence, giving a clear glass, which, when hot, appears of a feeble yellow colour.	The saturated glass upon charcoal, becomes milky at first, but in a short time clear; the antimony being reduced and dispersed in vapour. When treated with Tin the glass is green; this colour is owing to the reduced metal, but it soon becomes clear with strong blowing. The tin produces always a murky grey colour, when a very small proportion of the oxide of antimony is present.	Upon charcoal it is very easily reduced. The metal fumes and covers the charcoal with a coating of white oxide.	0
Soluble, forming a limpid glass, which is reddish when hot, and of a dull green colour on cooling. When perfectly cold, it appears of a beautiful green hue.	As in the oxidating flame. The colorations appear more marked; this is the same under the influence of tin.	In the Oxidating flame, upon platinum wire, it is dissolved with the formation of a dark brown yellowish glass, which upon cooling, becomes yellow and opaque. In the reducing flame the glass is opaque, and green when cold.	0
As with Borax.	As in the Oxidating Flame.	Soluble on the platinum wire, producing a limpid and colourless glass, which becomes white upon cooling. Upon charcoal it is reduced, volatilizes, and leaves upon the support a coating of oxide.	0
Soluble to a clear glass, which, by a moderate addition of acid, is yellowish-green, and on cooling, nearly colourless. Treated on charcoal, it becomes opaque, but, after cooling, of a fine green colour.	The glass assumes a dark dirty green colour, which on cooling changes to a fine clear green; this is analogous to the colour presented by oxide of chromium. Upon charcoal its behaviour is similar. An addition of tin has the effect of darkening this green tinge.	Fusible upon the platinum wire, with effervescence, giving a clear glass, which on cooling becomes milk-white. When treated upon charcoal, the mass is absorbed, and a large quantity of the Molybdic acid is reduced. When levigated, the scorise present the appearance of a steely-grey powder.	0



TABLE II.—BEHAVIOUR

ACIDS.	On Charcoal without Re-agents.		With Borax, upon Platinum Wire.	
	In the Oxidating flame	In the Reducing flame	In the Oxidating flame.	In the Reducing flame.
24. Tungstic Acid. $W_2O_5$	Unchanged, if the flame is not a very strong one.	Blackens,—but does not fuse.	Dissolves readily to a limpid colourless glass. A great proportion of the acid, gives a yellow when hot; and a greater quan- tity imparts to the glass the property of appearing opaque in the flame. If an excess be present, the glass becomes sponta- neously enamel-white, on cooling.	A small quantity of the acid does not alter the colour of the glass. A greater proportion ren- ders it yellow. A still greater quantity produ- ces a dark yellow when hot, which is yellowish- brown upon cooling. Tin darkens the colours, if the glass is not fully sa- turated.
25. Vanadic Acid. $VO_3$	Fusible.—The portions found in contact with the Charcoal are reduced and ab- sorbed. The others assume the colour and brilliancy of Graphite. They are the pro- xide of Vanadium.		Soluble to a limpid glass, which, by a small quantity of this acid, is colourless, but by a great- er addition, becomes yel- low.	The strongly yellow- coloured glass changes, so, that it appears brown when very hot, and, on cooling, of a beautiful chrome-green.
26. Tantallic or Columbic Acid. $Ta_2O_5$	Unchanged.		Soluble; forms a limpid colourless glass, which ap- pears opaque in the flame. If more tantallic acid be added, it appears enamel- white on cooling.	As in the Oxidating Flame.
27. Titanic Acid. $TiO_2$	Unchanged.		Readily soluble to a limpid colourless glass, which by a greater addi- tion of the acid, becomes yellow, and is colourless after cooling. At a cer- tain degree of saturation, the glass appears streaked with enamel-white; if an excess is present, it be- comes opaque on cooling.	A small proportion of the acid colours the glass yellow, while a larger quantity gives a dark yellow or brown shade. A glass when saturated, appears enamel-blue in the flame.

¶ *Description of the Phenomena presented by the sublimates of some Metals, when melted or heated strongly upon Charcoal, before the Blowpipe-flame.*

*Selenium.*—Affords a shining steel-grey slightly metallic deposit, which presents a violet, but when in thin layers, a blue lustre. In



With Microcosmic Salt on Platinum Wire.		With Soda.	With a Solution of Cobalt, in the oxidating flame.
In the Oxidating flame.	In the Reducing flame.		
Soluble, producing a clear glass; if more strongly saturated, the glass appears yellow while hot.	A beautiful clear blue glass is obtained. If the tungstic acid is combined with iron, the colour will be blood-red. The glass containing the iron is coloured blue when tin is added, and green if there is a considerable portion of iron.	Soluble upon the platinum wire, forming a dark yellow glass, which when cooling becomes crystalline and opaque, with a tinge of white or yellow. Upon charcoal, with a small quantity of soda, a large quantity of the acid is reduced; by a greater addition of soda, the whole is absorbed by the charcoal. A conjunction of the shining metal, and the yellow oxide of tungsten, is often obtained with the soda.	0
As with Borax.	As with Borax.	It fuses with the soda, and is absorbed by the charcoal.	0
Dissolves readily and easily in large quantities, forming a colourless glass, which cannot be rendered opaque in the flame, but which becomes spontaneously so upon cooling.	As in the Oxidating Flame.	It combines with the soda, effervescence taking place, but it is neither fused nor reduced.	0
Dissolves easily, forming a limpid glass, which when containing a certain proportion of acid, is yellow when hot, and colourless upon cooling.	The glass treated in the oxidating flame, presents the following appearances; when hot the colour is yellow, becoming on cooling red, and after assumes a beautiful violet tinge. When a greater quantity of the acid is added, the colour produced is so intense, that the glass appears opaque, but has not an enamel aspect. If the metal contains iron, the glass upon cooling will be brownish-yellow, yellow, or brownish-red. An addition of tin destroys this colour produced by the iron, and the glass becomes violet.	Soluble, forming, after the effervescence and decrepitation has taken place, a dark yellow glass. The glass is not absorbed by the charcoal; after cooling it appears greyish-white or white. If the glass is heated red hot, it crystallizes, and develops so much caloric, that the bead spontaneously attains a white heat. The titanio acid is not reduced.	It is coloured black or greyish black.

the oxidating flame it readily changes its place upon the charcoal, and in the reducing flame the colour is bright blue.

*Tellurium*.—Covers the charcoal with an oxide, at the same distance from the assay as selenium; the deposit is white, with a red or dark yellow border, easily removable by the oxidating flame, and driven off by the reducing flame, producing a green, but, when selenium is present, a bluish-green appearance.



*Arsenic.*—The charcoal is covered with arsenious acid, which is white,—in thin layers, greyish, (from the charcoal being seen through it),—and is deposited at a long distance from the assay. It flies off, when treated in either flame.

*Antimony.*—Covers the charcoal with an oxide which is white, in thin laminæ bluish, and is deposited at a shorter distance from the assay than the arsenical vapour; when treated in the oxidating flame, it is driven from place to place unchanged, but in the reducing flame it changes its position, producing a feeble bluish green colour. The antimonial sublimate is not so volatile as the arsenical, so that by this means they can be readily distinguished.

*Bismuth.*—Covers the charcoal with an oxide. The sublimate is dark orange-yellow while hot, lemon-yellow when cold, and when in thin layers bluish; it is not deposited nearly so far from the assay as the antimonial sublimate; it can be driven from place to place either by the oxidating or the reducing flame. It gradually diminishes, a portion of it being volatilized.

*Lead.*—Covers the charcoal with an oxide at the same distance from the assay as bismuth. The sublimate is, while hot, of a dark citron-yellow, upon cooling, sulphur-yellow, and when in thin laminæ, bluish; in other respects, it behaves like the sublimate from bismuth.

*Cadmium.*—Covers the charcoal with an oxide at the same distance from the assay as bismuth. The sublimate is best seen when cold; its colour is reddish brown, in thin films yellow: it can be volatilized in either flame.

*Zinc.*—Covers the charcoal with oxide, which is not at all so far from the assay as that of bismuth or lead. When hot, it is yellow and strongly phosphorescent; when cold, nearly white. It does not volatilize in the oxidating flame. A drop of nitrate of cobalt added to it in the oxidating flame, colours it beautifully green, which is best seen when cold.



*Tin.*—Covers the charcoal with oxide. This oxide is feebly yellow, and moderately phosphorescent when hot ; on cooling it is white, and almost touches the assay. It assumes, with cobalt solution, a bluish-green colour, which may be readily distinguished from that produced with oxide of zinc.

*Silver.*—If heated alone in the oxidating flame, it covers the charcoal with a slight dark-red precipitate. In combination with a little lead, it produces at first, a yellow sublimate of oxide of lead ; but when the silver becomes more minutely disseminated, a dark red sublimate appears beyond the yellow oxide of lead. If the silver contains a little antimony without lead, only a slight red sublimate is obtained. If it contains both lead and antimony, it assumes a carmine red colour, after the greater part of these metals are volatilized.

*Sulphur, Chlorine, Bromine, and Iodine.*—In examinations with the blowpipe, the operator does not always encounter the pure substances, which are recognized by the characteristic sublimates they yield, when heated upon charcoal,—but sometimes meets with combinations which deposit a white sublimate, admitting of being driven from place to place in the oxidating and reducing flames, thus resembling oxides of tellurium or antimony ; these are particularly the following:—sulphurets of potassium and sodium ; chlorides of potassium, sodium, ammonium, lead, and mercury ; bromides of potassium and sodium, and iodides of potassium and sodium. The most volatile of these combinations are the chlorides of ammonium, mercury, and lead ; the first two volatilize without melting, the others require to be strongly treated in the reducing flame, and the majority of them are absorbed by the charcoal before sublimation takes place. The sublimates from the most volatile are generally deposited at some distance from the assay, while those of the less volatile lie nearer the assay, and are more difficultly displaced by the flame. These sublimates are distinguishable from those of the oxides of tellurium and antimony, in as much as they disappear in the reducing flame without producing any colour.



¶ *Qualitative Examination of Minerals, Ores, and the Products of Metallurgic operations for Metallic and Non-metallic Bodies, before the Blowpipe.*

(a) EXAMINATION OF THE METALLIC OXIDES WHICH FORM ALKALIES AND EARTHS.

§ 1. POTASH—[K O].—*Presence in the Mineral Kingdom.*

Potash is found in combination with acids only, as for example :—

(a) With Sulphuric Acid, as well *per se*, as in combination with Earths and Water, namely, *per se*, in *Sulphate of Potash* [KO, SO<sup>3</sup>]; with Sulphate of Alumina, in *Alum* [KO, SO<sup>3</sup> + Al<sup>2</sup>O<sup>3</sup>, 3 SO<sup>3</sup> + 24 aq]; and with Sulphates of Lime and Magnesia, in *Potasso-Polyhallite* [KO, SO<sup>3</sup> + Mg, SO<sup>3</sup> + 2 (CaO, SO<sup>3</sup>) + 2 aq].

(b) With Nitric Acid, in *Saltpetre* [KO, NO<sup>5</sup>], but generally in combination with Sulphate and Carbonate of Lime, and Chloride of Calcium, and,

(c) With Silicic Acid, in various silicates of different bases, as for example, in *Felspar* [KO, SiO<sup>3</sup> + Al<sup>2</sup>O<sup>3</sup>, 3 SiO<sup>3</sup>]; *Leucite* [3 KO, 2 SiO<sup>3</sup> + 3 Al<sup>2</sup>O<sup>3</sup>, 2 SiO<sup>3</sup>]; *Apophyllite* [KO, 2 SiO<sup>3</sup> + 8 (CaO, SiO<sup>3</sup>) + 16 aq], *Potash-Tourmaline* (*Schorl*); and in some micaceous minerals, &c.

*Examination for Potash.*

The easily-fusible salts of potash, with the exception of the phosphate and borate, are recognized when held in the points of the forceps, no soda being present, by the intense violet colour given to the outer flame.

If the mineral contains a small quantity only of potash, no colouring will ensue, unless the splinter of it is held properly in the apex of the flame; great care must also be taken to have the assay operated upon, free from all extraneous matters.

When soda or lithia is also an ingredient of the mineral, this mode is not applicable, as the flame will be coloured yellow by the former, and red by the latter. Harkort resorts to the following method for the determination of potash,



when the quantity of this alkali is not too small. Oxide of nickel is dissolved in borax, until the glass upon cooling assumes a dark brown colour; to this glass is then added a portion of the substance under examination, and the mixture is treated in the oxidating flame; if the glass upon cooling retains its brown colour, potash is either absent or in too minute a quantity to be detected; but if, on the other hand, the bead acquires a bluish tint, similar to a diluted solution of oxide of nickel in ammonia, it indicates the presence of this alkali. Lampadius observes, that the potash and oxide of nickel glass is blue, while the oxide of nickel and soda glass is brown.

When substances, for instance native silicates, contain only traces of potash, the above method cannot with certainty be resorted to; therefore, the moist way must also be applied, as subsequently described: 50 to 75 milligrammes of the fine powdered body are mixed with twice their volume of soda, and pressed into a cavity in a plate of charcoal, and smelted in the oxidating flame: if much magnesia or lime is present, the assay will fuse with difficulty. The ignited mass is now removed from the support with the platinum pincers, and any adhering carbonaceous matter burnt away. The mass is pulverized between paper in an agate mortar, then treated in a porcelain basin with hydrochloric acid, and evaporated to dryness over the flame of the lamp. Water is now added, to dissolve the formed metallic chlorides, and after some time, the solution is poured from the undissolved silica into another basin. This liquid is evaporated until it becomes highly concentrated, and then a few drops of an alcoholic solution of chloride of platinum and sodium are added. If potash is present, the double salt of chloride of platinum and potassium, which is insoluble in alcohol, will precipitate as a lemon yellow powder. If there is only a small quantity of alkali present, the precipitate will not be perfectly visible for some minutes.\*

\* [The presence of organic matters does not prevent this reaction.]—*Trans.*



In combinations of sulphuric acid, which contain both potash and soda,—as, for instance, Polyhallite,—it is only requisite to reduce on charcoal the sulphate to a sulphuret, and treat with hydrochloric acid, water, &c., as in the previous example.

By the preceding methods, one per cent. of potash in silicates, and other substances, can be recognized.

Berzelius has given a similar examination, in one of his treatises upon the quantitative separation of potash from soda.\*

§ 2. SODA—[NaO].—*Presence in the Mineral Kingdom.*

Soda is never found *per se*, but always in combination, as,—

(a) With Chlorine, in *Rock Salt* [NaCl]—(Chloride of Sodium);

(b) With Fluorine and Fluoride of Aluminum, in *Kryolite* [ $3(\text{NaFl}) + \text{Al}^2\text{Fl}^3$ ];

(c) With Carbonic Acid and Water, in *Natron* [NaO,  $\text{CO}^2 + 10\text{aq}$ ], and *Trona (Urao)* [ $2\text{NaO}, 3\text{CO}^2 + 4\text{aq}$ ]; also with Carbonic Acid, Carbonate of Lime, and Water, in *Gaylussite* [ $\text{CaO}, \text{CO}^2 + \text{NaO}, \text{CO}^2 + 6\text{aq}$ ];

(d) With Sulphuric Acid, in *Glauber Salt* [NaO,  $\text{SO}^3 + 10\text{aq}$ ], which is often contaminated with carbonate of soda, chloride of sodium, and chloride of calcium;

(e) With Sulphuric Acid and Sulphate of Lime, in *Glauberite* [NaO,  $\text{SO}^3 + \text{CaO}, \text{SO}^3$ ];

(f) With Nitric Acid, in *Nitrate of Soda* [NaO,  $\text{NO}^5$ ];

(g) With Boracic Acid and Water, in *Borax (Tincal)* [NaO,  $2\text{BO}^3, + 10\text{aq}$ ]; and,

(h) With Silicic Acid, in various Silicates,—as for example, in *Tetartine* or *Albite (Soda Felspar)* [NaO,  $\text{SiO}^3 + \text{Al}^2\text{O}^3, 3\text{SiO}^3$ ]; in *Sodalite* from Vesuvius [NaCl +

\* [Oxalate of nickel, when melted with borax, gives a reddish yellow transparent glass, which, on the addition of a substance containing potash, becomes opaque, and of a dirty blue colour. This reaction is not prevented by the other alkalies, and therefore is a very characteristic test of the presence of potash.]—*Trans.*



$2 (\text{Al}^2 \text{O}^3) + 2 (3 \text{NaO}, \text{Si O}^3) + 2 (\text{Al}^2 \text{O}^3, \text{Si O}^3) ]$ ; in *Achmite*  $[ 3 (\text{NaO}, \text{Si O}^3) + 2 \text{Fe}^2 \text{O}^3, \text{Si O}^3 ]$ ; in *Soda Spodumene* (*Oligoklas*)  $3 (\text{NaO}, \text{Si O}^3) + 4 \text{Al}^2 \text{O}^3, 2 \text{Si O}^3 ]$ ; in *Nepheline*  $[ \frac{2 \text{NaO}}{2 \text{KO}} \} \text{Si O}^3 + 2 (\text{Al}^2 \text{O}^3, \text{Si O}^3) ]$ ; in *Labradorite*  $[ \text{NaO}, \text{Si O}^3 + \text{Al}^2 \text{O}^3, \text{Si O}^3 + 3 (\text{CaO}, \text{Si O}^3 + \text{Al}^2 \text{O}^3, \text{Si O}^3) ]$ ; in *Soda Tourmaline*; in *Analcime*  $[ 3 \text{NaO}, 2 \text{Si O}^3 + 3 (\text{Al}^2 \text{O}^3, 2 \text{Si O}^3) + 6 \text{aq} ]$ ;—&c. &c.

*Examination for Soda.*

When a soda compound is smelted in the apex of the blue flame, the soda is readily recognized, from the reddish yellow (according to Merlet, wax-yellow) colour given to the outer flame. The flame partakes of this colour, but in a less degree, even when the body contains a large quantity of potash or lithia. According to Von Kobell, when chloride of potassium is mixed with 1-25th or 1-30th part of chloride of sodium, only the soda reaction is given.

The splinter from the soda mineral is held like that of potash, in the points of the platinum forceps, when the soda reaction ensues.

§ 3. LITHIA—[ Li O ].—*Presence in the Mineral Kingdom.*

This alkali is found always in combination, as in,—

(a) *Amblygonite*, which is a Phosphate of Alumina and Lithia, mixed sometimes with a Fluorine compound; its formula is  $[ 2 \text{Li O}, \text{P O}^5 + 4 \text{Al}^2 \text{O}^3, 3 \text{P O}^5 ]$ ;

(b) Many Silicates of different bases; as, for example, in *Petalite*  $[ \frac{3 \text{Li O}}{3 \text{NaO}} \} 4 \text{Si O}^3 + 4 (\text{Al}^2 \text{O}^3, 4 \text{Si O}^3) ]$ , it contains also hydrofluoric acid; *Lithion Spodumene*  $[ 3 \text{Li O}, 2 \text{Si O}^3 + \text{Al}^2 \text{O}^3, 2 \text{Si O}^3 ]$ ; *Tourmaline*, *Lepidolite*, and many other Micas which contain hydrofluoric acid.

*Examination for Lithia.*

Compounds of lithia, when heated in the apex of the blue flame, upon the platinum wire, tinge the outer flame crimson. This characteristic appearance is best shown with the chloride of lithium. When the chloride is mixed with a salt of



potash, the red colour alone is produced. When, on the contrary, the salt of lithia is contaminated with a salt of soda, then the reaction of soda only is observable, and this is also the case when a lithia salt contains both potash and soda salts.

Lithion minerals, as for example, lithion mica from Altenberg and Zinnwald, give, when heated in the apex of the blue flame, an intense carmine colour; but if the alkali is in very small quantities only, the reaction will be indistinct.

Turner has given the following method for detecting traces of lithia in silicious minerals. Knead into a paste with water, one part of fluor spar, one and a half part of bisulphate of potash, and a portion of the fine powdered mineral for investigation, then smelt the mixture in the ring of the platinum wire, in the apex of the blue flame. If the body contains lithia, the outer flame will assume a crimson tinge; if not, it will be coloured violet, from the presence of potash. Merlet employs for this test, one part of the fine powdered mineral, and two parts of the mixture of fluor spar and bisulphate of potash. The amblygonite from Chursdorf, in Saxony, which contains 11 per cent. of lithia, gives to the outer flame an intense carmine colour, provided that soda is not present. If soda is present, the outer flame will be coloured more yellow than red. With bisulphate of potash and fluor spar, if only traces of lithia are contained therein, the red reaction will be given; but here if soda is also present, its reaction will prevail.

§ 4. *AMMONIA*—[ $\text{NH}^3$ ].—*Presence in the Mineral Kingdom.*

Ammonia is not met with in a free state, but in combination with chlorine and acids only, namely,

(a) With Chlorine, in native *Sal-ammoniac* [ $\text{NH}^4, \text{Cl}$ ] (chloride of ammonium.)

(b) With Sulphuric Acid, in *Mascagnin* (sulphate of ammonia,) [ $\text{NH}^4 \text{O}, \text{SO}^3 + \text{aq}$ ]; and,

(c) With Sulphuric Acid, Alumina, and Water, in *Ammon-alum* [ $\text{NH}^4 \text{O}, \text{SO}^3 + \text{Al}^2 \text{O}^3, 3 \text{SO}^3 + 24 \text{aq}$ ].



*Examination for Ammonia.*

The ammoniacal salts are very easily recognized, by treating them in a glass tube, mixed with carbonates of soda, potash, or lime, over a spirit-lamp. Carbonate of ammonia sublimes, which is known by its odour, and also by its colouring reddened litmus-paper blue. Its presence is infallibly shewn by holding over the mouth of the tube, a glass rod moistened with hydrochloric acid, for if a trace only of ammonia is eliminated, a white cloud will be produced.\*

§ 5. BARYTES—[Ba O].—*Presence in the Mineral Kingdom.*

Barytes is found:—

(a) As a Carbonate, in *Witherite* [BaO, CO<sup>2</sup>]; and, in combination with Carbonate of Lime and Manganese, in *Baryto-calcite* [4 (BaO, CO<sup>2</sup>) + 4 (CaO, CO<sup>2</sup>) + MnO, CO<sup>2</sup>];

(b) As a Sulphate, in *Heavy Spar* (Sulphate of Barytes) [BaO, SO<sup>3</sup>]; and, in combination with Sulphate of Lime, in *Calcareous Heavy Spar* [BaO, SO<sup>3</sup> + CaO, SO<sup>3</sup>];

(c) In Silicic Acid combinations, containing Barytes, Alumina, and also Water,—as in *Barytes Harmotome* [3 Ba O, 2 SiO<sup>3</sup> + 5 (Al<sup>2</sup>O<sup>3</sup>, 2 SiO<sup>3</sup>) + 24 aq]; and in a Silicic Acid compound containing Barytes, Strontian, Alumina, and Water, as *Brewsterite* [3 ( $\begin{smallmatrix} \text{Sr} & \text{O} \\ \text{Ba} & \text{O} \end{smallmatrix}$ ) SiO<sup>3</sup> + 4 (Al<sup>2</sup>O<sup>3</sup>, 3 SiO<sup>3</sup>) + 18 aq].

*Heavy Spar* sometimes forms a constituent in metallic gangues; it is therefore necessary to examine for Barytes in the Ores, and also in the Slags produced in smelting them.

*Examination for Barytes.*

(a) *Witherite* is readily recognized before the blowpipe, as it behaves like pure Barytes.

*Baryto-calcite*, *per se*, is quite infusible. It becomes, however, when strongly ignited, alkaline. A sample from

\* [When dry salts of ammonia are triturated in an agate mortar with the caustic alkalies, or alkaline earths, the well-known penetrating odour of ammonia becomes immediately perceptible.]

None of the ammoniacal compounds can sustain an incipient red heat, without being volatilized or decomposed. This arises from the gaseous nature of the alkali.]—*Trans.*



Cumberland which I examined, gave, when strongly heated in the reducing flame, and moistened, no stain to silver.

It dissolves, with tumefaction, in borax, and forms a coloured glass like manganese, which, in the reducing flame, becomes diaphonous.

It is dissolved in microcosmic salt, under powerful vesiculation, to a pellucid glass, which, if more strongly saturated, assumes the manganesian colour. It is transparent in the reducing flame, and opaque when cold.

When heated with soda, it is decomposed; the barytes and soda are absorbed by the charcoal, and the lime and protoxide of manganese remain behind.

(*b*) Heavy Spar, preferable in a powdered state, as it decrepitates very strongly, when heated upon charcoal in the reducing flame, is converted into sulphuret of barium, which gives off, when moistened, an hepatical smell, and possesses an astringent taste.

With soda, it melts to a pellucid limpid glass, which boils when strongly heated, and becomes absorbed as a strong hepatical mass, by the charcoal.

As Celestine (Sulphate of Strontian) behaves in a similar manner, the assay (in order to ascertain which is present), must be treated with hydrochloric acid and alcohol, as will be described under the head of Strontian. The chloride of barium does not give a coloured flame. The heavy spar is distinguished from Celestine by the former, *per se*, being difficultly fusible, while the latter can readily be melted to a globule. From Celestine it is also distinguished by colouring the outer flame yellowish, when held with a forceps in the apex of the blue flame.

Calcareous Heavy Spar, *per se*, and with soda, behaves like Heavy Spar; with this difference, that when heated with soda, the lime is not likewise absorbed by the charcoal, but remains as an infusible mass. This phenomenon is better remarked during the ignition, the lime running to various parts of the charcoal, and appearing slightly phosphorescent.

(*c*) In Barytes-Harmotome, and in Brewsterite, the barytes cannot be distinguished alone or with glacial fluxes. It is



therefore necessary to proceed as follows :—Smelt one part of the powdered sample, with one part of soda, and one part of borax, upon charcoal, to a bead. Pulverize the smelted assay, treat with hydrochloric acid, evaporate to dryness, and dissolve in an excess of water. Filter, to separate the silica, and add to the filtrate diluted sulphuric acid, or bisulphate of potash, to precipitate the barytes and strontian. Filter, wash, and reduce the residue to sulphurets of barium and strontium, then treat with hydrochloric acid and alcohol, to determine whether the two are present. If the alcohol flame is coloured red, strontium is present. The alumina, which is not precipitated by bisulphate of potash, or diluted sulphuric acid, may be separated from the solution in the manner described at page 109 (Note.)

In assorted ores and slags, barytes cannot be detected by the blowpipe alone; it is therefore always convenient to have recourse to the moist way.\* Such substances, after previously roasting those which contain arsenic or metallic sulphurets, must be melted (according to page 65), with one part soda and one part borax; and, if some difficultly-reducible metals are present, 200 or 300 milligrammes proof lead, or 80 to 100 milligrammes of fine silver, must be added. The fused assay is treated with hydrochloric acid and water, (see page 67). To this liquid, from which the silica has been separated by filtration, bisulphate of potash or diluted sulphuric acid is added, to precipitate the barytes. According to this method, it can be confounded with sulphates of strontian and lime only. But if the substance is very dilute, sulphates of barytes and strontian only are precipitated, as sulphate of lime is soluble in an excess of water. The precipitate is now affused on the filter with water, and dried; if barytes only is present, after treating the residue left on the filter with hydrochloric acid and alcohol, and igniting, no red colour will be imparted to the flame of the spirit. If coloured red, the two earths are contained therein. The other ingredients in

\* Very dilute sulphate of soda precipitates barytes and strontian, but not lime; hyposulphate of soda precipitates barytes, and not strontian.]—*Trans.*



the solution are detected as stated under Lime, Magnesia, and Alumina, in their silicic acid combinations.

§ 6. *STRONTIAN*—[SrO].—*Presence in the Mineral Kingdom.*

(a) Strontian occurs as a Carbonate in *Strontianite*, [SrO CO<sup>2</sup>]; it is also a very small ingredient of *Arragonite* (*Carbonate of Lime*), and, with Sulphate of Barytes and a trace of Carbonate of Lime, in *Stromnite*.

(b) As a Sulphate, in *Celestine*, [SrO, SO<sup>3</sup>]; and sometimes in *Heavy Spar*.

(c) In a Silicic Acid combination of Barytes, Strontian, Alumina, and Water, as *Brewsterite*. If such minerals occur in metalliferous gangues, a very minute quantity of Strontian may be detected in the dressed ores, and in the slags produced therefrom.

*Examination for Strontian.*

Strontianite alone, and with fluxes, comport itself before the blowpipe-flame, like the pure earth. If dissolved in hydrochloric acid, and the solution evaporated slowly to dryness, the dried residue then burned with alcohol, the intense characteristic carmine colour will be visible.

The pyrognostic properties of Arragonite\* will be mentioned under Lime.

Stromnite is most readily determined as follows:—The pulverized sample is treated on charcoal in the reducing flame, and the presence of strontian afterwards recognized by the tint communicated to the flame of alcohol, as previously stated.

Celestine fuses to an opalescent glass, and colours the outer flame carmine-red, when heated in a pair of forceps. When the glass is treated on charcoal, the sulphate of strontian is reduced to sulphuret, and when moistened, behaves towards silver like sulphuret of barium. Celestine with soda behaves like heavy spar. When treated like Strontianite, it imparts a beautiful crimson hue to the alcoholic flame.

\* [When heated to incipient redness, it tumefies, forms a light white powder, and a very small quantity of water collects in the beak of the matrass.]—*Trans.*



The casual presence of Strontian in Heavy Spar, is determined by reducing to sulphurets, and treating with hydrochloric acid and alcohol; the strontian is recognized by the colour imparted to the spirit when inflamed.

When Chloride of Strontium is treated on the ring of the platinum wire, in the apex of the blue flame, the whole flame is immediately perceived of a deep crimson; however, when the assay is fused, the red colour vanishes, and thus the chloride of strontium is distinguishable from the chloride of lithium. The presence of chloride of barium, combined with chloride of strontium, prevents the appearance of the red colour in the outer flame.

Substances which neither colour the outer blowpipe-flame nor the flame of alcohol, such as Brewsterite, assorted ores, slags, &c., must be transposed to a state in which the strontian is separated from the other ingredients, and then investigated. The procedure is conducted in a manner similar to that given under barytes. When chloride of strontium contains chloride of calcium, it can be discovered by treating the mixture with soda upon charcoal. The strontian (and any barytes) is absorbed, while the lime remains upon the surface of the support as an infusible mass.

Chloride of calcium, when heated on the ear of the platinum wire, communicates a red tinge to the outer flame. The colour, however, is more feeble with the lime salt than with the chloride of strontium.\*

§ 7. LIME—[Ca O].—*Presence in the Mineral Kingdom.*

Lime is met with—

- (a) As Fluoride of Calcium, in *Fluor Spar* [Ca Fl];
- (b) As Carbonate of Lime, [Ca O, CO<sup>2</sup>] in *Chalk, Lime-*

\* [A solution of sulphate of lime precipitates salts of barytes, but not of strontian. The sulphate of lime is perfectly soluble in hydrochloric acid, which distinguishes it from the sulphates of barytes and strontian. Winkelblech remarks, that chromate of potash readily gives a yellowish precipitate of chromate of barytes, with solutions of the salts of this earth. The precipitate is *insoluble in Sal-ammoniac*, (chloride of ammonia).

A yellowish precipitate is also produced when chromate of potash is boiled with a soluble salt of strontian.]—*Trans.*



stone, *Calcareous Spar*, *Kalk Tuff* (*Tufa*), *Rock Milk* (*Bergmilch*), *Schiefer Spar* (*Slate Spar*), *Anthraconite*, *Stinkstone*\*, *Marl*, and in bituminous marly slate; further, with a trace of Carbonate of Strontian, in *Arragonite*, also with carbonic acid, in *Dolomite* (bitter spar)  $[CaO, CO^2 + 3(MgO, CO^2)]$  &c., &c., and with Carbonate of Barytes, in *Baryto-calcite*;

(c) As Sulphate, in *Gypsum* (*Selenite*)  $[CaO, SO^3 + 2aq]$ , and *Anhydrite*  $[CaO, SO^3]$ ; further, with Sulphate of Potash and Sulphate of Magnesia, in *Polyhallite*; with Sulphate of Soda, in *Glauberite*, and with Sulphate of Barytes, in *Calcareous Heavy Spar*;

(d) As Phosphate of Lime, with Chloride of Calcium,—the latter being sometimes more or less replaced by Fluoride of Calcium,—in *Apatite*†  $[Ca \left\{ \begin{smallmatrix} Cl \\ Fl \end{smallmatrix} + 3(3CaO, PO^5) \right\}]$ ; or,  $[CaCl + 3(3CaO, PO^5)] = [CaFl + 3(3CaO, PO^5)]$ ;

(e) As Arseniate of Lime, in *Pharmacolite*‡  $[2CaO, AsO^5 + 6aq]$ , and *Hardingerite*  $[2CaO, AsO^5 + 4aq]$ ;

(f) As Tungstate of Lime, in *Schwerstein*  $[CaO, WO^3]$ ;

(g) As Titanite and Silicate of Lime, in *Titanite* and *Sphene* (brown and yellow *Menaccanite*)  $[CaO, 3TiO^2 + CaO, 2SiO^3]$ , and  $[CaO, 2TiO^2 + 2(CaO, SiO^3)]$ ;

(h) As Tribasic Tantalate of Lime and Yttria, containing traces of proto-tungstate of iron, lime, and oxide of uranium, in Dark *Yttro-tantalite*  $\left[ \begin{smallmatrix} 3Y \\ 3Ca \end{smallmatrix} O \right\} TaO^3$ ]; and with a considerable quantity of tribasic proto-tungstate of iron, in Black *Yttro-tantalite*  $\left[ \begin{smallmatrix} 3Y \\ 3Ca \\ 3Fe \end{smallmatrix} O \right\} + \left\{ \begin{smallmatrix} Ta \\ W \end{smallmatrix} O^3 \right\}$ ]: the Yellow *Yttro-tantalite* containing a large proportion of peroxide of uranium, its formula is  $[3YO \left\{ \begin{smallmatrix} Ta \\ U \end{smallmatrix} O^3 \right\}]$ ;

(i) As neutral and bibasic Borate of Lime, with Bi-silicate of Lime, in *Datolite*  $[CaO, 2BO^3 + CaO, 2SiO^3 + aq]$ ,

\* [From the strongly-fetid odour it exhales when slightly rubbed.]

† [It was named by Werner, from *απαταω*, to deceive; in allusion to its being easily mistaken for certain other minerals.]—*Trans.*

‡ [From the Greek; in allusion to its containing a poisonous ingredient.]—*Trans.*



and *Botryolite*\* [ 2 ( 2 Ca O, 2 BO<sup>3</sup> ) + 2 ( Ca O, 2 Si O<sup>3</sup> ) + 2 aq ] ;

(k) In Silicates; and indeed :—

(1) In a Silicate where the Lime alone is in combination with the acid, namely, in *Tabular Spar* (*Wollastonite*), [ 3 Ca O, 2 Si O<sup>3</sup> ] ;

(2) In Silicates of Potash, Lime and Water,—for example, *Apoklas* (*Apophyllite*) [ 9 {  $\frac{\text{Ca O}}{\text{K O}}$  } Si O<sup>3</sup> + 16 aq ] ; which generally contains hydrofluoric acid ;

(3) In Silicate of Lime, or Lime and Alkalies, with Silicate of Alumina and water of crystallization ; *e. g.*, in—

*Stilbite*, [ 3 Ca O, Si O<sup>3</sup> + 3 ( Al<sup>2</sup> O<sup>3</sup>, Si O<sup>3</sup> ) + 18 aq ] ;

*Desmine* [  $\frac{\text{Na O}}{\text{Ca O}}$  } 3 Si O<sup>3</sup> + 3 ( Al<sup>2</sup> O<sup>3</sup>, 3 Si O<sup>3</sup> ) + 6 aq ] ;

*Heulandite* [ 3 ( Ca O, Si O<sup>3</sup> ) + 4 ( Al<sup>2</sup> O<sup>3</sup>, 3 Si O<sup>3</sup> ) + 18 aq ] ;

*Laumonite* [ 3 Ca O, 2 Si O<sup>3</sup> + 3 ( Al<sup>2</sup> O<sup>3</sup>, 2 Si O<sup>3</sup> ) + 12 aq ] ;

*Scolezite* [ Ca O, Si O<sup>3</sup> + Al<sup>2</sup> O<sup>3</sup>, Si O<sup>3</sup> + 9 aq ] ;

*Prehnite*† [ 2 ( 3 Ca O, Si O<sup>3</sup> ) + 3 ( Al<sup>2</sup> O<sup>3</sup>, Si O<sup>3</sup> ) + 3 H O, Si O<sup>3</sup> ] ; *Potash Harmotome* [ 3 K O, 2 Si O<sup>3</sup> + 2 ( 3 Ca O, 2 Si O<sup>3</sup> ) + 10 ( Al<sup>2</sup> O<sup>3</sup>, 2 Si O<sup>3</sup> ) + 45 aq ] ; &c.

(4) In Silicates without water ; *e. g.*,—

*Labradorite* [  $\frac{3 \text{ Na O}}{3 \text{ Ca O}}$  } Si O<sup>3</sup> + 3 ( Al<sup>2</sup> O<sup>3</sup>, 2 Si O<sup>3</sup> ) ] ;

*Scapolite* (*Wernerite*) [  $\frac{3 \text{ Fe O}}{3 \text{ Ca O}} \left\{ \frac{3 \text{ Mg O}}{3 \text{ Na O}} \right\} 2 \text{ Si O}^3 + \left\{ \frac{\text{Al}^2 \text{ O}^3}{\text{Fe}^2 \text{ O}^3} \right\} 2 \text{ Si O}^3 ] ;$

*Anorthite* [ 3 {  $\frac{3 \text{ Ca O}}{3 \text{ Mg O}}$  } Si O<sup>3</sup> + 8 ( Al<sup>2</sup> O<sup>3</sup>, Si O<sup>3</sup> ) ] ;

(5) In Silicates of Lime and Magnesia, in which the alkaline earths are more or less replaced by Protoxides of Manganese and Iron, and the Silicic acid, sometimes by Alumina. To these belong :—

(a) *Amphibole* (*Hornblende*), which, according to Bonsdorff, contains hydrofluoric acid, which, with Lime, forms fluoride of calcium. Berzelius gives it the following general

\* [ From the Greek, owing to the resemblance in form to grapes. ]—*Trans.*

† [ From Colonel Prehn, its discoverer. ]—*Trans.*



formula [ Ca Fl + 5 ( Ca O, Si O<sup>3</sup> + 3 Mg O, 2 Si O<sup>3</sup> ) ]; *Tremolite* or *Grammatite*, *Asbestos Tremolite*, *Asbestos*, *Asbestiform Actynolite*, *Byssolite*, the *Hornblendes*, &c. Further:—

(b) Pyroxenes; *e. g.*, *Diopside* or *Alalite*, white *Malacolite*, *Sahlite*, are represented by the formula [ 3 Ca O, Si O<sup>3</sup> + 3 Mg O, 2 Si O<sup>3</sup> ]; also

(c) The Ferriferous Pyroxenes, as *Ferriferous Malacolite* [ 9 Ca O, 6 Si O<sup>3</sup> + {  $\frac{3 \text{ Fe O}}{3 \text{ Mg O}}$  } + 2 Si O<sup>3</sup> ]; the *Hedenbergite* [ {  $\frac{3 \text{ Ca O}}{3 \text{ Fe O}}$  } + 3 Si O<sup>3</sup> ]; and the *Lievrite* (*Yenite*) [ 3 Ca O, Si O<sup>3</sup> + 4 ( 3 Fe O, Si O<sup>3</sup> ) ];

(6) In Silicates of Lime and Magnesia, with Alumina, in which the Alumina is often replaced by Iron and Manganese, *e. g.* in *Zoisite* [ 2 ( 3 Ca O, Si O<sup>3</sup> + 5 {  $\frac{\text{Al}^2 \text{ O}^3}{\text{Fe}^2 \text{ O}^3}$  } Si O<sup>3</sup> ); *Pistacit* (*Epidote*) [ 3 Ca O, Si O<sup>3</sup> + 4 {  $\frac{\text{Al}^2 \text{ O}^3}{\text{Fe}^2 \text{ O}^3}$  } Si O<sup>3</sup> ]; *Idiocrase* (*Vesuvian*), *Egeran*, and many Granates, the general formula for which is [ {  $\frac{3 \text{ Mg O}}{3 \text{ Fe O}} \frac{\text{O}}{\text{O}}$  } {  $\frac{3 \text{ Ca O}}{3 \text{ Mn O}}$  } } Si O<sup>3</sup> + Al<sup>2</sup> O<sup>3</sup>, Si O<sup>3</sup> ]; *Gehlenite* [ {  $\frac{3 \text{ Ca O}}{3 \text{ Mg O}} \frac{\text{O}}{\text{O}}$  } + {  $\frac{2 \text{ Si O}^3}{2 \text{ Al}^2 \text{ O}^3}$  } ]; &c., &c.

(7) In Silicates of Yttria, Oxide of Cerium, Alumina, Lime, Iron, and Manganese, namely, in *Cerine*, which contains Cerium, Silicic acid, Iron, Lime, Alumina, Titanic acid, Manganese, and Water; *Allanite* [ 3 Fe O, Si O<sup>3</sup> + 3 Ce O, Si O<sup>3</sup> ) + 3 Ca O, Si O<sup>3</sup> + 2 Al<sup>2</sup> O<sup>3</sup>, Si O<sup>3</sup> ]; *Orthite* (a

species of *Serpentine*) 4 [ {  $\frac{3 \text{ Ca O}}{3 \text{ Fe O}} \frac{\text{O}}{\text{O}}$  } {  $\frac{3 \text{ Mn O}}{3 \text{ Ca O}} \frac{\text{O}}{\text{O}}$  } } Si O<sup>3</sup> + 3 ( Al<sup>2</sup> O<sup>3</sup>, Si O<sup>3</sup> )

+ 9 aq ]; and *Pyrorthite* [ 3 Ce O, Si O<sup>3</sup> + 3 ( Al<sup>2</sup> O<sup>3</sup>, Si O<sup>3</sup> ) + Carbon, Lime, Yttria, Manganese, Iron, &c.

(8) In poly-basic Silicates, with one or more Borates mixed with them, *e. g.*, *Axinite* [ 2 ( {  $\frac{3 \text{ Ca O}}{3 \text{ Mg O}} \frac{\text{O}}{\text{O}}$  } , Si O<sup>3</sup> + 2 {  $\frac{\text{Al}^2 \text{ O}^3}{\text{Fe}^2 \text{ O}^3} \frac{\text{O}^3}{\text{O}^3}$  } Si O<sup>3</sup> ) + {  $\frac{\text{B O}^3}{\text{Si O}^3}$  } ]; and lastly,—



(9) In Silicates of Soda, Lime, and Alumina, mixed with Sulphates; *e. g.*, *Lapis Lazuli*,\* *Nosin (Spinellane)* [ $\text{Na O}$ ,  $\text{Si O}^3 + 3 (\text{LO}, \text{Si O}^3) + 6 (\text{Al}^2 \text{O}^3, 2 \text{Si O}^3)$ ], &c.

Leading gangues of ores very often eviscerate Calcareous and Fluor Spars; lime more frequently forms a principal component part of several ores, which are assayed in the dry way, and also an ingredient of the slags, scoriæ, and recrements, obtained therefrom.

*Examination for Lime.*

Fluor spar is very readily recognized before the blowpipe. When slightly heated, it assumes a luminous appearance, which appears greenish in the dark. On the temperature increasing, it decrepitates. Heated on charcoal as an impalpable powder, it melts to an opaque glass.

Fluor spar can be recognized with the greatest degree of certainty, by heating it with Gypsum, Heavy Spar, and Celestine, whereby it melts in the flame to a clear bead, which is opalescent when cold.

If Calcareous Spar (Carbonate of Lime) be heated in the blowpipe flame upon charcoal, carbonic acid escapes, and during its evolution the lime acquires a peculiar lustre. If the residue, which is caustic lime, is treated with water, a great heat is produced, and the mass re-acts alkaline.† When strongly heated in the apex of the blue flame, upon the pincers, the Calcareous Spar, after being causticized, colours the

\* [Known as Lazurstein, Lazulite, and Azure Stone.

Kane, in treating of this mineral, gives its composition as follows:—

Silica . . . . .	35·8
Alumina . . . . .	34·8
Soda . . . . .	23·2
Sulphur . . . . .	3·1
Carbonate of Lime . . .	3·1

100·0

And remarks, that it is difficult to deduce a formula from these numerical results, as the state of combination of the sulphur is not well understood.]—*Trans.*

† [In this combination the temperature rises sufficiently high to inflame gunpowder.]—*Trans.*



outer flame carmine red, but much weaker than a strontian salt. This mineral behaves towards borax and microcosmic salt (in which fluxes it is dissolved with effervescence), as also towards soda, like Lime. The other varieties of Carbonate of Lime comport themselves similarly; but if they contain metallic oxides, they impart to the fluxes the characteristic colours of these oxides.

Arragonite, when smelted on charcoal, decomposes, and fusible particles remain. This residue re-acts alkaline on moistened reddened litmus paper, and when held in the apex of the blue flame, between the platinum forceps, the outer flame is coloured carmine-red, from the presence of Strontian. Arragonite dissolves, with effervescence, in borax and microcosmic salt, forming a clear glass. It is not at all effected by soda; the alkali is absorbed by the charcoal, and the lime assay remains on the surface.

Dolomite, Rantemspar, and Brown-spar, are infusible before the blowpipe; when freed from their carbonic acid they are alkaline. They dissolve in borax and microcosmic salt to a clear glass, generally coloured by oxide of iron and manganese, and are not at all effected by soda.

Lime and Magnesia are only recognized with certainty, before the blowpipe, by the assistance of the moist way, as will be described under Magnesia.

In Baryto-calcite the lime is determined as mentioned with Barytes.

Sulphuric Acid combinations :—

(a) Gypsum and Anhydrite are difficultly smeltable in the oxidating flame, to an enamelled glass. If the fusion is performed on charcoal in the reducing flame, they are reduced to sulphurets, which, when moistened, have an hepatic smell, and re-act alkaline with reddened litmus paper. With borax they dissolve with effervescence to a clear glass, which on cooling is yellow or dark yellow. When more Gypsum or Anhydrite is added, the bead is brown and opaque when cold. They behave with microcosmic salt and soda like pure lime. Gypsum and Anhydrite melt readily to a clear glass, which is enamel white on cooling; when the blowing is continued,



the bead tumefies and becomes infusible. The characteristics for distinguishing Gypsum from Heavy Spar and Celestine, are as follows:—1. The slight hardness of the Gypsum; 2. Its ready fusibility, in comparison with the Sulphate of Barytes; and, 3, principally, the comportment of Gypsum with soda, as regards the barytes and strontian, which are absorbed by the charcoal, while the lime remains.

(*b*) Polyhallite, when heated in a glass tube, liberates water; on charcoal it fuses to a clouded red globule, which in the reducing flame becomes white, and appears vesicular, its taste is saline and somewhat hepatic. Smelted in the ring of the platinum wire, it colours the outer flame yellow, from the presence of soda.

With borax it dissolves easily, with violent effervescence, to a transparent glass; but if a large quantity of the mineral has been added, the bead will be coloured by the iron, opaque on cooling. It is decomposed by soda, and produces an argillaceous mass, which in the reducing flame assumes a yellowish hue, from being contaminated with hepar, (sulphuret of antimony.) With fluor spar it melts to an unclear pearl.

Lime and magnesia are not readily separated by oxalate of ammonia and microcosmic salt, from an aqueous solution of the mineral, treated with hydrochloric acid. The contents of potash can only be found in this case according to the method given under the head of this alkali.

(*c*) Glauberite, when treated *per se* on charcoal, becomes at first white, and then melts to a transparent pearl. This also occurs in the reduction flame, and the assay becomes hepatic. By continuing the blast, the sulphuret of sodium is absorbed by the charcoal, and the lime remains.

When the powdered mineral is kneaded into a paste with water, and held on the ring of the platinum wire, in the oxidating flame, the outer sphere is coloured yellow, owing to the presence of soda.

When smelted with soda on charcoal, the assay is decomposed, and the alkali, with the reduced sulphuret of sodium, is absorbed by the support, and the lime remains behind.



(*d*) The pyrognostic properties of Calcareous Heavy Spar, have already been treated of under Barytes.

Apatite alone fuses with difficulty on the edges. It dissolves tardily in soda, forming a clear glass, which often partakes of the iron colour; when there is a slight excess of the mineral, the assay becomes turbidly streaked, and with a still greater addition, is opalescent when cold.

It dissolves in large quantities in microcosmic salt to a clear glass, which is, however, slightly tinged from the presence of oxide of iron. When slightly saturated, the bead becomes opaque on cooling, and exhibits facets, which are, however, less perceptible than those produced by phosphate of lead, when heated in the reduction flame and allowed to refrigerate. If the glass be perfectly saturated, a milk-white globule results, and no facets are observable.

With an equal part of soda, Apatite produces a vesicular infusible mass, and when more of the alkali is added, it is absorbed by the charcoal. Chlorine, phosphoric, and hydro-fluoric acids are determined according to the method given under their separate heads.

The presence of lime can only be ascertained with the assistance of the moist way. The substance should be dissolved in hydrochloric acid, the solution diluted with spirits of wine of about  $63^{\circ}$ , and the lime precipitated by weak sulphuric acid. The supernatant liquor can, after filtration, be examined with ammonia for alumina. As the compound of lime with phosphoric acid, is precipitated by ammonia unchanged, from a solution of the phosphate of lime diluted with water; consequently, alumina and peroxide of iron cannot first be precipitated by ammonia. It is necessary to separate the lime first, by means of dilute sulphuric acid.

Pharmacolite is readily discovered by tritulating with soda, and treating the mixture on charcoal in the reducing flame. The arsenic acid is reduced and volatilizes, which is ascertained by the smell. The soda is absorbed by the charcoal, and the lime remains behind.

Schwerstein (Tungstate of Lime) fuses on the edges to a partially translucent bead.



It dissolves readily in borax to a transparent glass, in the oxidating flame, which soon appears crystalline and milk-white. This assay is not coloured when mixed with tin and treated in the reducing flame.

It soon liquefies with microcosmic salt in the oxidating flame, forming a colourless glass, which in the reducing flame partakes while hot of a green colour, and on refrigeration of a beautiful blue. The glass when treated with tin on charcoal in the reducing flame, assumes a darker colour, which on cooling is green. By continued treatment in the reducing flame, with a sufficient addition of tin, the Tungsten separates, and the glass only contains a slight greenish hue.

With soda it tumefies and becomes rounded on the edges. If a small quantity of the pulverized mineral be smelted with four times its volume of soda, on a platinum wire, or in a platinum spoon, and the assay affused over the flame of a lamp with water, tungstate of soda, and the excess of alkali added, will be dissolved, and the lime collects at the bottom of the spoon. The tungstic acid is separated from this solution, according to the method given under the head of this acid, and the lime is determined with soda on charcoal.

Sphene and Titanite, alone before the blowpipe, slightly intumesce, and fuse on the edges to a dark glass.

With borax they readily melt to a yellowish diaphonous glass, but in the reducing flame the colour of titanium is not produced.

In microcosmic salt, Sphene and Titanite dissolve with difficulty; the undissolved part becomes opaline. The amethystine tinge is afforded, particularly when the assay is treated upon charcoal mixed with tin in the reducing flame. With soda they fuse to an opaque glass, but in no proportion give a transparent one; on cooling, the globule is white, or greyish white. When an excess of the alkali has been added, the greater part of the assay is absorbed by the charcoal, and the lime remains behind.

If Sphene or Titanite be smelted with six times its volume of bisulphate of potash, in a platinum spoon, and the melted mass affused over the flame of a lamp with a sufficient quan-



tity of water, (the liquid must not be allowed to boil), the titanitic acid and sulphate of lime dissolves, leaving a siliceous residue. The titanitic acid deposits from the filtered solution upon boiling, and the lime can be separated by oxalate of ammonia. Should there be any doubt as to the nature of the precipitates, they can be separately examined, after being well washed, before the blowpipe,—that is, the lime with soda on charcoal, and the titanitic acid according to the method given under the head of Titanium.

The siliceous deposit may contain titanitic acid, and a portion of sulphate of lime, if the mineral had not been previously reduced to a fine powder, or if an adequate quantity of water was not added.

The pyrognostic properties of Ytthro-tantalite are given under Yttria.

Datholite and Botryolite (both from Arendal, in Norway,) behave before the blowpipe, according to Berzelius, as follows:—

When treated alone in tubes, they evolve aqueous vapour. On charcoal they vesiculate like borax, and then melt into a transparent globule, which sometimes, from impurity or unequal limpidity, is either rose-red or iron-green. They dissolve readily in borax to a clear bead, without assuming the colours above mentioned.

With microcosmic salt they easily fuse, leaving behind a silicious compage; a greater addition of the assay, gives at first an opaque glass, and lastly an enamel white one.

They dissolve with a little soda to a clear glass; on a greater addition the bead is opaque on cooling, and with an excess, the whole assay is absorbed by the charcoal.

With gypsum they fuse with greater difficulty than Fluor Spar, to a clear globule, which remains so on cooling.

With a solution of cobalt, Datholite and Botryolite give an unclear blue glass. The lime is determined in these minerals by smelting with bisulphate of potash, and treating the glacial residue with an adequate quantity of water, and decomposing the clear solution, after separation of the silica, with oxalate of ammonia. The boracic acid is recognized by a special test.



In Silicates the aid of the moist way is always requisite for determining the lime, which is very seldom ascertained with certainty before the blowpipe.

This earth, which forms the only base of Tabular Spar, can be readily found by melting the powdered mineral with an excess of bisulphate of potash, and treating the fused mass with sufficient water. The sulphate of lime formed, and the sulphate of potash, dissolve, while the silica remains behind, sometimes with a portion of the gypsum. The earth is precipitated after filtration by oxalate of ammonia; the oxalate of lime is then collected on a filter, and, after being washed, treated with soda on charcoal, or with microcosmic salt upon the platinum wire. Should the mineral contain traces of alumina, this earth can be precipitated by caustic ammonia, before the lime is separated by the oxalate, and afterwards tested with a solution of cobalt (provided there is no peroxide of iron present to prevent the pale-blue reaction,) upon charcoal. To obtain the silica quite free from sulphate of lime, the siliceous residue on the filter must be treated with a *very weak* hydrochloric acid solution, and filtered. This second filtrate must not be added to the first, because if it is not properly diluted, a precipitate of sulphate of lime may occur on the addition of ammonia. Silicates (including furnaced ores, scoriæ, &c.) with several bases, are sometimes not at all decomposed by bisulphate of potash. This, however, is effected, if the same way is employed as given under Barytes for furnaced ores and slags.\*

If the solution obtained after melting the silicate with soda and borax, &c., (given under Barytes,) contains protoxide of iron, it must be converted into the peroxide with a few drops of nitric acid; then add sal-ammoniac, and precipitate the alumina and peroxide of iron by a slight excess of ammonia. The method of separating these two constituents will be given under Alumina, when the siliceous compounds are noticed. The lime which is in solution, combined probably

\* [Fresenius recommends for the decomposition of silicates, a mixture of carbonate of soda and potash.—See *English edition*, by Bullock, page 66.]  
—*Trans.*



with portions of magnesia, protoxide of manganese, and oxide of cobalt, is examined as follows. Hydrosulphate of ammonia (sulphuret of ammonia) is added to the solution, until all the manganese and cobalt is precipitated. The liquid is then filtered, and the residue washed with water, containing some of the precipitate. After affusing for some time, the filter containing the sulphurets is burnt, the residue pulverized, and examined with microcosmic salt on platinum wire for cobalt, and with soda (to which a small quantity of saltpetre has been added) on the platinum foil, in the oxidating flame, for manganese.

To the filtrate is added a few drops of hydrochloric acid, and the whole is treated over the lamp, until no sulphuretted hydrogen escapes. The menstruum is then filtered from the sulphur, the solution saturated with ammonia, the lime precipitated as an oxalate, and separated by filtration. The magnesia is precipitated by microcosmic salt as ammonia-phosphate, but before adding the microcosmic salt, it must be ascertained whether all the lime has been abstracted. When no manganese or cobalt is present, there will be two filtrations only, to separate the lime from the magnesia. If the silicate contains traces of protoxide of manganese only, they must be examined for in the precipitated magnesia with soda and saltpetre, as will be given under Manganese.

The protoxide of manganese is converted into a higher oxide by the nitre which combines with the alkali, forming a clear limpid glass, which is driven from one part of the platinum foil to another, while the magnesia remains in one spot upon the support, and is readily seen.

The examination of Silicates, which, besides lime, may contain yttria and protoxide of cerium, is given under Yttria.\*

\* [When solutions of lime are so very dilute, that sulphuric acid causes no precipitate, on the addition of alcohol, a deposit of sulphate immediately appears.

Döbereiner, about five years ago, gave an excellent method for the separation of lime and magnesia, which is nearly as follows:—Evaporate the hydrochloric acid solution of the two earths to dryness, in a large porcelain crucible, and then ignite to expel the excess of acid. Add chlorate of



§ 8. *MAGNESIA*—[Mg O].—*Presence in the Mineral Kingdom.*

This earth occurs as follows :—

(a) In *Aluminate*, as also in *Aluminate of Magnesia (Spinel)* [Mg O, Al<sup>2</sup> O<sup>3</sup>]; and as an Aluminate of the Protoxide of Iron and Magnesia, in *Zeylanite (Pleonaste)* [ $\begin{smallmatrix} \text{Mg} & \text{O} \\ \text{Fe} & \text{O} \end{smallmatrix} \} \text{Al}^2 \text{O}^3$ ];

(b) In *Brucite (Condrodite, Machureite)* [Mg Fl + 3 (3 Mg O, Si O<sup>3</sup>)];

(c) With Carbonic acid, in *Magnesite* [ $\begin{smallmatrix} \text{Mg} & \text{O} \\ \text{Fe} & \text{O} \\ \text{Mn} & \text{O} \end{smallmatrix} \} \text{CO}^2$ ]; and with Carbonic acid and Carbonate of Lime, in many *Carboniferous Spars*, as Dimeric in *Dolomite*, Isomeric in *Tarandite*, Americ in *Brown Spar*, &c.;

(d) With Sulphuric acid and water, in *Sulphate of Magnesia* [Mg O, S O<sup>3</sup> + H O + 6 aq]; and with Sulphuric acid, Sulphate of Potash, and Sulphate of Lime, in *Polyhallite*.

(e) With Boracic, in *Boracite* [Mg O, 2 B O<sup>3</sup> + 2 (Mg O, B O<sup>3</sup>)];

(f) In Silicates :—

(<sup>1</sup>) In which Magnesia forms the principal base; *e. g.*, in *Speckstein* [6 Mg O, 5 Si O<sup>3</sup> + 4 aq]; *Meerschaum* [Mg O, Si O<sup>3</sup> + aq]; *Picrosmine (Parallolite)* [3 Mg O, 2 Si O<sup>3</sup>]; *Serpentine* (hydrate of magnesia with subsilicate of magnesia) [3 Mg O, 6 H O + 2 (3 Mg O + 2 Si O<sup>3</sup>)]; *Olivine* [ $\begin{smallmatrix} 3 & \text{Mg} & \text{O} \\ 3 & \text{Fe} & \text{O} \end{smallmatrix} \} \text{Si O}^3$ ,] &c.;

(<sup>2</sup>) In Silicates of Lime and Magnesia, with Silicates of Alumina, without water; *e. g.*, *Anorthite* [3 Mg, Si O<sup>3</sup> + 2 (3 Ca O, Si O<sup>3</sup>) + 8 Al<sup>2</sup> O<sup>3</sup>, Si O<sup>3</sup>] or [3 Mg O, Si O<sup>3</sup> + 2 Al<sup>2</sup> O<sup>3</sup>, Si O<sup>3</sup> + 2 (3 Ca O, Si O<sup>3</sup> + 3 Al<sup>2</sup> O<sup>3</sup>, Si O<sup>3</sup>)]—Berzelius, and according to Abich [3 R O, 2 Si O<sup>3</sup> + 2 (R<sup>2</sup> O<sup>3</sup>, 2 Si O<sup>3</sup>) + 6 (3 Ca O, Si O<sup>3</sup>) + 3 (Al<sup>2</sup> O<sup>3</sup>, Si O<sup>3</sup>)];

potash in small crystals, until the eliminated chlorine gas is perceived. When the dry mass is affused with water, chlorides of calcium and potassium dissolve, and a residue of pure hydrated magnesia remains. The liquid can now be filtered, to separate the magnesia, and oxalate of ammonia added, to precipitate the lime.]—*Trans.*



(<sup>3</sup>) In Silicates of Potash or Soda, with Silicates of Magnesia, Alumina, and Protoxide of Iron, with and without water, in which the alkalis are often replaced by Magnesia and Protoxide of Iron; *e. g.*, in *Prismatic Magnesian Mica*, to which *Potstone*, *Green Earth*, *Chlorite*, and *Talc* belong;

(<sup>4</sup>) In Silicates of Lime and Magnesia, in which the alkaline earths are more or less replaced by Protoxides of Iron and Manganese, and the Silicic acid sometimes by Alumina; to these belong *Amphibole* and the *Magnesio-Pyroxenes*;

(<sup>5</sup>) In Silicates of Lime and Magnesia with Alumina, in which the alkaline earths are sometimes replaced by Protoxide of Iron, and the Alumina by the Peroxide; *e. g.*, in *Magnesio-Idiocrase*; in the *Black Granate of Arendal*; in *Soapstone* [ $3 \text{ Mg O}, 2 \text{ Si O}^3 + \text{Al}^2 \text{ O}^3, 2 \text{ Si O}^3 + 6 \text{ aq}$ ]; in *Blue* and *Red Dichroite*, (*Iolite*, *Cordierite*)  $\left[ \begin{smallmatrix} 3 \text{ Mg O} \\ 3 \text{ Fe O} \\ 3 \text{ Mn O} \end{smallmatrix} \right\} \text{ Si O}^3 + \text{Al}^2 \text{ O}^3, 2 \text{ Si O}^3$ ] and [ $3 \text{ Mg O}, 2 \text{ Si O}^3 + \left\{ \begin{smallmatrix} 3 \text{ Al}^2 \text{ O}^3 \\ 3 \text{ Fe}_2 \text{ O}_3 \end{smallmatrix} \right\} \text{ Si O}^3$ ];

(<sup>6</sup>) In Silicates with Phosphates; *e. g.*, in *Sordawalite*, seemingly a mixture of [ $2 \text{ Mg O}, \text{P O}^5$ ] with [ $3 \text{ Mg O}, 2 \text{ Si O}^3 + \text{Al}^2 \text{ O}^3, 2 \text{ Si O}^3 + 2 (3 \text{ Fe O}, 2 \text{ Si O}^3 + \text{Al}^2 \text{ O}^3, 2 \text{ Si O}^3)$ ];

(<sup>7</sup>) In Silicates with metallic Fluorides; *e. g.*, in *Chondrodite* [ $3 (\text{Mg Fl}) + 4 (3 \text{ Mg O}, \text{Si O}^3)$ ].

If some of the above mentioned minerals occur as well in the gangues as in the ores, the ores prepared in the dry way, and also the recrements and scoriæ obtained therefrom, generally contain a small quantity of Magnesia, which can readily be determined.

#### *Examination for Magnesia.*

(*a*) Aluminate, Spinel, and Zeylanite, are readily recognized before the blowpipe, as they give characteristic glasses, owing to the metallic oxides they contain, but magnesia *per se* is not detectible with certainty. To arrive at this earth, the finely powdered mineral must be heated with two parts of soda and three parts of borax, upon charcoal, the fused mass dissolved in hydrochloric acid, the solution evaporated to dryness; again dissolved in water, and the liquid filtered, to



separate the silica. Nitric acid must then be added to the filtrate, to convert the protoxide of iron into peroxide, and two spoonfuls of sal-ammoniac introduced; the iron, alumina, and any oxide of chromium, must then be precipitated by ammonia, and separated from the supernatant liquor. The filtrate must now be boiled, and the magnesia precipitated by microcosmic salt as ammonio-phosphate. This precipitate must be collected on a filter,edulcorated with water, and then treated on charcoal. It fuses to a white crystalline translucent pearl, and when treated with a few drops of cobalt solution, assumes a violet colour, which appears reddish in the flame of the lamp. When the Spinel dissolves in borax, with effervescence, it is a sign that there is some carbonate of lime between the interstices. When this occurs, the lime is to be precipitated by oxalate of ammonia, before the addition of the microcosmic salt. The aluminous precipitate can be examined with borax or microcosmic salt upon the platinum wire, for iron and chromium, or else treated with potash, which dissolves the alumina, and leaves the peroxide of iron as a reddish brown, and the oxide of chromium as a dirty green deposit.\*

If the mineral contained iron and chromium, and the residue only appears ferruginous, the mass may be smelted upon the platinum wire with saltpetre, and the fused mass treated with water. The chromate of potash (formed), and the undecomposed nitrate, dissolve, while the iron remains behind. The chromic acid can be detected in the solution, according to the method given under Chromium.

\* [When analyzing in the Giessen Laboratory, a compound containing alumina, peroxide of iron, and oxide of chromium, I proceeded as follows:—To a cold hydrochloric acid solution of the compounds, I added a solution of caustic potash, which precipitated the peroxide of iron as a reddish brown powder, which was filtered off. I boiled the solution, which contained an excess of the caustic alkali; the oxide of chromium precipitated as a green deposit, and was separated by filtration; to the filtrate I added chloride of ammonium, to precipitate the alumina. This is an excellent method, especially for qualitative analyses. The alumina is soluble in potash, but the oxide of chromium is not precipitated by this alkali in a cold state.]—*Trans.*



(*b*) Brucite, when moistened, after being treated on charcoal, re-acts alkaline with reddened litmus paper. It comport itself towards fluxes similarly to magnesia.

(*c*) Carbonate of Magnesia :—

(<sup>1</sup>) Magnesite (when non-contaminated) behaves like Brucite :

(<sup>2</sup>) Carbonate of Magnesia with Carbonate of Lime, as Dolomite, Brown Spar, &c.

These compounds behave like carbonate of lime before the blowpipe, so that the magnesia cannot be distinguished from the lime. It must, therefore, be obtained (after separating the iron and lime, as previously given) as ammonio-phosphate, and then treated on charcoal *per se*, and also with equal parts of soda and nitre upon the platinum wire. It ought to melt to a white crystalline pearl, easily soluble in microcosmic salt, provided no trace of manganese is present, producing a colourless glass, remaining so when brought in contact with a crystal of saltpetre. The smallest portion of manganese, if present, will be infallibly detected by treating the magnesian precipitate with soda and nitre on platinum foil.

If an appreciable quantity of manganese is contained in the specimen, it is better to precipitate it by hydrosulphate of ammonia; then filter, and free the solution from sulphuretted hydrogen, as given in the Lime examination.

(*d*) Sulphate of Magnesia (Epsomite) is immediately recognized on treating with a solution of cobalt.

(*e*) Boracite fuses with intumescence on charcoal; it is difficult to obtain a clear pearl, as it becomes drusy upon cooling, (that is, the whole surface of the charcoal presents crystalline needles). The glass while hot appears slightly yellowish, from the presence of peroxide of iron, but on refrigeration it is opaline. It readily dissolves in borax, forming a transparent ferruginous coloured glass.

Boracite also dissolves with facility in microcosmic salt to a clear glass, here and there turbidly streaked, and if an excess is present, the bead will be opaque when cold.

When it is smelted with soda sufficient to produce a clear



glass, the assay on cooling presents crystals and large facets. With more of the alkali, the glass is similar to a magnesian bead not sufficiently saturated. Heated in a powdered state upon charcoal, with a few drops of a cobalt solution, the whole concretes to a blue mass.

When melted with bisulphate of potash, and the fused assay dissolved in water, the magnesia can be precipitated and examined as before stated.

Should the Boracite contain borate of lime also, it is readily discovered by adding to the aqueous sulphate solution a little sal-ammoniac, and then oxalate of ammonia, before precipitating the magnesia with microcosmic salt.

The Boracic Acid is determined according to the process given under this head.

(*f*) In Silicates in which magnesia forms the only base, *e. g.* Speckstein, Meerschaum, Picrosmine, and Serpentine, the magnesia is ascertained the most readily by smelting the mineral with bisulphate of potash, and treating the vitreous residue with water; by this means the insoluble silicic acid is separated, and the filtrate containing sulphate of magnesia, can be heated with microcosmic salt, and examined as before remarked. If the mineral contains lime, as Olivine, it must be separated previously to precipitating the magnesia. Silicates containing several bases, to which the majority of the prepared ores and slags belong, cannot have the magnesia determined with certainty before the blowpipe, either alone or with bisulphate of potash; therefore, they must be examined in the same manner as given under Barytes and Lime.\*

#### § 9. ALUMINA—[ $\text{Al}^2\text{O}^3$ ].—*Presence in the Mineral Kingdom.*

Alumina is very abundant, being found:—

\* [The precipitation of the Basic Ammonio-phosphate of Magnesia, is greatly facilitated when the menstruum is boiled. When there is a *very small* quantity of magnesia present, it is better to allow the liquid, after the addition of the precipitant, to repose for some hours.

The separation of magnesia from lime, iron, &c., are treated of in Parnell's excellent work, "Elements of Chemical Analysis."—*Trans.*



(a) As pure Alumina, with slight traces of Silica and Peroxide of Iron, in *Corundum*, *Sapphire*, *Ruby* and *Diamant Spar*;

(b) As Hydrate of Alumina, in *Diaspore* [ $\text{Al}^2 \text{O}^3 + \text{aq}$ ]; and *Gibbsite* [ $\text{Al}^2 \text{O}^3 + 3 \text{aq}$ ];

(c) With Magnesia, as Magnesio-Alumina, in *Spinel*; with Protoxide of Iron, as Ferruginous Aluminate of Magnesia, in *Zeylanite*; with Oxide of Zinc, &c., Zincous Alumina, in *Automolite* (*Gahnite*) [ $\left\{ \begin{smallmatrix} \text{Zn} & \text{O} \\ \text{Mg} & \text{O} \\ \text{Fe} & \text{O} \end{smallmatrix} \right\} \text{Al}^2 \text{O}^3$ ]; and with Oxide of Lead, as Aluminate of Lead, in *Plombgomme* [ $\text{Pb O}, \text{Al}^2 \text{O}^3 + 6 \text{aq}$ ];

(d) With Fluorine and Fluoride of Sodium, in *Cryolite* [ $3 (\text{Na Fl}) + \text{Al}^2 \text{O}^3, 3 \text{Fl}$ ];

(e) With Sulphuric acid and water, in *Aluminite* [ $\text{Al}^2 \text{O}^3, \text{S O}^3 + 9 \text{aq}$ ]; with Sulphuric acid, Potash, Peroxide of Iron and Water, in *Potash-Alum*; with the same constituents without Potash, but with Ammonia, in *Ammonia-Alum*; and with Sulphuric acid, Potash, Silicic acid, Peroxide of Iron, and Water, in *Alum-stone*.

Formulæ for the two above-mentioned Alums:—

*Potash-Alum* [ $\text{K O}, \text{S O}^3 + \text{Fe}^2 \text{O}^3, 3 \text{S O}^3 + 24 \text{aq}$ ];

*Ammonia-Alum* [ $\text{N H}^4 \text{O}, \text{S O}^3 + \text{Fe}^2 \text{O}^3, 3 \text{S O}^3 + 24 \text{aq}$ ];

(f) With Phosphoric acid and Lithia, in *Amblygonite*; with Phosphoric Acid, Water, a little Fluorine, Lime, Protoxides of Iron and Manganese, in *Wavellite* [ $2 \text{Al}, 3 \text{Fl} + 3 (4 \text{Al}^2 \text{O}^3, 3 \text{P O}^5) + 18 \text{aq}$ ]; with Phosphoric acid, Magnesia, Silica, Protoxide of Iron, and Water, in *Lazulite* [ $3 (5 \text{Mg O}, 2 \text{P O}^5) + 4 (5 \text{Al}^2 \text{O}^3, 3 \text{P O}^5) + 4 \text{Fe O}, \text{P O}^5 + 15 \text{aq}$ ] &c. in varying proportions;

(g) In Silicates, *e. g.*:—

(<sup>1</sup>) Where Alumina is the only base, as in *Cyanite* [ $2 \text{Al}^2 \text{O}^3, \text{Si O}^3 + 3 (\text{Al}^2 \text{O}^3, \text{Si O}^3)$ ];

(<sup>2</sup>) In Silicates of Potash and Lime; *e. g.*, in *Zoisite* (*Epidote*);



(<sup>3</sup>) In Silicates of an alkali, or of an alkaline earth, and Silicates of Alumina, combined with water of crystallization.

To these belong *Chabasite*  $\left[ \begin{smallmatrix} 3 \text{ Ca O} \\ 3 \text{ Na O} \\ 3 \text{ K O} \end{smallmatrix} \right] \text{SiO}^3 + 3 (\text{Al}^2 \text{O}^3, 2 \text{SiO}^3) + 15 \text{aq}$ ]; *Mesolite*  $[\text{NaO}, \text{SiO}^3 + \text{Al}^2 \text{O}^3, \text{SiO}^3 + 2 \text{aq} + 2 (\text{CaO}, \text{SiO}^3 + \text{Al}^2 \text{O}^3, \text{SiO}^3 + 6 \text{aq})]$ ,—*Mesolite* may be considered as a mixture of *Mesotype* and *Scolezite*; *Mesotype*  $[\text{CaO}, \text{SiO}^3 + \text{Al}^2 \text{O}^3, \text{SiO}^3 + 3 \text{aq}]$ ; *Analcime*  $[3 \text{NaO}, 2 \text{SiO}^3 + 3 (\text{Al}^2 \text{O}^3, 2 \text{SiO}^3) + 6 \text{aq}]$ ; *Stilbite*, *Lamonite*, *Harmotome*, *Prehnite*, &c.;

(<sup>4</sup>) In Silicates of the Alkalies or Alkaline Earths, with Silicates of Alumina, without water; *e. g.*, *Felspar*, *Albite*, *Petalite*, *Spodumene*, *Leucite*, *Labradorite*, *Scapolite*; the formula of the last is  $\left[ \begin{smallmatrix} 3 \text{ Ca O} \\ 3 \text{ Na O} \end{smallmatrix} \right] 2 \text{SiO}^3 + 2 (\text{Al}^2 \text{O}^3, \text{SiO}^3)$ ]; further, in *Elaolite*  $\left[ \begin{smallmatrix} 3 \text{ K O} \\ 3 \text{ Na O} \end{smallmatrix} \right] \text{SiO}^3 + 3 (\text{Al}^2 \text{O}^3, \text{SiO}^3)$ ]; *Sodalite*, *Anorthite*, &c.;

(<sup>5</sup>) In Silicates of Potash or Lithia, with Silicates of Magnesia, Alumina, Protoxide of Iron, and Manganese, without water; in *Mica*, which sometimes contains Fluorine.

(<sup>6</sup>) In Silicates of Potash or Soda, with Silicates of Magnesia, Alumina, and Protoxide of Iron, with and without water, in which the alkalies are more or less replaced by magnesia or protoxide of iron; *e. g.*, in *Potstone*, *Green-earth*, *Chlorite*, and *Talc*;

(<sup>7</sup>) In Silicates of Lime and Magnesia with Alumina, in which the alkaline earths are often replaced by the protoxides of iron and manganese; *e. g.*, in *Pistacit*, *Idiocrase*, in many varieties of *Granates*, in *Gehlenite*, *Soapstone*, *Dichroite*, and *Karpholite*; the last sometimes contains traces of Fluorine, its general formula is  $\left[ \begin{smallmatrix} 3 \text{ Mn O} \\ 3 \text{ Fe O} \end{smallmatrix} \right] \text{SiO}^3 + 3 (\text{Al}^2 \text{O}^3, \text{SiO}^3) + 6 \text{aq}$ ];

(<sup>8</sup>) In Silicates of Alumina and Metallic Oxides; *e. g.*, in *Staurolite*, (from St. Gothard)  $\left[ \begin{smallmatrix} 3 \text{ Al O} \\ 3 \text{ Fe}^2 \text{ O} \end{smallmatrix} \right] \left\{ \begin{smallmatrix} 2 \text{ Si O}^3 \\ 2 \text{ Al}^2 \text{ O}^3 \end{smallmatrix} \right\}$ ]; and *Allophane*, from Schneeberg, which contains Oxide of Copper.



(<sup>9</sup>) In Silicates containing Glucina; *e. g.*, *Emerald* and *Beryl*, the formula for both of which is  $\left[ \begin{smallmatrix} 3 \text{ G}^2 \text{ O}^3 \\ 3 \text{ Al}^2 \text{ O}^3 \end{smallmatrix} \right] 8 \text{ SiO}^3$ ;\* further in *Euclase*  $\left[ \begin{smallmatrix} \text{Al}^2 \text{ O}^3 \\ \text{G}^2 \text{ O}^3 \end{smallmatrix} \right] \text{ SiO}^3$ ; and in *Cymophane* or *Chrysoberyl*  $[4 \text{ Al}^2 \text{ O}^3, \text{ SiO}^3 + 2(\text{G}^2 \text{ O}^3, 4 \text{ Al}^2 \text{ O}^3)]$ ;

(<sup>10</sup>) In Silicates which contain *Yttria* and Oxide of Cerium; *e. g.*, *Cerine*, *Allanite*, *Orthite*, and *Pyrorthite*;

(<sup>11</sup>) In Silicates containing Fluorides; *e. g.*, *Topaz*  $[\text{Al}^2 \text{ O}^3 + 2(2 \text{ Al}, 3 \text{ Fl}) + 6(\text{Al}^2 \text{ O}^3, \text{ SiO}^3)]$ ;

(<sup>12</sup>) In Polybasic Silicates, which contain small quantities of one or more Borates, as the various *Tourmalines*  $[\text{KO}, \text{LO}, \text{NaO}, \text{CaO}, \text{MgO}, \text{FeO}, \text{MnO}, \text{Al}^2 \text{ O}^3, \text{ SiO}^3, \text{ BO}^3]$ ; and in *Axinite*;†

(<sup>13</sup>) In Silicates of Soda, Lime, and Magnesia, combined with Sulphates; *e. g.*, in *Lazulite*, *Nosin*, &c.

(*h*) In various kinds of rocks; *e. g.*, *Fullers' Earth*, *Common Clay*, *Loam*, *Marl*, *Porphyry*, *Granate*, *Mica Slate*, *Clay Slate*, *Alum Slate*, *Sandstone*, *Gneiss*, &c.‡

As metallic gangues are generally classed under one or the other of the above-mentioned rocks, and as, in obtaining the ores, the adhering stony matrix cannot be completely separated from the former, therefore it is that alumina generally forms a considerable constituent of many ores prepared in the dry way, and consequently also an ingredient of the slags obtained in the smelting of such ores.

#### *Examination for Alumina.*

(*a*) *Corundum*, *Sapphire*, *Ruby*, *Diamant Spar*, comport themselves before the blowpipe, according to *Berzelius*, as follows:—

\* [When coloured green by Oxide of Chromium, it is the true *Emerald*, but when perfectly colourless and transparent, it is the *aqua marina*, which is a valuable gem.]—*Trans.*

† [Mr. W. K. O'Sullivan, who has examined many of the *Tourmalines*, has always found in them more or less *Manganese* and *Lime*.]—*Trans.*

‡ [Mr. W. K. O'Sullivan, in analyzing very lately, *Clay Slates*, &c., in the *Giessen Laboratory*, found in them, besides the usual given constituents, traces of *Sulphur*, *Phosphoric Acid*, *Chlorine*, and *Fluorine*.]—*Trans.*



Alone, they remain perfectly unchanged, as well in a powdered state as in fragments.

With borax they fuse perfectly (though with difficulty), to transparent colourless glasses, which do not become streaked with dull laminæ.

With microcosmic salt they fuse to a clear glass only, when added to the flux in a powdered state.

They are not attacked by soda.

When moistened with a solution of cobalt, and held in the state of a fine powder for a long time in the oxidating flame, they produce a beautiful blue colour.

By these reactions before the blowpipe, alumina may be immediately recognized.

(b) Hydrate of Alumina:—

(<sup>1</sup>) Diaspore, when heated in a matrass, decrepitates violently, and splits into small white brilliant scales, without giving out much water. These scales behave themselves with fluxes like pure alumina. Exposed to heat in the oxidating flame, with a few drops of a cobalt solution, this mineral affords a beautiful blue hue.

(<sup>2</sup>) Gibbsite, when heated in a flask, yields much water. It is infusible on charcoal, and behaves with fluxes like pure alumina, affording the special appearance with a solution of cobalt.

(c) Aluminate:—

(<sup>1</sup>) Spinel and Zeylanite. The method for the examination of these minerals for alumina, has been already described under Magnesia. The precipitate obtained by means of ammonia, after being well affused with water, should be treated with a solution of potash, and heated in the same manner as in the examination of silicates for alumina (which will be hereafter given), the solution diluted with water, filtered from the remaining oxides, decomposed with an adequate quantity of chloride of ammonium, and boiled, by which the alumina is thrown down. This precipitate can be tested with a solution of cobalt, after being purified with water.

(<sup>2</sup>) Automolite (Gahnite). This mineral, when heated on charcoal *per se*, is unalterable. With borax and microcosmic



salt (even when in the state of an impalpable powder) it is with difficulty fused. The resulting beads present the ferruginous tinge. It is not fusible with soda; it scintillates and affords a dark slag; when this is reduced to a very fine powder, mixed with soda, and placed on charcoal in a good reducing flame, the charcoal becomes coated with a ring of oxide in the commencement of the operation. It gives no reaction for manganese on a platinum foil.

With borax and soda together, it fuses to a clear glass, which becomes opaque and dark green on cooling.

I found a perfectly pure fragment of this mineral, from Sweden, to consist of alumina, oxide of zinc, magnesia, protoxide of iron, and silicic acid.

The analysis was performed in the following manner:—About 75 milligrammes of the fragment, very finely powdered, were melted into a bead, with five times as much of a mixture consisting of two parts of soda and three of borax, on charcoal in the reducing flame. The bead was kept in the reducing flame, until all the oxide of zinc was reduced, and the metal volatilized. The charcoal thus became coated with a sublimate of oxide of zinc, which when moistened with a solution of cobalt, and ignited in the oxidating flame, afforded on cooling a green colour. The fused bead was removed from the charcoal, placed on another piece of charcoal, and kept in a state of fusion in the reducing flame for some time, in order to be certain that all the oxide of zinc was reduced and volatilized. But as no sublimate of oxide of zinc formed, the melted bead was thrown on the anvil, by inverting the piece of charcoal, in order to obtain it free from carbonaceous particles. On cooling, it afforded a copperas green colour, and remained transparent. It was then pulverized, dissolved in hydrochloric acid, the solution evaporated to dryness, and the dry chloride dissolved in water. The aqueous solution was filtered, by which a small trace of silicic acid remained behind on the filter, which gave with soda the reactions of pure silicic acid. The solution was then heated with a few drops of nitric acid, in order to convert the protoxide of iron into the peroxide, and a little sal-ammoniac



dissolved in it to prevent the precipitation of magnesia by ammonia. Ammonia was then dropped into the solution until it was over-saturated; by which a flocculent precipitate of a yellowish white colour was thrown down, which, on being filtered, well washed, and subsequently treated with potash (as will be mentioned in the examination of silicates for alumina), proved to be oxide of iron and alumina; to the filtered liquid oxalate of ammonia was added, which produced no milkiness from the precipitation of oxalate of lime. But an addition of phosphate of soda and ammonia produced a precipitate, which, on being filtered, proved before the blow-pipe to be basic phosphate of ammonia and magnesia.

(<sup>3</sup>) *Plombgomme*. According to Berzelius, this mineral gives the following reactions before the blowpipe, from which its constituents can be readily determined. Heated alone in a flask, it gives off aqueous vapours, and sometimes decrepitates with violence.

On charcoal it loses its transparency, becomes white, swells up like a zeolite, and semifuses under a strong temperature, without, however, being rendered fluid.

With borax and microcosmic salt, it fuses into a colourless transparent glass. The microcosmic bead becomes, with a certain addition of the assay, opaque on cooling.

It does not fuse with soda; small lead globules, however, appears on all sides when the experiment is made on charcoal. It gives a beautiful pure blue colour with a solution of cobalt.

(*d*) Fluoride of Sodium with Fluoride of Aluminum; as Cryolite, from Greenland:—

Heated alone in the matrass, it affords some water, and decrepitates without losing its transparency.

Held with the platinum forceps in the apex of the blue flame, it colours the exterior flame of a strong yellow, from soda.

Blown upon in an open tube (the flame being conducted into the tube), it strongly attacks the glass, and the moisture which collects in the tube, reacts like hydrofluoric acid. It readily fuses on charcoal to a clear bead, which becomes



turbid on cooling. After long exposure to the flame, the bead spreads, the fluoride of sodium is absorbed by the support, and an aluminous crust remains on the surface.

It readily fuses, even in large quantities, to a clear glass, with borax and microcosmic salt, which on cooling, becomes milk-white. According to Berzelius, the bead with microcosmic salt treated in the reducing flame, should sometimes become somewhat red on cooling, from its containing a trace of copper.

It fuses with soda to a clear glass, which expands and becomes milk-white on cooling.

A small quantity of the powdered mineral, moistened with a solution of cobalt, fuses in the oxidating flame, on charcoal, to a blue bead.

(e) Combinations of Alumina with Sulphuric Acid; as Potash- and Ammonia-Alum, Aluminite, and Alum-stone:—

If the first two salts be heated in a glass flask, they tumefy and give off water; by stronger heat the Potash-Alum gives off sulphurous acid, and sulphate of ammonia sublimes from the Ammonia-Alum, which is for the most part dissolved in water; sulphurous acid is also given off in this case. The residual dry mass from both salts, behave with fluxes, and with solution of cobalt (with which last they give a bright blue colour), like alumina. Aluminite yields at first much water, and as it approaches ignition eliminates sulphurous acid, which may be known by its smell, and by its reaction with moistened litmus paper.

It behaves with fluxes, and with solution of cobalt, exactly as alumina.

Alum-stone at first yields water, and by stronger heat a sublimate of sulphate of ammonia, which is soluble in water. The crystallized specimens decrepitate, affording a fine powder.

On charcoal it contracts when exposed to a strong flame, but does not melt.

With microcosmic salt it is easily melted, leaving behind a skeleton of silicic acid.



(f) Combinations of Alumina with Phosphoric Acid :—

In these combinations, to which Wavellite, Lazulite, and Amblygonite belong, alumina is readily distinguished by the behaviour of these minerals to fluxes, and particularly to solution of cobalt, with which it produces a beautiful blue. It is required, however, to find the other constituents, namely, the lime in Wavellite, and the magnesia in Lazulite. These minerals, as they are not easily soluble in muriatic acid, must be first fused to a glass, with equal parts of borax and soda, on charcoal in the oxidating flame: this glass should then be pulverized, dissolved in hydrochloric acid, the protoxide of iron converted by means of nitric acid into peroxide; the solution, as it contains phosphoric acid, diluted with alcohol; and the lime precipitated by dilute sulphuric acid, the peroxide of iron and alumina by ammonia, and the magnesia by phosphate of soda and ammonia. Alumina sometimes falls with the peroxide of iron, as phosphate of alumina; it can in this case be separated from peroxide of iron by means of potash, and precipitated from the warm diluted solution by chloride of ammonium, as will be immediately mentioned in the case of silicates and varieties of rocks. The silicic acid in Lazulite is detected, by treating with hydrochloric acid the specimen melted with soda and borax; the phosphoric acid in both minerals, as well as the fluorine, and the protoxide of manganese in Wavellite, are detected by special experiments, which are given in different parts of this book, for these minerals; and the protoxide of iron in Wavellite and Lazulite may be detected by treating them with borax, and also in the precipitate obtained by ammonia.

(g) Silicates and Rocks :—

The alumina contained in silicates, and in the different kinds of rocks to which the picked ores and slags belong, cannot be well detected, except in a few of its combinations, by its behaviour to vitreous fluxes and solution of cobalt. In most cases it is necessary to employ the method which is given under Barytes and Lime.

The precipitate thus obtained by ammonia, which usually consists of alumina and peroxide of iron, and also very slight



traces of magnesia, or protoxide of manganese, when the substance under investigation contains much of these bodies, must be well washed on the filter, and heated with potash while still moist in a porcelain basin, until the whole of the alumina is dissolved out, and the oxide of iron appears in the solution in a finely-divided state, of a reddish brown colour, pure or mixed with slight traces of precipitated magnesia, or protoxide of manganese, which in a qualitative examination may be entirely overlooked. The alkaline solution of the alumina is then to be diluted with water, filtered from the oxide of iron, mixed with an excess of sal-ammoniac, and slightly boiled, in order to re-precipitate the alumina; or it may be supersaturated with hydrochloric acid, and the alumina precipitated by a solution of carbonate of ammonia. The precipitated alumina, after being filtered and well washed, can be tested with solution of cobalt before the blowpipe. If the substance contains no iron, the precipitate by ammonia appears perfectly white, and need not in this case be treated with potash, but tested immediately with solution of cobalt.

Silicates containing glucina, yttria, and oxide of cerium, in which it is intended to seek for these substances at the same time, should be treated according to the methods which will be given for the examination of Glucina, Yttria, and Oxide of Cerium.

§ 10. *GLUCINA*—[ $G^2O^3$ ].—*Presence in the Mineral Kingdom.*

Glucina is not frequently found in nature; it occurs only in siliceous combinations, which are the following:—

(a) Silicate of Alumina and Glucina, as the *Emerald*, the *Beryl*, (*Euclase*).

(b) Basic Silicate of Alumina, with Alumino-Glucina; as *Cymophane* and *Chrysoberyl*; and

(c) The Bi-silicates of Glucina and Peroxide of Iron, with Sesqui-silicate of Protoxides, and Oxy-sulphuret of Manganese; as *Helvine* [ $3(MnS, MnO) + 3MnO, 2SiO^3 + G^2O^3, 2SiO^3 + Fe^2O^3, 2SiO^3$ ]; from Schwarzenberg, in Saxony.



*Examination for Glucina.*

These glucinous minerals are so constituted, that the presence of glucina cannot be ascertained with certainty by their behaviour to vitreous fluxes, because both alumina and silica, and the metallic oxides present in them, completely hinder the proper reaction of glucina, which, even when free from them, is not very striking. The metallic oxides, such as oxide of iron, oxide of chromium, protoxide of manganese, and oxide of tin (which last forms a constituent of the emerald, found in granular fragments at Broddbo and Finbo), are easily detected, the first two by the colours which they produce with borax and microcosmic salt, the third by soda on platinum foil, and the last by reduction with soda.

The presence of glucina in metals, is best ascertained in the moist way, as for example by the following method:—

Part of the mineral very finely powdered, must be fused on charcoal, with a mixture of one and a half parts of soda and one of borax, in the reducing flame; pulverized, dissolved in muriatic acid, evaporated to dryness; the dry mass re-dissolved in water, the solution separated from the insoluble silica by filtration, and heated with a few drops of nitric acid, in order to convert the protoxide of iron into the peroxide, if it should happen to contain any. Ammonia is then to be added to the solution, until every thing is precipitated. The precipitate is to be well washed, and heated with a solution of potash. If it consists of alumina, glucina, peroxide of iron, oxide of chromium, and protoxide of manganese, the two earths will be dissolved out, while the metallic oxides remain undissolved behind, and may be easily recognized after being filtered, before the blowpipe. The alkaline solution of alumina and glucina, must be decomposed either by boiling it moderately with sal-ammoniac, or by hydrochloric acid and ammonia, as has been already mentioned in speaking of the examination of silicates for alumina; the precipitated earths are filtered from the liquid, and dissolved in a little hydrochloric acid and carbonate of ammonia, until a voluminous precipitate, which consists of alumina and glucina, is produced. As glucina is soluble in carbonate of ammonia,



an excess of this reagent should be added, by which the precipitate diminishes, and the glucina re-dissolves. The mass should then be filtered, and the filtrate boiled; during the ebullition the ammonia is expelled, and the glucina falls down in the state of a carbonate, which, after being separated by filtration, can be tested before the blowpipe. It should shew the reaction given in Table I. The alumina can also be tested before the blowpipe with a solution of cobalt.

The presence of sulphur in Helvine is detected, as well as in the solution, in hydrochloric acid, of the mineral melted with soda and borax, by its evolving sulphuretted hydrogen, which is readily distinguished by its smell, as also by a special examination for sulphur.

§ 11. YTTRIA—[YO].—*Presence in the Mineral Kingdom.*

This earth occurs but seldom, though it is found in many different states of combination, as for example:—

(a) As Di-phosphate of Yttria, in Yttria-Phosphate or Phosphate of Yttria  $[3YO, PO^5]$ ;

(b) As a combination of Fluoride of Calcium, with Fluoride of Yttrium, and Fluoride of Cerium, in variable proportions, in *Yttro-Cerite*  $[CaFl + YFl + CeFl]$ ;

(c) As Titanate of Yttria, with Titanate of Zirconia, the Protoxides of Cerium, Manganese, and Iron, Lime and other bases, in *Polymignite*, from Fredrikswärn.

(d) As Tri-basic Tantalate\* of Yttria and Lime, with a perceptible quantity of Tri-basic Tungstate of the Protoxide of Iron, in the *Black Yttro-Tantalite*, from Finbo, Ytterby, and Kararfvet; as Tri-basic Columbate of Yttria and Lime, mixed with a little Tungstate of the Protoxide of Iron, in the *Dark Yttro-Tantalite* from Ytterby; and as Tri-basic Columbate of Yttria and Oxide of Uranium, in *Yellow Yttro-Tantalite*, from Ytterby and Finbo,  $[3YO \{ \begin{smallmatrix} Ta & O^3 \\ U_2 & O^3 \end{smallmatrix} \}]$ ;

(e) In Siliceous combinations:—

(1) As Tri-basic Silicate of Yttria, with Sex-basic Silicate

\* [Tantalum and Columbium are synonymous; Columbium is used on the Continent, Tantalum in England.]—*Trans.*



of Cerium and Protoxide of Iron, in *Gadolinite*, from Ytterby, Finbo, and Broddbo,  $\left[ \begin{smallmatrix} 6 \text{ Fe O, Si O}^3 + 2 (3 \text{ Y O, Si O}^3) \\ 6 \text{ Ce O, Si O}^3 + 2 (3 \text{ Y O, Si O}^3) \end{smallmatrix} \right]$ ;

(2) In a Siliceous combination of Protoxide of Cerium, Alumina, Lime, Yttria, Protoxides of Iron, and Manganese, with chemically combined water, in *Orthite*; and,

(3) In a Siliceous combination of Protoxide of Cerium, Yttria, Protoxide of Manganese, Alumina, and Lime, which at the same time contains carbon and water, in the *Pyrrhotite*.

*Examination for Yttria.*

(a) The phosphate of yttria can be easily recognised by its behaviour before the blowpipe, from its giving a regulus of phosphuret of iron with boracic acid and iron, as will be subsequently mentioned under the head Phosphoric Acid, and by its being with difficulty dissolved by microcosmic salt.

But if it is required to detect with accuracy the presence of yttria itself, the assistance of the moist way must be had recourse to, and the following method pursued.

The mineral, in the state of a very fine powder, is first to be mixed with four times its weight of soda, and the mixture fused in a platinum spoon, in the flame of the spirit lamp, until it ceases to effervesce. The fused mass, together with the spoon, should then be placed in some water, in a small porcelain vessel, and heated to the boiling point over the flame of the spirit-lamp. The phosphate of soda formed, and the excess of carbonate of soda employed, are dissolved by the water, while the yttria, with some oxide of iron, which was contained in the mineral as basic phosphate of iron, remains behind undissolved. The residue separated from the fluid by filtration, is thenedulcorated. Phosphoric acid may be detected in a small portion of the fluid, by the method which will be given under this acid.

Theedulcorated residue of yttria and peroxide of iron, must be dissolved while still moist, in a little hydrochloric acid, the solution diluted with water, one or two small spoonfuls of pulverized tartaric acid added, and the vessel allowed to stand in the heat until the acid is completely dissolved.



According to Rose, tartaric acid possesses the property of preventing the precipitation by alkalies, in any solutions of metallic oxides and earths, and to these yttria and oxide of iron belong. The solution is then saturated with ammonia, and sulphuret of ammonium added till it ceases to throw down a black precipitate. The iron is thus separated as a sulphuret, while the yttria remains unchanged in the solution. If the supernatant liquid appears only yellow, when the precipitate has sunk completely to the bottom, it should be filtered; but if green, the whole should be first allowed to digest in a very gentle heat until it becomes yellow. The sulphuret of iron, after its separation by filtration, may be roasted on charcoal, and the resulting black or magnetic oxide, with borax. The filtered solution should be evaporated to dryness, and the dry mass ignited in a platinum spoon over the spirit-lamp, until all the charcoal of the tartaric acid is consumed, and the yttria appears on cooling perfectly white. The dried mass should be placed in the spoon by degrees, as, during the decomposition of the acid, a portion of the yttria may be lost, by being projected out of the recipient.

The yttria obtained in this manner, should behave itself before the blowpipe in the manner described in Table I.

Peroxide of iron may be separated in another way from yttria, by employing the method given by Demarçay, in the *Annales de Chimie et de Physique*, April, 1834, page 398. Rather more than the necessary quantity of carbonate of barytes should be gradually added to a cold solution of yttria and peroxide of iron, in muriatic acid, and the fluid stirred with a glass rod, so long as carbonic acid gas is evolved; the peroxide of iron is precipitated, while the yttria remains in the solution. The supernatant liquid is then separated from the precipitate by filtration, the barytes thrown down by dilute sulphuric acid, and the yttria by potash or ammonia.

(b) Ytthro-cerite is readily recognised before the blowpipe, but its yttria cannot be detected by this means alone.

The Ytthro-cerite from Finbo, when heated in a flask, yields some water, which has an empyreumatic odour. The dark



coloured variety loses its colour by this treatment, and becomes white.

It is infusible when heated on charcoal *per se*; with gypsum it fuses to a bead, which does not become clear, however, at any temperature.

It readily fuses with borax and microcosmic salt to a transparent yellow glass, which is colourless when cold, but at a certain point of saturation translucent.

With soda, unless too much be used, it fuses on charcoal to a clear glass, which upon cooling, and also when the blast is directed on it for a long time, becomes turbid; a larger addition of soda converts it into a difficultly fusible enamel, which remains on the charcoal while the excess of soda is absorbed.

The Yttro-cerite from Broddbo, decrepitates but slightly when heated in a flask. It is infusible on charcoal, but on heating it becomes first milk-white, then brick-red, but, however, always unequally coloured. It does not fuse with gypsum.

It behaves to borax and microcosmic salt in a nearly similar manner to oxide of cerium, as it contains a large quantity of fluoride of cerium mixed with it.

It is acted upon by soda, tumefies a little, but is not dissolved. The greater part of the soda is absorbed by the charcoal, leaving a grey scoria behind.

If it be required to separate the yttria from Yttro-cerite, it must be done in the moist way, according to the following method; the finely powdered mineral is to be dissolved in hydrochloric acid, heated to ebullition, the solution evaporated nearly to dryness, diluted with water, and filtered in case any undissolved particles are observed. From this diluted solution, alumina, yttria, and protoxide of cerium may be precipitated by an excess of ammonia; the whole should then be warmed, and the precipitate separated by filtration. Lime is precipitated from the filtrate by oxalate of ammonia. The precipitate obtained by ammonia, should be edulcorated with hot water, until the water which percolates through the filter is no longer rendered turbid by oxalate of ammonia, and di-



gested at a moderate heat while still moist with caustic potash, in order to separate the alumina from the yttria and protoxide of cerium. When the digestion is complete, the alkaline solution is to be diluted with water and filtered. The alumina may be separated from the filtered solution slightly heated, by the necessary quantity of sal-ammoniac, or by ammonia from the solution, rendered acid by muriatic acid. In both cases, the precipitate after filtration must be well edulcorated, and then tested with a cobalt solution. The undissolved yttria and protoxide of cerium is to be well washed with hot water, dissolved in hydrochloric acid, and the solution diluted with water. A crust of crystallized sulphate of potash must then be placed on the surface of the liquid, and the whole allowed to stand about twenty-four hours. During this time the solution will become saturated with sulphate of potash, with which the yttria and protoxide of cerium will combine, forming double salts; of which the one formed with yttria is soluble in a saturated solution of sulphate of potash, while that formed with protoxide of cerium, on the other hand, is insoluble, and falls to the bottom as a white amorphous powder. After the lapse of the above mentioned time, the remaining crust is removed, the precipitate separated by filtration, and edulcorated with a saturated solution of sulphate of potash. After the edulcoration, it is to be dissolved in boiling water, the protoxide of cerium precipitated from the still warm solution by potash, filtered, the precipitate edulcorated, and ignited in the platinum spoon. During the ignition it is converted into peroxide, and becomes of a cinnamon brown colour. The yttria which remains in solution, is also to be thrown down from the warm solution by potash, filtered, and ignited in the platinum spoon. It must possess a white colour after the ignition; if it becomes brown, it contains peroxide of iron, which can be separated after the earth has been dissolved in hydrochloric acid, by the method already given for phosphate of yttria. A separate examination is required for the detection of hydrofluoric acid, which will be subsequently given.

(c) Polymignite, from Fredrikswärn, behaves in the



following manner before the blowpipe, according to Berzelius.

Heated in a bulb tube, it yields no water; it is infusible on charcoal, and does not change its colour or brilliancy. Borax readily dissolves it to an iron-coloured glass, which by a larger addition acquires the property of becoming streaked with turbid lines, by which it generally appears of a brownish yellow colour. From a larger addition it becomes turbid *per se* on cooling; with tin the bead becomes of a red colour, passing into yellow.

It is difficultly dissolved by microcosmic salt, the glass assuming a reddish colour in the oxidating flame, which is not altered on the addition of tin. It is decomposed by soda, becoming greyish red, but does not fuse; it gives the same reaction as manganese on platinum foil.

In order to separate the yttria, the moist way must be had recourse to, as in the case of the first two minerals. The method of procedure is as follows; the finely powdered mineral is to be fused with from six to eight times its weight of bi-sulphate of potash, in a platinum spoon, and the fused mass digested with a sufficient quantity of water, until the whole is dissolved out of the spoon; the sulphates of the protoxides of iron, cerium, and manganese, of yttria, lime, and magnesia, are dissolved, while the titanick acid, in combination with zirconia, and a part of the protoxide of iron, remains behind; the whole must then be kept for some time at a moderate ebullition, and filtered. The protoxide of cerium may be separated from the solution by a piece of sulphate of potash, in the manner already described under Ytthro-cerite. To the solution, from which the double salt has been separated by filtration, a few drops of nitric acid are to be added, in order to convert the protoxide of iron into the peroxide; a little sal-ammoniac should also be added, to prevent the precipitation of protoxide of manganese and magnesia, if it should contain them, and the yttria and peroxide of iron thrown down with ammonia. Lime is separated from the filtered solution, by oxalate of ammonia, and magnesia and protoxide of manganese, by microcosmic salt. The precipitate obtained



by ammonia, must be dissolved in hydrochloric acid, and the peroxide of iron separated from the yttria, by the method already given for the decomposition of phosphate of yttria.

(d) Berzelius gives the following as the behaviour of Yttro-tantalite before the blowpipe. Heated *per se* in a bulb tube, it gives off water, and if previously black becomes yellow. Some specimens become mottled with dark spots, which remain unchanged in the heat; they become white on ignition, while the upper part of the glass is attacked. The water eliminated, colours brazil-wood paper at first yellow, and then bleaches it.

Borax dissolves it to an almost colourless glass, which by a larger addition, becomes turbid on cooling.

It is at first decomposed by microcosmic salt, the tantalic acid remaining undissolved as a white skeleton, which fuses, however, in a strong blast. The black variety of Yttro-tantalite from Ytterby, produces a glass which becomes of a light rose colour by a strong reducing flame, on cooling, derived from the presence of tungsten. The dark and yellow varieties from Ytterby, become on cooling, of a slight but beautiful green colour, from the presence of uranium. The Yttro-tantalite from Finbo and Kararfvet, gives a strong ferruginous tint, which becomes darker when the test for oxide of uranium is employed.

It is decomposed without solution by soda. It exhibits on platinum foil the presence of manganese. Traces of tin are obtained by reducing with soda and borax; the Finbo variety, however, contains so much iron, that the tin cannot be recognised.

The preparation of yttria from Yttro-tantalite is attended with much difficulty. The separation can only be performed in the moist way, and safest when more than 100 milligrammes of the mineral are employed, for which larger vessels are required than the blowpipe operator can conveniently carry about with him.

(e) Siliceous combinations:—

(1) Gadolinite, from Ytterby, Finbo, and Broddbo;—

Berzelius gives the following account of their behaviour



before the blowpipe. These Gadolinites are of two varieties, one of which,  $\alpha$ , is of such a vitreous nature, that it appears like black glass; the other,  $\beta$ , on the contrary, has a splintery fracture, and not so conchoidal; the former contains no glucina, but it is very probable that the latter contains some per cents.

Variety  $\alpha$ , *per se*, in a bulb tube, undergoes no change, and gives off no water; if it be nearly heated to the point of fusion it appears to glow for an instant as if it had caught fire; it tumefies a little, and if the fragment be large, fissures appear here and there in it, and the colour assumes a light greyish green; no volatile matter is, however, given off. The same phenomena take place on charcoal; it does not fuse, but in a strong heat it becomes black on the edges.

Variety  $\beta$ , *per se*, swells out into dendritic ramifications, and becomes white, at the same time giving off moisture. The above-mentioned luminous appearance is very rarely observed. In all other points both behave alike to fluxes.

It is readily dissolved by borax, forming an intense ferruginous-coloured glass, which becomes of a deep bottle-green in the reducing flame.

It is difficultly dissolved by microcosmic salt. The glass assumes a ferruginous colour, and the fragment employed becomes rounded on the edges, but it remains white and opaque, so that the silicic acid cannot be separated from the phosphoric acid; by this it is principally distinguished from the Kararfvet variety. Soda dissolves it to a reddish brown semi-fused scoria. The variety  $\beta$  melts into a bead with soda, when too large a quantity of the flux is not employed. None of them exhibit the slightest trace of manganese on platinum foil.

The moist way must be had recourse to, in order to separate yttria from Gadolinite. The method of procedure is as follows:—

The finely powdered mineral is to be fused to a bead, with an equal bulk of soda and borax, on charcoal, in the oxidating flame; the bead pulverized, the powder dissolved in hydrochloric acid, the solution evaporated to dryness, the dry mass



dissolved and filtered from the insoluble silicic acid. A few drops of nitric acid are then to be added to the solution, and the whole heated, in order to convert the protoxide of iron into the peroxide, and the yttria, protoxide of cerium, peroxide of iron, and glucina, when present, thrown down by ammonia. Lime may be precipitated from the supernatant liquid, when present, by oxalate of ammonia. The precipitate by ammonia, afteredulcoration with hot water, must be treated in the heat while still moist, with a solution of caustic potash, in order to separate any glucina present; the alkaline solution should then be diluted with water, and the glucina separated from it by sal-ammoniac, in the way already given under the head Glucina. The residue, afteredulcoration, is then to be dissolved while still moist, in a small quantity of muriatic acid, and the protoxide of cerium separated by sulphate of potash, by the method given for the decomposition of Yttrocerite. The peroxide of iron and the yttria are precipitated by a solution of potash; the precipitate, afteredulcoration, dissolved in hydrochloric acid, and both constituents separated by the method given for the analysis of Phosphate of Yttria.

(<sup>2</sup>) Orthite, and (<sup>3</sup>) Pyrorthite, contains only from three to four per cent. of yttria; it cannot be separated in a pure state from this mineral in the dry way, and only by the moist way, when a large quantity (as 100 milligrammes) is employed.

The behaviour of both these minerals before the blowpipe, is as follows:—

Orthite, from Finbo and Gottliebsgang, as well as that from Granit, near Stockholm, and Söderköping;—

*Per se* in a bulb tube, it yields water, and at a high temperature assumes a light colour. It tumefies and becomes yellowish brown on charcoal, and at length fuses under ebullition, to a black blebby glass.

It is easily dissolved by borax; the glass becomes blood-red in the reducing flame, when hot, and yellow on cooling. In the reducing flame it becomes of a ferruginous green.

It is decomposed, with the usual appearances, by microcosmic salt.



With soda it tumefies; with a very small quantity it fuses, but with a large quantity it intumesces to a greyish yellow recrement. On platinum foil it shows the presence of manganese.

Pyrorthite from Kararfvet;—

*Per se* in a bulb tube, it at first gives off a large quantity of water, which towards the end becomes yellow, and has an empyreumatic odour, the mineral becoming coal black. Moderately heated on charcoal, and then ignited at a single point, it begins to become luminous, and afterwards, of itself, continues to glow without flame or smoke. If a number of small fragments be laid together, or a small heap of coarse powder be taken, the combustion proceeds better; by a moderate heat it is also increased. When the combustion is ended, the assay is white or greyish white; with different pieces this is not always the same, and sometimes it even passes into red. The fragments are now so light, that they cannot be kept on the charcoal in the blast. Held in the forceps, they fuse with difficulty to a black, on the surface dull bead.

With borax it fuses readily to a glass, which behaves similarly to the borax glass of Orthite.

It is with difficulty dissolved by microcosmic salt. The porous fragment remains on the surface of the bead, so long as the mass is molten, but on cooling it is absorbed; but if the bead be re-heated, it comes to the surface as before.

It behaves itself to soda like Orthite.

§ 12. ZIRCONIA— $[\text{Zr}^2\text{O}^3]$ .—*Presence in the Mineral Kingdom.*

This earth is found in combination with Silicic and Titanic acids, namely:—

(a) As Silicate of Zirconia, in the *Zircon* and *Hyacinth*  $[\text{Zr}^2\text{O}^3, \text{SiO}^3]$ ;

(b) In a combination of Sesqui-basic Silicates of Soda and Lime, with Silicates of the Peroxide of Iron, Manganese, and Zirconia, also Chloride of Sodium, in *Eudyalite*  $[\text{Na Cl} + \{ \frac{3}{3} \text{CaO}, 2\text{SiO}^3 \} + \{ \text{Zr}^2\text{O}^3, \text{SiO}^3 \}];$



(c) In a combination of the Titanates of Zirconia, Yttria, Protoxides of Cerium, Manganese, and Iron, Lime and other bases, in *Polymignite*. \*

*Examination for Zirconia.*

Zirconia cannot be detected by the aid of the blowpipe alone, either in those minerals where it forms only a secondary constituent, or even in those where it forms the primary ingredient; but the minerals themselves may be immediately recognised by their behaviour before it.

(a) The Zircon and Hyacinth from Ceylon, Finbo, Fredrikswärn, and Expailly, behave themselves, according to Berzelius, in the following manner:—

*Per se*, the colourless transparent varieties remain unchanged. The clear red Hyacinth loses its colour, and becomes either perfectly limpid, or very slightly yellow. The opalestic brown from Fredrikswärn, loses its colour and becomes white, similar to fractured glass. The dark variety from Finbo, gives off a little moisture, becomes milk-white, and appears scopiform. None of them can be fused, either in the state of powder or of their splinters.

Zircon is fused with difficulty to a clear glass by borax, which, at a certain saturation, becomes streaked with turbid lines, and from a still greater quantity, becomes translucent on cooling.

Zircon remains intact with microcosmic salt; a fragment subjected to its action, retains the sharpness of its edges, and when Zircon powder is employed, it is so little changed that it cannot be determined whether it was attacked or not.

The glass remains perfectly colourless or milk-white when the powder is added to it, as well in the oxidating as in the reducing flame.

It is not fused with soda; the soda, however, attacks it slightly on the edges, but it afterwards sinks into the charcoal. Most specimens of Zircon shew the presence of manganese, when treated on platinum foil.

\* [The name is derived from *πολυς*, much, and *μυγνωω*, I mix; in allusion to its number of ingredients.]—*Trans.*



(*b*) Eudyalite, according to Berzelius, exhibits the following behaviour before the blowpipe:—

In a bulb tube it gives off moisture; it readily fuses on charcoal to a greyish glass, passing into green.

It is readily dissolved by borax, to a glass slightly coloured by iron, which does not become turbidly streaked.

It is easily decomposed by microcosmic salt; the siliceous skeleton swells up so much, that the bead generally loses its form. By this peculiarity it distinguishes itself from the granites, to which it is otherwise similar.

Soda dissolves it to a glass, which is not very fluid; with more soda it sinks into the charcoal. It shows the presence of manganese on platinum foil.

The comportment of Polymignite before the blowpipe, has been already given at page 126–7. The assistance of the moist way must be had recourse to, if it be desired to separate zirconia from the Zircon, Hyacinth, or Eudyalite, with the blowpipe; the method of procedure being as follows:—The mineral, in the state of a very fine powder, is first fused to a glass, for the Zircon, with a mixture of one and a half parts of soda, and three parts of borax, to one of the mineral; and for Eudyalite, two parts of soda, and one and a half parts of borax, on charcoal, in the oxidating flame; the glass pulverized, the powder treated with hydrochloric acid, and the solution evaporated to dryness, in order to be able to separate perfectly the silica. The evaporation should not be effected too rapidly, nor at too strong a heat, as in that case, a portion of the zirconia would remain undissolved with the silica. The dry mass thus obtained, is then to be treated with a sufficient quantity of water, and the silicic acid separated by filtration. If the mineral should happen to contain iron, as is the case with Eudyalite, the greater part of it will be found in the solution as protoxide, which should be converted into peroxide, by the addition of a few drops of nitric acid. When manganese is present, it will also be in the solution as protoxide, and should be rendered incapable of precipitation by ammonia, by the addition of a little sal-ammoniac.

Zirconia and peroxide of iron should then be precipitated



by ammonia, the whole heated to ebullition, in order that the precipitate, which is very voluminous, may collect, and thus be more easily filtered. If the mineral contains lime, it remains behind in the solution, and may be precipitated by oxalate of ammonia.

Nothing now remains to be done, but to separate the peroxide of iron from the zirconia, which can be performed in two ways:—

1. The precipitate is welledulcorated with boiling water, dried, ignited in a platinum spoon, and treated with hydrochloric acid. The peroxide is by this means almost perfectly dissolved, while the zirconia, which, after ignition is insoluble in this acid, remains nearly pure behind.

2. The precipitate, while still moist, is dissolved in hydrochloric acid, and the oxide of iron precipitated by sulphuret of ammonium, as in the method given at page 124, for the separation of oxide of iron and yttria. The liquid filtered from the sulphuret of iron, is to be evaporated to dryness, and the dry mass ignited in a platinum spoon, over the spirit lamp, until the whole of the charcoal of the tartaric acid is burned off, and the zirconia appears perfectly white.

A much more simple method may be employed for the detection of zirconia, in the Zircon or Hyacinth, as they contain no lime. The solution is first neutralized in a test-glass with potash, and a crystal of sulphate of potash laid in it, of such a size that it is sufficient to saturate perfectly the solution, and the whole warmed. As soon as a portion of the crystal has dissolved, a double compound of sulphate of zirconia and potash forms, which falls to the bottom of the glass as a flocculent basic salt, and when a sufficient quantity of the crystal has dissolved, the whole of the zirconia is precipitated in this state, and can be obtained by filtration; it should then be welledulcorated, and boiled with caustic potash, by which it is decomposed, leaving pure hydrate of zirconia behind.

Zirconia cannot be obtained with successful results, from even a quantity of 100 milligrammes of Polymignite, owing to the presence of titanitic acid, to which zirconia is in many respects similar.



§ 13. *THORINA*\*—[Th O].—*Presence in the Mineral Kingdom.*

This earth is found in combination with silicic acid, in a rare mineral, *Thorite* [ $3\text{ThO}, \text{SiO}_3 + 3\text{aq}$ ]; which has been only analyzed by Berzelius. It contains 57.91 per cent. of Thorina, besides Lime, Magnesia, Oxides of Iron, Manganese, Uranium, Tin, Silica, Alumina, Potash, Soda, and Water.

Professor Wöhler has since detected this earth in small quantity (3 per cent.) in *Pyrochlore*, from the Ural mountains.

*Examination for Thorina, (or Thoria).*

Owing to the scarcity of Thorite, I have not had an opportunity of examining a specimen for Thorina before the blowpipe. The following is the method given by Berzelius:—†

The finely powdered mineral is digested with hydrochloric acid; when the whole mass, with disengagement of chlorine, becomes converted into a yellow gelatinous mass, which is to be evaporated to dryness in a water bath. The dried mass is then to be treated with water, filtered, and sulphuretted hydrogen passed through the liquid, by which some sulphurets of lead and tin are thrown down. It is again filtered, the thorina precipitated with caustic ammonia, the precipitate filtered and edulcorated. It is now rendered impure only by the oxides of iron, manganese, and uranium. To free it from these, the precipitate is to be dissolved while still moist in dilute sulphuric acid, the solution evaporated until very little of it remains. During the evaporation, a white slightly cohering soft mass is deposited in abundance; this is neutral sulphate of thorina. It has the curious property of being very slightly soluble in hot, particularly in boiling water. The acid is then decanted off, the white salt thrown on a filter and edulcorated from the adhering mother liquor, dried, and strongly ignited, by which the earth is obtained pure and white. The decanted acid liquor, and the water of edulcoration, as they contain more thorina, are then

\* [Graham remarks, that the density of Thorina is 9.402, and, therefore, superior to all other earths.]—*Trans.*

† [This method, which is not given in Plattner, I have translated from Berzelius's *Lehrbuch*, 3te Aufl., Bd. 2., S. 397.]—*Trans.*



to be evaporated to a small bulk, saturated with carbonate of potash, and mixed with a boiling saturated solution of sulphate of potash. On cooling, the excess of sulphate of potash, with a double salt of sulphate of thorina and potash, which is perfectly insoluble in a saturated solution of the former salt, crystallize out of the liquid. It is then washed with a solution of sulphate of potash, dissolved in warm water, and precipitated with ammonia. The precipitate yields, on ignition, an earth slightly tinged yellow, by a trace of manganese, which may be purified by treating it as in the first method. It is obtained perfectly isolated only after ignition.

( $\beta$ ) EXAMINATION OF METALS AND THEIR OXIDES.

§ 1. CERIUM—[Ce].—*Presence in the Mineral Kingdom.*

Cerium occurs:—

(a) As Protoxide, in combination with Fluorine, thus, singly in *Fluoride of Cerium*  $[Ce^2Fl^3 + 3(Ce^2O^3, HO)]$ ; and with Yttria and Lime, in *Ytthro-cerite*;

(b) As Protoxide, with Carbonic Acid, in *Carbonate of Cerium*  $[CeO, CO^2]$ ;

(c) As Protoxide, in Titanic Acid combinations, namely, with Lime, Thorina, Protoxides of Uranium, Iron, and Manganese, in *Pyrochlore*; and with Zirconia, Yttria, Lime, Protoxides of Manganese and Iron, and other bases, in *Poly-mignite*;

(d) As Protoxide, in Siliceous combinations, namely:—Singly, in *Cerite*  $[3 Ce O, SO^3 + 3 aq]$ ; with Protoxide of Iron, Lime, and Alumina, in *Cerine* and *Allanite*; with Yttria and Protoxide of Iron, in *Gadolinite*; with Protoxide of Iron, Yttria, Lime, Protoxide of Manganese, and Water, in *Orthite*; and with Yttria, Protoxide of Iron, and Manganese, Alumina, Lime, together with a little Carbon and Water, in *Pyrorthite*.

*Examination for Cerium.*

In minerals, in which no other colouring matter, or at most only traces of such exist, besides protoxide of cerium,



as in Fluoride of Cerium, Ytthro-Cerite, Carbonate of Cerium, and Cerite, the cerium is readily detected with fluxes. Red or dark yellow beads are obtained with borax and microcosmic salt in the oxidating flame, according as a large or small quantity has been dissolved, and the mineral contains a large or a small proportion of protoxide of cerium; these beads, on cooling, and also when subjected to the reducing flame, lose their colour to such a degree, that the bead of microcosmic salt becomes perfectly colourless. If the mineral contains a slight trace of iron, the borax bead becomes greenish in the reducing flame.

Protoxide of cerium cannot be recognised in this way, in such minerals as contain at the same time protoxide of iron, uranium, or titanlic acid, as for instance, Pyrochlore, Polymignite, Cerine, Allanite, Gadolinite, Orthite, and Pyrorthite. The operator succeeds better with such minerals, by melting them in a finely powdered state, with from six to eight times their bulk of bi-sulphate of potash, dissolving the fused mass in a large quantity of water, and boiling the solution for some time, in order to precipitate titanlic acid, if the mineral contains any. If the substance contains a large quantity of titanlic acid, and at the same time zirconia and protoxide of iron, the titanlic acid, with the zirconia, and a part of the protoxide of iron, remains behind. If the precipitation of the titanlic acid takes place, it is to be separated by filtration, and the protoxide of cerium thrown down from the filtered solution, in the manner described at page 126; or in a far shorter way, as follows:—

First, the fluid is boiled for a short time in a test tube; if it remains clear, the whole of the titanlic acid has been separated; when this is the case, a crystal of sulphate of potash, rather more than sufficient to saturate the liquid, must be suspended in it, and the whole heated anew. If the solution contains protoxide of cerium, it falls of a white colour, as a double salt of sulphate of potash and cerium, as soon as the solution becomes saturated with the sulphate of potash. This occurs even when the quantity of cerium present is exceedingly small, the solution at first being only slightly tur-



bid, but subsequently, on allowing the vessel to stand undisturbed for some time, the undissolved portion of the sulphate of potash crystal, becomes covered with a white light friable powder. If the mineral contains zirconia, without titanitic acid, it will be thrown down along with the cerium double salt, as basic sulphate of zirconia and potash, unless, in the commencement, care be taken to remove it from the aqueous solution of the fused mass. Thorina, in case it is present, will also be thrown down with the cerium. When lime is present in the mineral, a portion of it is dissolved in the treatment of the fused mass with water, as sulphate of lime, and is also separated by the sulphate of potash. The resulting precipitate should therefore, after separation by filtration, be welledulcorated, and tested for cerium with borax; the double salt is by this means decomposed, the sulphuric acid and potash separate as sulphate of potash, and flow down the platinum wire, if the end with the loop be held higher than the other. The borax bead remains clear, provided it is not saturated, and by it a very small trace of oxide of cerium may be recognised, by the colour given at page 76.

§ 2. *MANGANESE*—[Mn].—*Presence of this Metal in the Mineral Kingdom, and in the Scorice from Smelting Works.*

Manganese occurs in nature :—

- (a) As a Sulphuret, in *Mangan-Blende* [Mn S];
- (b) As an Oxide, alone, as well as in combination with other Metallic Oxides; *e. g.*, *per se* in *Grey Oxide of Manganese* [Mn O<sup>2</sup>]; as a Hydrate, in *Black Wad* [BaO, Mn<sup>2</sup> O<sup>3</sup>, HO] and [Mn<sup>2</sup> O<sup>3</sup>, HO + 2 (Fe<sup>2</sup> O<sup>3</sup>, HO)];\*

\* [A sample analyzed by Wackenroder, gave,—

Peroxide of Manganese . . . . .	32.73	Mn O <sup>2</sup>
Peroxide of Lead . . . . .	12.33	Pb O <sup>2</sup>
Protoxide of Lead . . . . .	8.0	Pb O
Peroxide of Iron . . . . .	9.33	Fe <sup>2</sup> O <sup>3</sup>
Oxide of Copper . . . . .	4.0	Cu O
Oxide of Cerium . . . . .	0.33	Ce O
Silicic Acid . . . . .	0.13	Si O <sup>3</sup>
Quartz . . . . .	2.60	Quartz
Water . . . . .	31.33	HO

100.78—*Trans.*]



With Oxide of Cobalt and Water, in *Black Earthy Cobalt*  $[\text{CoO}, \text{Mn}^2\text{O}^3 + 3 \text{ aq}]$ ; \* and with Peroxide of Iron, Protoxide of Iron, and Oxide of Zinc, in *Franklinite*, (*Dodecahedral Iron Ore*)  $\left[ \begin{smallmatrix} \text{Mn} & \text{O} \\ \text{Fe} & \text{O} \\ \text{Zn} & \text{O} \end{smallmatrix} \right\} \frac{\text{Mn}^2\text{O}^3}{\text{Fe}^2\text{O}^3}]$ ; or, according to Kobell,  $[2 \text{ Zn O}, 3 \text{ Mn O}^2 + 5 (\text{Fe O}, \text{Fe}^2\text{O}^3)]$ ;

(c) As a Protoxide, in a Phosphoric Acid combination containing Protoxide of Iron and a small quantity of Lime; *e. g.*, *Triplite* (*Phosphate of Manganese and Iron*),  $[4 \text{ Mn O}, \text{PO}^5 + 4 \text{ Fe O}, \text{PO}^5]$ ;

(d) As Protoxide, with Tungstic Acid (Scheelie Acid) and Tungstate of Iron, in *Wolfram*  $[\text{Mn O}, \text{WO}^3 + 3 (\text{Fe O}, \text{WO}^3)]$ ;

(e) As Protoxide, with Tantalic Acid, Protoxide of Iron, Oxide of Tin, Tungstic Acid, and Lime, in various *Tantalites*;

(f) In an oxidized state, in Silicic Acid compounds; *e. g.*, *per se*, in *Red Siliciferous Oxide of Manganese*  $[3 \text{ Mn O}, \text{SiO}^3]$ ; further, with Peroxide of Iron, Alumina, and Water, in *Umber*; with Protoxide of Iron, Protochloride of Iron, and Water, &c., in *Pyrosmalite*  $[\text{Fe}^2, \text{Cl}^3 + \text{Fe}^2\text{O}^3, 6 \text{ H O}]$  or  $[4 (3 \text{ FeO}, 2 \text{ SiO}^3 + 3 \text{ Mn O}, 2 \text{ SiO}^3)]$ ; with Alumina, Lime, and sometimes Magnesia, in *Granate*;—general formula  $\left[ \begin{smallmatrix} 3 \text{ Mg O} \\ 3 \text{ Fe O} \\ 3 \text{ Ca O} \\ 3 \text{ Mn O} \end{smallmatrix} \right\} \text{Si O}^3 + \text{Al}^2\text{O}^3, \text{Si O}^3]$ ; with Protoxide of Iron, Alumina, and Water, in *Chloropal*; and also as an extremely small ingredient of numerous other Silicates.

Manganese is very seldom met with in the metallic state, in the scoriæ or recrements from the smelting process. It is generally in combination with oxygen, sulphur, or other metals, or else is taken up by the silicates which mostly compose the slags.

#### *Examination for Manganese.*

Substances containing no other metals save manganese, give coloured glasses with borax and microcosmic salt in the

\* [According to Döbereiner's analysis, the formula is  $[\text{Co}^2\text{O}^3, \text{Mn}^2\text{O}^3 + 3 \text{ aq}]$ .—*Trans.*



oxidating flame, and can be very readily recognized when dissolved in the named fluxes on a platinum wire, and the pearl treated in the reducing flame. After oxidation, the glasses appear amethystine, and by reduction colourless. The amethyst tinge is not affected on exposure to the oxidating flame, if a substance at the same time contains other colouring ingredients, but it is, in the reducing flame. If the presence of manganese is considerable, the pearl must be suddenly removed after reduction, because the colour returns if allowed to cool gradually, owing to a higher state of oxidation ensuing.

When the quantity of manganese is so inconsiderable that it affords no amethyst colour, either with borax or microcosmic salt, in the oxidating flame, a microcosmic glass must be formed, in which a sufficient quantity of the substance under examination has dissolved, and brought in contact with a crystal of nitre, as described with the re-agents, page 30; by which means the assay despumates, and the pellicle, on cooling, assumes in proportion to the quantity of manganese present, an amethystine or slight rosy appearance. This procedure is not applicable in compound combinations, in which other metals or metallic oxides present themselves, which likewise impart a colour to the borax or microcosmic glass; and recourse must be had to soda, which is decidedly the most characteristic re-agent for manganese. In a substance containing less than 0.1 per cent. oxide of manganese, the detection of this metal succeeds very readily in the following manner:—The substance under examination is reduced to an impalpable powder, mixed with from two to three times its volume of soda, and the mixture melted in the oxidating flame on platinum foil. The oxide of manganese dissolves in the soda, forming a transparent green mass, which flows round the undissolved portion, and appears distinctly bluish-green on cooling. Should the quantity of manganese be less than 0.1 per cent., this green appearance is not produced with soda alone, but when two parts of this alkali, and one part nitre, are applied, all the manganese is converted into a higher oxide; the soda is coloured characteristically green,



even by the smallest trace of this ingredient, which colour becomes distinct on cooling. A similar method must be employed for the determination of manganese in Rose Quartz. The fine quartz powder, with soda and nitre, is kneaded into a paste with water, placed in the ring of the platinum wire, and melted to a pearl before the oxidating flame. This pearl is, while hot, transparent and colourless, on cooling, opaque, and when cold, slightly, but very distinctly, green.

If the substance consists of metallic sulphurets and arseniurets, it must be well roasted on charcoal, previous to the treatment on platinum wire, with soda, or soda and nitre.

When manganese forms a constituent of a substance, at the same time containing, for instance, silica and oxide of cobalt, as in picked ores, no green is obtained with soda, but a bluish-coloured mass, consisting of silicate of soda and dissolved oxide of cobalt, which perfectly destroys the reaction of manganese. But if the silica is previously removed from the substance under examination, the slightest trace will again be perceptible. The manner of proceeding is as follows:—If the substance to be treated contains metallic sulphurets and arseniurets, it must be roasted on charcoal, and melted to a pearl, in proportion to the quantity of silicic acid, with an equal part of soda, and twice its volume of borax, on charcoal in the reducing flame. If arseniated metallic oxides are in the assay, they become reduced, and cohere, forming a readily-fusible metallic globule. This is generally the case with cobalt or nickel ores, where arsenic-cobalt, arsenic-nickel, or the three metals, are obtained in combination. On cooling, the glass is separated from the reduced metallic arseniurets, powdered, dissolved in hydrochloric acid, and perfectly evaporated to dryness; the metallic chlorides dissolved in water, and separated from the silicic acid by filtration; oxide of cobalt, oxide of nickel, protoxide of iron, protoxide of manganese, &c., precipitated from the solution by potash, in an excess of which the alumina dissolves. The precipitate, separated by filtration, washed, and dried, is examined for manganese with soda and nitre, as already described.



§ 3. ZINC—[Zn]—*Presence in the Mineral Kingdom, and in the Scoriae of Smelting Furnaces.*

Zinc is found in nature :—

(a) With Sulphur, as in *Zinc Blende* [Zn S], which is often contaminated with other Sulphurets; *e. g.*, Sulphurets of Iron, Lead, Cadmium, Silver, &c.

(b) In an oxidized state, with Earths, or other Metallic Oxides, as with Alumina, Magnesia, and Protoxide of Iron, also Aluminous Oxide of Zinc, in *Automalite* or *Gahnite*, which is an Aluminate of Zinc and of Iron. By some mineralogists it is considered as a variety of Spinel, and as it contains twenty-five to thirty per cent. of the Oxide of Zinc, it has been called *Zinciferous Spinel*,—

$= \left[ \begin{matrix} \text{Zn} & \text{O} \\ \text{Mg} & \text{O} \\ \text{Fe} & \text{O} \end{matrix} \right\} \text{Al}^2\text{O}^3$ ]; some varieties contain Silicic Acid, and traces of Manganese, Lime, Sulphur, and Cadmium; with a small quantity of Iron and Manganese, in *Red Oxide of Zinc*,\* and with a large proportion of Iron and Manganese, in *Franklinite*, or *Zinc Oxydé Ferrifère*. The formula, according to Kobell, is  $\left[ \begin{matrix} \text{Mn} & \text{O} \\ \text{Fe} & \text{O} \\ \text{Zn} & \text{O} \end{matrix} \right\} \begin{matrix} \text{Mn}^2\text{O}^3 \\ \text{Fe}^2\text{O}^3 \end{matrix} \right]$ . This formula, supposing the Manganese to exist in the state of Red Oxide, can be represented as follows:— $\left[ \begin{matrix} \text{Fe} & \text{O} \\ \text{Zn} & \text{O} \end{matrix} \right\} \left\{ \begin{matrix} \text{Mn}^3\text{O}^4 \\ \text{Fe}^2\text{O}^3 \end{matrix} \right\}$ . †

(c) As an Oxide, with Sulphuric Acid and Water, in *Sulphate of Zinc* [Zn O, SO<sup>3</sup> H O + 6 aq];

(d) As a Carbonate, in *Calamine* [Zn O, CO<sup>2</sup>]; and with Carbonic Acid and Water, in *Zinc Bloom* [2 (Zn O, 3 H O) + 3 (2 Zn O, CO<sup>2</sup>)]; and,

(e) In combination with Silicic Acid and Water, in *Siliceous Oxide of Zinc* [2 (3 Zn O, Si O<sup>3</sup>) + 3 aq]; Zinc is also found as an accompanying ingredient in many Silver,

\* [Oxide of Zinc, usually mixed with some Red Oxide of Manganese.]—*Trans.*

† [Red Oxide of Manganese [Mn<sup>3</sup> O<sup>4</sup>] is a double oxide, being a compound of single equivalents of Protoxide [Mn O] and Deutoxide of Manganese [Mn<sup>2</sup> O<sup>3</sup>]. It forms the mineral Hausmanite, which differs from Manganite in having Protoxide of Manganese in the place of Water.—*Graham.*]—*Trans.*



Lead, and Copper furnace products; *e. g.*, in the *Rohstein*,\* *Bleistein*,† *Kupferstein*,‡ *Tutty*,§ &c.

*Examination for Zinc.*

This examination is very simple; the substance either containing much or little sulphuret of zinc, or else this metal in an oxidized state, is treated with a sufficient quantity of soda, upon charcoal, in the reducing flame. Metallic zinc volatilizes, but on coming in contact with the air, is again oxidized. When a considerable quantity of this metal is present, the zinc flame is produced, and the charcoal becomes coated with flowers of zinc, but if a small quantity only, the charcoal is covered with an oxide at a short distance from the assay, and no alteration is observed in the flame.

The sublimate, which, when hot, is yellow, and after refrigeration, white, gives, when treated in the oxidating flame with a few drops of cobalt solution, a beautiful characteristic green colour, which is properly recognized only on cooling. When the assay contains much lead, or if an operator is examining metallic lead for zinc, he will find, on treating the substance in the reducing flame, that the oxide of zinc sublimate is not deposited so far from the assay as the lead one, but it may often occur that the lead sublimate is mixed with zinc. To determine if such has taken place, heat the sublimate with a solution of cobalt in the oxidating flame; the lead, if any is present, becomes reduced by the glowing charcoal, and volatilizes, while the oxide of zinc remains, and gives the characteristic green tinge. Too strong a blast must

\* [Rohstein is the product of the first smelting of Copper Ores.]—*Trans.*

† [Bleistein is a similar product, obtained in the smelting of cupriferous and other Lead Ores.]—*Trans.*

‡ [Kupferstein is a product obtained by smelting wasted Bleistein with the slags deposited in the Rohstein process, or with Quartzose Copper Ores. It contains from twenty-eight to forty per cent of copper. Kupferstein is principally composed of the Sulphurets of Copper, Lead, Iron, and Silver.]—*Trans.*

§ [Tutty consists principally of Calamine, and other volatile products, which have been eliminated in the smelting furnace.]—*Trans.*



not be directed upon the moisture, or else the zinc will separate from the charcoal, and is therefore apt to be blown away. This can be avoided by rubbing the spot upon the charcoal where the sublimate generally rests, with a few drops of a cobalt solution; a single drop of the liquid is sufficient for ascertaining the presence of a very small proportion of zinc. When this metal is present in minute quantities only, a strong flame must be deflected upon the substance, which will then become a pyrophorus, and if the assay contains lead or bismuth, they will volatilize.

§ 4. COBALT—[Co].—*Presence in the Mineral Kingdom, and in the Scoriæ of Smelting Furnaces.*

Cobalt is found in the following states :—

(a) Metallic, mixed with other metals, as with Arsenic, and sometimes traces of Iron, Copper, and Sulphur, in *White Cobalt*  $[\text{Co}, 2\text{As}]$ , at times  $[\text{Co}, 3\text{As}]$ ; with Arsenic, Iron, and sometimes a little Manganese, in *Tin-White Cobalt*  $[\text{CoAs} + \text{FeAs}]$ ; with Selenium and Seleniuret of Lead, in Seleniuret of Lead and Cobalt  $[\text{Co}, 2\text{Se} + 3(\text{PbSe})]$ , &c.\*

(b) As a Sulphuret in *Cobalt-kies*  $[2\text{Co}, 3\text{S}]$ , with often traces of Sulphuret of Iron, and Sulphuret of Copper; and with Arsenic and a trace of Iron, in *Bright White Cobalt*  $[\text{Co}, 2\text{S} + \text{Co}, 2\text{As}]$ ;

(c) As an Oxide, with Manganese and Water, in variable proportions, in black, brown, and yellow *Earthy Cobalt*;

(d) As Oxide with Sulphuric Acid and Water, in *Red Vitriol*  $[3\text{Co}, \text{SO}^3 + 8\text{aq}]$ ;†

(e) Also in an oxidized state with Arsenic Acid and Water, in *Cobalt Bloom*  $[3\text{CoO}, \text{AsO}^5 + 6\text{aq}]$ .

\* [Rose's analysis of the Seleniuret of Lead and Cobalt :—

Lead	.	.	.	.	.	63.92
Cobalt	.	.	.	.	.	3.14
Selenium	.	.	.	.	.	31.42
Iron	.	.	.	.	.	0.45

98.93—*Trans.*]

† [Winkelblech analyzed a compound which gave the following formula,— $\text{MgO}, \text{SO}^3 + 3(\text{CoO}, \text{SO}^3) + 28\text{aq}$ ].—*Trans.*



This metal also occurs, in a small proportion, in different furnace products, namely, in such as are produced from ores. To these belong, *e. g.*, Lead and Cobalt Speiss, the principal constituents of which are Nickel and Arsenic, and perhaps also many Iron, Lead, and Kupfer-steins, which fall with the slags, &c.

*Examination for Cobalt.*

Cobalt is a metal which is oxidized with great facility, and in this state it imparts to the glasses of borax and micro-cosmic salt a beautiful blue colour, both in the oxidating and reducing flames; but simple and complex combinations cannot be treated in this manner.

If, in the previously described minerals, seleniuret of lead, and the seleniuret of lead and cobalt, are excepted, this metal can be easily determined, if those which are combined with sulphur and arsenic are first roasted upon charcoal, and their fused assays treated with borax in the oxidating flame. The minerals and products which contain neither manganese nor iron, give immediately a beautiful blue glass, which, if a great addition of the substance is present, appears black and opaque. If it contains a large quantity of manganese or iron, the glass in the first case will be violet, and then green; but if it is operated upon in the reducing flame, the red colour of the manganese, and the yellow of the iron disappear, the bead acquires a bottle-green tinge, and then becomes either pure blue from cobalt, or greenish-blue from iron and cobalt. If copper or nickel are present, the oxides of these metals also dissolve in the borax, and colour the flux so, that the characteristic cobalt blue is not perceptible. If such a glass be treated sufficiently long in the reducing flame, these oxides are deposited in the metallic state, and the blue cobalt colour appears, which, if iron is at the same time present, is mixed with a bottle-green hue.

The seleniuret of lead and seleniuret of lead and cobalt, also easily-fusible furnace products, in which cobalt forms only a small constituent, are treated alone; but the difficultly-fusible furnace products, are mixed with two or three times



their volume of proof lead, and treated with borax on charcoal in the reducing flame, until the glass partakes of the colour imparted to it by the readily oxidizable and non-volatile metals. If the glass be covered with the apex of the reducing flame, so as to allow a free access of atmospheric air, only cobalt, iron, and manganese oxidize, which dissolve in the flux, and a more certain determination is effected. In the treatment of a substance containing cobalt, iron, and manganese, the iron is converted into black oxide, giving a bottle-green colour, and the manganese becomes protoxide, and dissolves without colouring the flux; therefore, the cobalt blue and bottle-green colours only result: the green tinge is very readily distinguished from the colour imparted by peroxide of iron, *per se* in the reducing flame, if the quantity of cobalt be very small. The presence of cobalt can also be recognized, if a portion of the glass, free from metallic particles, is fused in the ring of the platinum wire, and the assay then treated for a short time in the oxidating flame. The iron in this case becomes peroxide, and the glass assumes a green colour, produced by the yellow from the iron and the blue from the oxide of cobalt. When an exceedingly minute quantity of cobalt is present, the glass only partakes of the ferruginous tint, which is not affected by a small quantity of manganese, but which may be destroyed if there is much present. If the mineral under examination contains, (besides cobalt,) copper, nickel, bismuth, zinc, iron, antimony, and arsenic, a great part of the zinc, antimony, arsenic, as well as a small portion of bismuth and lead (which is added) will be volatilized, when the substance is treated with borax, the copper and nickel remaining behind unchanged. Should the glass, after subjection to a good reducing flame, present no cobalt or cobalto-ferruginous colour, but quite a foreign aspect, the assay must again be heated, and during the fusion a small quantity of the liquid glass removed from the metallic bead with the forceps, and treated for a long time upon another part of the charcoal with the oxidating flame; the reducible metallic oxides are precipitated, and the glass, upon refrigeration, acquires only the cobalt or cobalto-ferruginous tinge.



§ 5. NICKEL—[Ni].—*Presence in the Mineral Kingdom, and in the Products of Smelting Furnaces.*

Nickel occurs in nature as—

(a) Metallic with Iron, in *Native Iron*; with Arsenic in *Arsenical Nickel (Kupfernickel)* [Ni,As]; which sometimes contains traces of Cobalt, Iron, Manganese, and Sulphuret of Lead; and with a larger proportion of Arsenic, in the *Weissnickel* of the German mineralogists [Ni,2As];

(b) In the state of a Sulphuret, either *per se* or combined with other Sulphurets, namely, in a simple combination in *Sulphuret of Nickel (Haarkies)* [NiS]; in compound combinations with Antimony and Arsenic, in *Antimonial Nickel (Nickelspies-glanzerz)*, with Arsenic and Iron, in *Nickelglance* [Ni,2S+Ni,2As]; and as an exceedingly small constituent in *Siberian Needle Ore*;

(c) As Oxide with Arsenic Acid and Water, together with traces of Cobalt, Iron, and Sulphuric Acid, in *Nickel Ochre* [3Ni,AsO<sup>5</sup>+9aq]. Further, Nickel also forms a constituent of many of the products of smelting works, when the ores employed contain it. Nickel concentrates itself either in Roh-, Blei-, and Kupfer-stein, in the fusion of cupriferous Silver and Lead ores, or it deposits itself as a peculiar product in combination with Arsenic and other Arsenious alloys, called Lead Speiss. It is also found in Schwartzkupfer, obtained from the above-mentioned ores, or as a principal constituent in combination with Arsenic, in the Cobalt-speiss, which is deposited in the preparation of Smalts.

*Examination for Nickel.*

This examination is not difficult, provided the substance under investigation does not contain a combination of different sulphuretted and arsenical metals; but far more difficult and tedious when any complex combinations occur. I will here review the above-mentioned substances singly.

Native Iron, which contains only a very small quantity of Nickel, should be treated with borax on charcoal, in the oxidating flame, until the glass becomes of such a deep green



hue that it appears almost opaque on cooling, (as long as metallic iron is present, the iron dissolved cannot be perfectly raised to the state of peroxide); the undissolved iron should then be removed with the forceps from the fluid, and the latter subjected for a considerable time to the reducing flame. If the iron contains nickel, the latter will be dissolved as oxide with it, but will be again precipitated in the metallic state by a perfect reducing flame, and may be seen with the microscope in a finely-divided state, and of an almost silver-white colour, on the side of the bead tinged only with protoxide of iron. If the quantity of nickel present is so small, that it cannot be recognized in the metallic state by this method, the experiment should be repeated in the following manner:—The nickeliferous iron is to be first treated on charcoal with a moderately large quantity of borax, in the oxidating flame, until the glass is saturated; the undissolved iron is then to be removed from the fluid glass, and replaced by a small piece of pure lead. The glass must then be subjected to a good reducing flame, in order to reduce the nickel and allow it to form a button with the lead, and the whole allowed to cool. During the refrigeration a part of the nickel separates and forms a pellicle, which at first moves about irregularly on the surface of the still fluid lead button, but which becomes fixed, on the solidification of the lead, and when perfectly cold appears greyish-white. If the quantity of nickel present be not so extremely minute, the whole surface of the button will be coated, but if the contrary be the case, a part only; if none be present, the surface of the lead will be bright on cooling.

The borax glass saturated with protoxide of iron and oxide of nickel, after subjecting it, with the addition of lead, to the reducing flame, until all the oxide of nickel is reduced, can also be finely pulverized, and the powder treated with water in a porcelain capsule over the spirit lamp. The borax glass dissolves in great part in the water, and the metallic nickel, with the protoxide of iron, remains behind. If the solution be then cautiously poured off, and replaced by pure water, the presence of metallic nickel in the residue may be



soon recognized, by dipping a magnetic bar a little below the surface of the water, and carefully observing if any metallic particles are attracted by it.

The presence of Nickel in Arsenical Nickel (Kupfer-nickel), Sulphuret of Nickel, (Haarkies), Antimonial Nickel (Nickelspies-glanzerz), Nickelglance, and in the Lead and Cobalt Speiss, may be easily detected: it is merely necessary to heat a fragment in a glass tube, then roast it on charcoal, and treat the roasted specimen with borax in the oxidating flame, when the colour of oxide of nickel is obtained. If such a glass be subjected to the oxidating flame, the nickel will be reduced and deposited on the side of the bead. If the substance contains cobalt or iron, the glass becomes coloured blue or green; if, on the other hand, it be free from them, it will be colourless. When the roasted substance, which still generally contains arseniate of nickel, is fused with soda and borax, on charcoal in the reducing flame, a white metallic button is obtained; which is not, however, ductile, but which is attracted by the magnet, and must therefore be a combination of nickel with arsenic, in very small proportion. The presence of nickel in these substances, may also be recognized in the following manner:—The substance is fused on charcoal in the oxidating flame, until the greater part of the arsenic is driven off, and when antimonial nickel is under examination, the greater part of the antimony also; and the residue combines, forming slightly fuming particles. These grains are then treated for some time with borax in the oxidating flame, in order to separate the easily oxidizable metals (iron and cobalt), and the whole allowed to cool. When cold, the grain is to be separated from the glass, and again fused on charcoal with borax in the oxidating flame. If the whole of the iron and cobalt were removed by the first treatment, the glass now exhibits the colour of nickel only, but if a trace of these metals remains behind, the glass is immediately coloured by their presence; in this case the treatment of the metallic particles must be performed a third time, when the proper colour of oxide of nickel is obtained. In needle ore, as well as in cupreous iron and Bleistein, and in the



plombiferous Kupferstein, the presence of a small trace of nickel may be detected in the following manner:—The substance is to be first well roasted, and then, with soda and borax, and about 50 to 100 milligrammes of proof lead, fused on charcoal in the oxidating flame. The button of lead thus obtained, contains the easily reducible metals of the roasted substance, and the nickel also; this may be recognized during the refrigeration of the button, in the way already given under the head Native Iron. Nickel ochre may be recognized as oxide of nickel, by its behaviour to borax and microcosmic salt; if the glass containing the oxide of nickel be treated on charcoal in the reducing flame, the nickel will be separated in the metallic state, and the glass sometimes shews a slight cobalt tinge. Treated with soda and borax together, on charcoal, nickel ochre is readily reduced to an easily fusible metallic button, which, on cooling, becomes white and brittle; it communicates the peculiar colour of nickel to the fluxes in the oxidating flame.

Plombo-Nickeliferous Black Copper, which often contains a number of other metals, namely, arsenic, iron, zinc, antimony, and cobalt, when examined for nickel, must be first treated with boracic acid on charcoal, and the glass kept always covered with the reducing flame, in order that the copper and nickel may not be dissolved, while the other metals are partly volatilized, and partly taken up as oxide by the acid. When the metallic button has been so far refined, that it begins to be coated with a thin incrustation, which consists of oxide of nickel, the process should be stopped, the solid copper button removed with the forceps from the fluid slag, placed on the anvil, and struck once or twice with the hammer, in order to free it perfectly from the dross. Though this button is malleable, it does not possess the colour of pure copper, but is rather white, according as the quantity of nickel present is large or small. If it be desired to examine further into the presence of nickel, the button must be treated with microcosmic salt, on charcoal in the oxidating flame, and the glass observed, whether it becomes of a beautiful green colour, and whether it retains this colour



on cooling. This green colour is derived from the combined greenish blue of copper, and the reddish brown of nickel; in this case the greenish blue, resulting from a large quantity of copper, which alone borax glass exhibits on cooling, should be observed. As nickel is more oxidizable than copper, this beautiful green colour is obtained even when the quantity of nickel present is very small.\*

\* [The following is inserted on account of the translator's attention having been directed to it by the author.]

Plattner's method for the detection of Nickel when contained in large proportions of Cobalt :—

As the examination of oxide of cobalt for traces of oxide of nickel, in the moist way, is very unsatisfactory, I have on this account directed my attention to experiments with the blowpipe, and have arrived at the following method, which is simple, and also decisive :—Fuse in the oxidating flame, to a bead, a moderate quantity of borax, in the loop of a platinum wire, with sufficient oxide of cobalt to give an opaque glass; remove the assay, and prepare one or two similar beads, and place the whole in a charcoal cavity, with a button of pure gold, weighing from 50 to 80 milligrammes. The operator must now treat in the reducing flame, until he is satisfied that the whole of the nickel is in a metallic state; the charcoal during the action must be inclined alternately backwards and forwards, so that the gold button may flow through the molten glass, and form an alloy with the reduced particles of nickel. When the golden globule solidifies, it must be extracted with a forceps, placed between paper, and struck with a hammer, so as to detect all the adhering vitreous parts. The auriferous button, which has become more or less grey, from the presence of nickel, and also more frangible than pure gold, is now to be mixed with microcosmic salt, and heated for some time in the oxidating flame. If the borax glass has not been in the first instance over-saturated with oxide of cobalt, a bead will be now obtained, which is coloured only by oxide of nickel, and will therefore appear brownish-red while hot, and when cold reddish-yellow. Should portions of oxide of cobalt be also reduced, as the cobalt is oxidized before the nickel, either a blue glass, coloured by oxide of cobalt, or a green one (if some nickel was also oxidized) will be obtained. In either case the glass must be separated from the button, mixed with more microcosmic salt, and heated in the oxidating flame, until it acquires a tinge. If the borax glass at the commencement had not been over saturated, the colour now obtained will proceed from nickel, although the oxide of cobalt contains a trace only; but if oxide of nickel be not present, the microcosmic bead remains perfectly colourless. The auriferous button, which still contains nickel, after treatment with microcosmic salt, can be obtained again in a pure state, by smelting the alloy on charcoal, with a quantity of pure lead, and then cupelling.]



§ 6. *IRON*—[Fe].—*Presence in the Mineral Kingdom, and in the Products of Smelting Furnaces.*

Iron occurs very abundantly in nature, thus:—

(a) Metallic, as *Native Iron*, which contains some per cent. of Nickel;

(b) In a Sulphuretted state, as well *per se* as in combination with other Sulphuretted and Arsenical metals, namely, *per se* in *Magnetic Iron Pyrites* [ $\text{Fe } 2\text{S} + 6(\text{Fe}, \text{S})$ ]; in *Common Pyrites* [ $\text{Fe}, 2\text{S}$ ]; with Arsenical Iron, in *Arsenical Iron* [ $\left\{ \begin{smallmatrix} \text{Fe} \\ \text{Co} \end{smallmatrix} \right\} 2\text{S} + \left\{ \begin{smallmatrix} \text{Fe} \\ \text{Co} \end{smallmatrix} \right\} 2\text{As}$ ]; with Sulphuret of Copper, in *Copper Pyrites* [ $\text{CoS} + 2\text{FeS}$ ]; and *Purple Copper* [ $3\text{CoS} + \text{Fe}^2\text{S}^3$ ]; with Sulphuret of Antimony, in *Hardingerite* [ $3\text{FeS} + 2\text{Sb}^2\text{S}^3$ ]; with Sulphuret of Antimony, Sulphuret of Silver, Sulphuret of Copper, and Sulphuret of Zinc, in *Weissgiltigerz* and *Graugiltigerz*; with the same Sulphurets and an admixture of Sulphuret of Arsenic, in *Grey Copper* [ $\left\{ \begin{smallmatrix} 4\text{FeS} \\ 4\text{ZnS} \end{smallmatrix} \right\} \left\{ \begin{smallmatrix} \text{Sb}^2\text{S}^3 \\ \text{As}^2\text{S}^3 \end{smallmatrix} \right\} + 2(4\text{Cr}, \text{S}) \left\{ \begin{smallmatrix} \text{Sb}^2\text{S}^3 \\ \text{As}^2\text{S}^3 \end{smallmatrix} \right\}$ ]; with Sulphuret of Copper and Sulphuret of Arsenic, in *Tennantite*; and with Sulphuret of Copper, Sulphuret of Arsenic, a little Sulphuret of Silver, and Sulphuret of Antimony, in the *Copper Blende*;

(c) As Protoxide or Peroxide, as well *per se* as combined with Metallic Oxides and Water, namely, as pure Oxide, in *Specular Iron* [ $\text{Fe}^2\text{O}^3$ ] (*Red Ironstone, Bloodstone*); as Oxide, combined with Water, and often with small quantities of Oxide of Manganese and Silicic Acid, in *Brown Iron Ore* and *Bog Iron Ore* [ $2\text{Fe}^2\text{O}^3 + 3\text{HO}$ ], which last contains some Silicic and Phosphoric Acids; as Oxide, contaminated with earthy substances, in *Argillaceous Iron Stone* (*Thoneisenstein*); as Black Oxide, with a very small admixture of Magnesia, in *Oxidulated Iron Ore* [ $\text{FeO} + \text{Fe}^2\text{O}^3$ ]; as Oxide, with Oxide of Chromium, Alumina, and Magnesia, in *Chromate of Iron* [ $\left\{ \begin{smallmatrix} \text{FeO} \\ \text{MgO} \end{smallmatrix} \right\} + \left\{ \begin{smallmatrix} \text{Cr}^2\text{O}^3 \\ \text{Al}^2\text{O}^3 \end{smallmatrix} \right\}$ ]; as Peroxide, with Oxide of Zinc, and Protoxide of Manganese, in *Franklinite* or *Dodecahedral Iron Ore*; and as Protoxide, with Oxide of Zinc, Alumina, Magnesia, and a very little Silicic Acid, in *Automolite*.

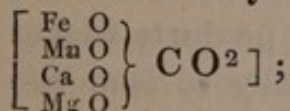


(d) As Protoxide, with Titanic Acid, in various degrees of saturation, in *Titaniferous Oxidulated Iron*, *Crichtonite*, *Mennaccanite*, *Nigrin*, *Iserine*, *Ilmenite*, &c.;

(e) As Protoxide, with Tungstic Acid and Tungstate of Protoxide of Manganese, in *Wolfram*;

(f) As Peroxide, with Arsenic Acid and Water, in *Skorodite*; as Black Oxide, with Arsenic Acid and Water, in *Arseniate of Iron (Cube Ore)* [ $3\text{FeO}, \text{AsO}^5 + 3\text{Fe}^2\text{O}^3, 2\text{AsO}^5 + 18\text{aq}$ ]; as Peroxide, with Arsenic Acid and Water, in *Pitchy Iron Ore* [ $2\text{Fe}^2\text{O}^3, \text{AsO}^5 + 12\text{HO}$ ];

(g) As Protoxide, with Carbonic Acid, and often with Carbonate of Protoxide of Manganese, with traces of Lime, Magnesia, and Water, in *Spathose Iron*, the general formula of which may be expressed by the following symbol—



(h) As Protoxide, with Sulphuric Acid and Water, in *Native Green Vitriol* [ $\text{FeO}, \text{SO}^3 + 6\text{aq}$ ]; as Oxide, with Sulphuric Acid and Water, in *Vitriol-oker* [ $2\text{Fe}^2\text{O}^3\text{SO}^3 + 6\text{aq}$ ];

(i) As Protoxide, with Phosphoric Acid and Water, in *Phosphate of Iron* [ $8\text{FeO}, 3\text{PO}^5 + 16\text{aq}$ ] or [ $3\text{FeO}, \text{PO}^5 + 6\text{aq}$ ]; as Protoxide, with Phosphoric Acid, and Phosphate of Protoxide of Manganese, with an admixture of Phosphate of Lime, in *Triplite*; as Oxide, with Phosphoric Acid and Water, in *Grüneisenstein*; and as Oxide, with Phosphoric Acid, Phosphate of Alumina, small quantities of Silicic and Hydrofluoric Acids, Lime, and Water, in *Kakozene*;

(k) In the oxidized state, with Silicic and Siliceous combinations, namely, as Protoxide, in *Hisingerite*, *Sideroschisolite*, and *Chlorophaeite* (Berzelius considers these three Silicates as simple, in which the intermingled substances are to be looked upon as extraneous); as Protoxide, with Silicate of Protoxide of Manganese, Protochloride of Iron and Water, in *Pyrosmalite*; as Protoxide, with Water and a little Alumina, in *Chamoisite*; as Protoxide, with Lime, Protoxide of Manganese, and Peroxide of Iron, in *Lievrite* [ $2 \left\{ \begin{array}{l} 3\text{CaO}, \text{SiO}^3 \\ 3\text{FeO}, \text{SiO}^3 \end{array} \right\} + 2\text{Fe}^2\text{O}^3, \text{SiO}^3$ ]; as Protoxide, with Soda, Water, and a



little Lime, in *Krokydolite*; as Peroxide, with Water and a little Protoxide of Manganese and Alumina, in *Chloropal*; as Peroxide or Protoxide, with Protoxide of Manganese, and generally with a little Alumina, Lime, and Magnesia, in many *Granites*; as Peroxide, with a little Peroxide of Manganese and Lime, in *Achmite*; as Protoxide, with Magnesia, and sometimes with the Oxides of Nickel and Manganese, in *Chrysolite* and *Olivine*; as Protoxide, with Protoxide of Cerium and Yttria, in *Gadolinite*; and as a secondary constituent in a number of other Silicates.

Iron also occurs in many furnace products, from Silver, Lead, Copper, and Tin ores, and in most slags, either as a principal or secondary ingredient. It would be superfluous to allude further to these products, as a part of the iron contained in the ores to be smelted, and the raw products which are to undergo further operations, is generally so long retained by them, that its presence can be detected even in Black Copper and Tin.

*Examination for Iron.*

The test for Iron, is, that the peroxide, as well as the protoxide, communicate a peculiar colour to borax and microcosmic salt, and that the iron is with difficulty separated from both these fluxes in a metallic state only. The operator should consider whether he has to do with metallic alloys alone, or combinations of metal with arsenic, or with oxides. If the substance under examination be of pure metallic alloys, composed of difficultly-fusible metals, it should be fused with borax on charcoal, in the oxidating flame, until the operator is certain that, besides other metallic oxides, protoxide or peroxide of iron has been dissolved also. If the compounds, however, contain much lead, tin, bismuth, antimony, or zinc, and that they readily fuse, the reducing flame should be employed; the flame should be principally directed on the glass, in order that too much of the latter metals may not be oxidized and dissolved. In both cases the glass, while still soft, is either removed from the metallic particle, and treated on any other part of the charcoal, in the reducing



flame, (where the easily-reducible metals will be separated, and the borax glass appear coloured of a bottle-green, from the black oxide of iron,—provided only oxide of cobalt does not prevent the reaction), or it is immediately treated, together with the particles of alloy, in a pure strong reducing flame, when the reducible metals which may happen to be dissolved are also precipitated in the metallic state, and again combine with the remainder of the alloy. If the alloy contains much tin, or if the bottle-green glass be treated on another part of the charcoal, with a particle of tin, for a few moments, in the reducing flame, the iron will be perfectly reduced to the state of protoxide, and the glass, on cooling, will be of a pure vitriol-green colour.

If the borax exhibits a blue colour, instead of the proper vitriol-green of protoxide of iron, oxide of cobalt is present, which destroys the iron tint. In this case the glass must be again softened in the reducing flame, the greater part of it removed from the charcoal with the forceps (but without taking any of the metal with it), and fused in the loop of the platinum wire. If the colour is then so deep that the glass is almost opaque, it must be flattened with the forceps while still soft, a part of it struck on the anvil, and the portion still adhering, diluted with more borax. The first thing then to be done, is to fuse the glass in the oxidating flame, until the protoxide of iron present is converted into the peroxide, and the glass is coloured while warm of a brownish-red or yellow. If the glass contains a slight trace of peroxide of iron only, it appears green while hot, and pure blue when cold. If the quantity of iron be somewhat considerable, the glass will be dark green while hot, and when cold of a beautiful green, because peroxide of iron, when not in excess, communicates to borax glass a yellow colour when cold.

The combination of metals with sulphur and arsenic, can be examined for iron in two ways. According to the first method, the specimen should be roasted on charcoal, small portions are then to be gradually dissolved in borax, on the platinum wire in the oxidating flame, and the glass examined



while hot and when cold, in order to see if it displays any colour. The proper iron tint is obtained immediately, with many of these compounds, which contain only metals whose oxides do not produce very intense colours; with many others, it is not however obtained, as for example, those which contain copper, but on the contrary a green colour results, which becomes lighter during the refrigeration, and when cold, possesses a colour composed of the yellow of oxide of iron, and the blue of oxide of copper. In this case the glass must be shaken from the platinum wire (see page 55) and treated on charcoal in the reducing flame, until the copper separates, and the glass exhibits the peculiar bottle-green tint of black oxide of iron, if oxide of cobalt is not present at the same time. The bottle-green glass can also be treated with tin, in order to recognise the vitriol-green colour of iron. The second method is as follows: the pulverized substance is mixed with proof lead and borax, and the whole fused on charcoal in the reducing flame, until the borax glass is coloured by the difficultly-reducible metals present. In the commencement, the mixture should be covered with the reducing flame; but as soon as the borax has run to a globule, the flame should be directed only on the latter, and free access of air allowed to the fused metal.

Compounds which readily fuse on charcoal, *per se*, can be treated without proof lead. An exceedingly small quantity of iron may in this way be detected in many Lead-Glances, particularly when the glass is treated with tin. If the borax exhibits a blue instead of a green colour, the glass must be further treated in the manner mentioned above for the metallic compounds.

In the compounds of oxide of iron with other metals, or with earths and acids, the iron is also best detected by fusion with borax.

Such compounds as may be supposed to contain neither oxides of copper, nickel, chromium, nor uranium, should be dissolved with borax on the platinum wire, in the oxidating flame, and the coloured bead examined while hot, and also during the refrigeration; if it exhibits the colour of iron, or



of iron and cobalt together, of which we have already spoken, the examination is complete; but if it presents another colour, as violet passing into red, the glass must be treated for some time longer in the reducing flame, by which the violet which is derived from manganese disappears, and the bottle-green colour of black oxide of iron remains. If such a substance contains much manganese, the glass treated in the oxidating flame will be a perfect dark red, while hot, and red when cold. In this case it is not possible to convert the manganese into protoxide on the platinum wire, it should therefore be removed from the wire to charcoal, and treated with tin, when the colour of the manganese disappears, and the vitriol-green colour of the protoxide alone remains, when oxide of cobalt is not present.

If a substance, besides iron and oxide of manganese, contains cobalt also, the glass treated with the oxidating flame appears more or less of a dark violet colour, which becomes when treated for a short time in the reducing flame, green while hot, and blue when cold; for example, this is the case with brown earthy cobalt, which really consists of oxide of cobalt and hydrate of oxide of manganese, but which is contaminated with oxide of iron. In a combination of much oxide of manganese, and oxide of cobalt, with very little oxide of iron, the last may be readily detected, by fusing the substance with bi-sulphate of potash, dissolving the fused mass in water, adding a few spoonfuls of sal-ammoniac to the solution, warming the whole, and when the salt is dissolved, precipitating the oxide of iron by ammonia. If the iron exists in the substance as protoxide, a few drops of nitric acid should be added to the sulphuric acid solution, and the whole warmed, in order to convert the protoxide into the peroxide. The oxide of iron can then be filtered from the solution,edulcorated, and tested with borax on the platinum wire. If the substance under examination contains oxide of copper, or nickel, it is always better to dissolve it with borax on charcoal, in the oxidating flame, then treat it in the reducing flame, in order to separate the copper and nickel in the metallic state, and thus obtain the proper ferruginous tint alone.



If a blue glass is obtained, it must be re-oxidized on the platinum wire, in the way already mentioned. When such a substance contains oxide of chromium, a green glass is obtained with borax, which shews the presence of chromium only. In such cases, the substance must either be mixed with three parts of saltpetre and one of borax, this mixture gradually fused in the loop of the platinum wire, the chromate of potash thus formed, dissolved in water, and the residue, afteredulcoration with water, dissolved with borax on a platinum wire, when the proper colour of iron will be obtained, provided no other coloured oxide be present, and all the oxide of chromium removed; or the iron can be reduced by soda on charcoal, and the metal obtained by rubbing the glass in a mortar, and separating the non-metallic parts. If the substance contains uranium, the ferruginous colour will also be obtained, but it will not proceed from the iron alone, but partly from uranium, which yields almost the same colour as iron. To obtain the proper colour of iron, the substance should be melted with bi-sulphate of potash, the fused mass dissolved in water, the protoxides of iron and uranium present converted into peroxides, by heating the solution with a few drops of nitric acid, and an excess of carbonate of ammonia added. The oxide of uranium which is at first precipitated along with the oxide of iron, re-dissolves again, so that the latter can be obtained by filtration, and after washing well with water, may be tested with borax. When the ammoniacal solution is boiled, the oxide of uranium falls as a yellow powder, and can also be tested before the blowpipe, being easily recognised with microcosmic salt.

If the substance contains tungstic or titanitic acids, the proper colour of oxide of iron is obtained with borax and microcosmic salt, in the oxidating flame, as both these acids yield only a slight yellow colour; in the reducing flame, on the other hand, the microcosmic salt glass becomes, during the refrigeration, more or less of a blood-red colour.

§ 7. *CADMIUM*—[Cd].—*Presence in the Mineral Kingdom.*

Cadmium occurs only in Zinc ores, thus:—



- (a) In a sulphuretted state, in many *Zinc-blendes*, as for example, in the *Splendent fibrous Blende*, from Przibram, and,  
 (b) As Oxide, in *Siliceous Oxide of Zinc* or *Galmei*.

The quantity present, does not, however, exceed from one to four per cent., either in the *Blendes* or in the *Galmei*.

*Examination for Cadmium.*

This metal can be recognized as oxide before the blow-pipe only, owing to its volatility. The cadmiferous mineral should be heated for some time in a pulverized state, on charcoal in the reducing flame, by which the metallic cadmium is volatilized, and in contact with the air, is immediately re-oxidized. The greatest part of this oxide deposits itself on the charcoal, and when perfectly cold, may be recognised by its reddish-brown colour; in a thin crust, however, it appears only yellow. When the quantity of cadmium present is not more than one per cent., it is generally difficult to obtain a sublimate of oxide of cadmium in this way; but if the powdered mineral be mixed with soda, and this mixture treated for a few moments on charcoal, in the reducing flame, a very evident sublimate of oxide of cadmium is obtained. If the blast be continued too long, a part of the zinc will also be volatilized, and will likewise deposit itself as oxide on the charcoal. The oxide of cadmium is, however, always deposited further from the assay than the zinc, and may be very easily distinguished.

§ 8. *LEAD*—[Pb].—*Presence in the Mineral Kingdom, and in the products of Smelting Furnaces.*

Lead is pretty generally distributed in nature. It is found:—

- (a) Metallic, in combination with other metals; *e.g.*, with Tellurium, in *Black Tellurium* (*Pyramidal Tellurium Glance*) [Pb Te] (mechanically mixed with Sulphurets of Lead and Gold); with Tellurium, Gold, and Silver, in *Yellow Tellurium* [Ag Te + 2(Pb, Te) + 3(2Au, 3Te)]; with Selenium, in *Seleniuret of Lead*, which often contains a trace of Cobalt; with Selenium and Copper, in *Seleniuret of Lead and Copper*



[  $\text{Pb Se} + \text{Cu Se}$  ], and *Seleniuret of Copper and Lead* [  $2 (\text{Pb Se}) + \text{Cu Se}$  ]; and with Selenium and Mercury, in *Seleniuret of Lead and Mercury* [  $\text{Pb Se}$  mixed with  $\text{Hg Se}$  ];

(b) In a sulphuretted state alone, and in combination with other metallic sulphurets, namely; *per se* in *Lead Glance* [  $\text{Pb S}$  ]; with Antimony and traces of Iron, Zinc, and Copper, in *Jamesonite* (*Acotomous Antimony Glance*) [  $2 (\text{Pb S}, \text{Sb}_3 \text{S}) + \text{Pb S}$  ]; with Antimony and Copper, in *Bournonite* [  $3 (\text{Cu S}) + \text{Sb}_3 \text{S} + 2 (3 \text{Pb}_3 \text{S} + \text{Sb}_3 \text{S})$  ]; with Bismuth, and a little Iron and Copper, in *Plumbo-Cupriferous Sulphuret of Bismuth*; with Antimony and a small quantity of Copper, in *Zinkenite* [  $\text{Pb S} + \text{Sb}_3 \text{S}$  ]; and with Bismuth, Copper, and a very small quantity of Tellurium and Nickel, in *Siberian Needle Ore* [  $\text{Cu S}, \text{Bi S} + 2 (\text{Pb S}, \text{Bi S})$  ];

(c) In combination with Chlorine; as in *Chloride of Lead* (*Cotunnite* from Vesuvius) [  $\text{Pb Cl}$  ]; and the *Basic Chloride of Lead* [  $\text{Pb Cl} + 2 \text{Pb O}$  ], from the Mendip hills, of Somersetshire;

(d) In an oxidized state, with Alumina and Water, in *Plombgomme* (*Hydrous Aluminate of Lead*) [  $\text{Pb O}, 2 \text{Al}^2 \text{O}^3 + 6 \text{aq}$  ]; and,

(e) As an Oxide, with Acids, partly alone, and partly combined with other Metallic Salts, *e. g.*:—

(1) With Carbonic Acid, in *White Lead Ore* (*Weissbleierz*) [  $\text{Pb O}, \text{CO}^2$  ]; and in *Black Lead Ore* (*Schwarzbleierz*), of the same composition, only containing a small proportion of free Carbon; also in *Earthy Carbonate of Lead* (*Bleierde*), which is mixed with Alumina, Silica, and Peroxide of Iron.

(2) With Sulphuric Acid, and about two per cent. of Water, in *Native Sulphate of Lead* [  $\text{Pb O}, \text{SO}^3$  ];

(3) With Arsenic or Phosphoric Acid, and Chloride of Lead, in *Brown and Green Arsenical Lead Ores* [  $\text{Pb Cl} + 3 (\text{Pb}^3 \text{As O}^5)$  ];

(4) With Chromic Acid, in *Prismatic Lead Spar* (*Chromate of Lead*) [  $\text{Pb O}, \text{Cr O}^3$  ]; with Chromic Acid and Chromate of Copper, in *Vauquelinite* (*Hemi-Prismatic Olive Malachite*) [  $3 \text{Cu}, 2 \text{Cr O}^3 + 2 (3 \text{Pb O}, 2 \text{Cr O}^3)$  ];



(5) With Molybdic Acid, in *Molybdate of Lead* (*Gelbleierz*) [ $\text{PbO}, \text{MoO}_3$ ];

(6) With Tungstic Acid, in *Scheelitine* (*Tungstate of Lead*) [ $\text{PbO}, \text{WO}_3$ ]; and

(7) With Vanadic Acid, in *Vanadate of Lead* (*Vanadiniferous Lead Spar*) [ $2\text{PbO} + \text{PbCl} + 3\text{PbO}, 2\text{VO}_3$ ];

Further, Lead forms an essential, and also an accompanying constituent in many furnace products:—

(a) Metallic, combined with other Metals; *e. g.*, with Silver, in *Workable Lead*; with Copper, as *Plombiferous Black Copper* (*Bleiisches Schwartzkupfer*), &c.

(b) In a Sulphuretted state, combined with other Metallic Sulphurets, as for example: with Sulphuret of Iron, in *Bleistein*; with Sulphuret of Copper, in *Bleischen Kupferstein*; and, according to the nature of the ores smelted, mixed also with various other Metallic Sulphurets, in *Tutty*, &c.

(c) In an oxidized state, to which belongs *Glätte* (*Litharge*) and the *Abstrich*, a greyish froth which is raked off the surface of the Workable Lead, in the process of extracting Silver therefrom. The Lead is first melted at a low heat, when the *Abstrich*, which is composed for the most part of Sulphurets of Lead, Antimony, &c., separates. Further, the *Cupel Grounds* saturated with Oxide of Lead, obtained in the refinement of Lead for Silver, which fall in the melting of *Plombiferous Schlichs*.\*

#### *Examination for Lead.*

The qualitative examination for Lead is very readily performed, in the following manner:—

When plombiferous compounds, which are met with in nature and furnace products, are treated on charcoal, in the oxidating flame, they give a sublimate which is very easily recognized. Other easily volatilized metals, which may be in combination with the lead, either fume away entirely, or also deposit an oxide upon the support. The oxide of lead sublimate, which is dark lemon-yellow, while hot, and sulphur-

\* [Metallic slimes, obtained in the washing of powdered ores.]—*Trans.*



yellow when cold, deposits nearer to the assay than the sublimate of some other metallic oxides, namely: those of tellurium, selenium, antimony, and arsenic, and is by this means distinguished. Should zinc also be an ingredient, the sublimate of oxide of lead will probably be contaminated with a quantity of the oxide of this metal, but the sulphur-yellow colour of the lead deposit, cannot however be mistaken, when the assay has perfectly cooled. Plombiferous sulphuretted compounds can be examined for lead by two methods. Either by treating them in the reducing flame on charcoal, with a small addition of borax to separate the iron, and obtain a lead sublimate, or by roasting the sample moderately on charcoal, and reducing with soda, when the lead is obtained pure by sifting the assay from carbonaceous matters, &c. If the substance contains bismuth, a deposit of this oxide will ensue in the first treatment, but it is darker than the oxide of lead; and in the second a friable metal is obtained, which, when the bismuth is present in an appreciable quantity, is supposed not to be lead. Should this occur, the substance must be smelted with bi-sulphate of potash, as given under the head of Bismuth. The sulphate of lead procured in this manipulation, is heated with soda on charcoal, by which means the pure metal, and an oxide of lead sublimate, is obtained. If much copper had also been present, black copper would result in the second method, but being so dissimilar from the oxide of lead, no mistake could occur in the discrimination.

Combinations of lead with chlorine, and substances which contain the lead in an oxidized state, with other metallic oxides, earths, or acids, must always be treated with soda on charcoal, in the reducing flame. By this means, metallic and oxide of lead are produced, which may be procured on sifting the pyrognostic residue from scoriæ. Should impure lead be obtained by this method, there must have been other metallic oxides present in the specimen; therefore, to insure an infallible test of the presence of this metal, the assay must be treated for a long time in the oxidating flame, when the characteristic lead sublimate is procured.



§ 9. *BISMUTH*—[Bi].—*Presence in the Mineral Kingdom, and in the products of Smelting Furnaces.*

Bismuth is found in nature:—

(a) Metallic, as *Native Bismuth* [Bi], which generally contains a very small quantity of Arsenic; with Tellurium, Sulphur, and a trace of Selenium, in *Telluric Bismuth* [BiS+4(Bi,Te)]; and also as a small ingredient in a compound of Arsenic, Cobalt, Iron, and a little Copper, Nickel, and Sulphur, with a trace of Manganese, in *Bismuth Cobalt Ore*, from Schneeberg;

(b) In a Sulphuretted state, *per se*, and also in combination with other Metallic Sulphurets, namely: *per se*, in *Bismuth Glance* [BiS]; with Copper, in *Cupreous Bismuth* (*Cuprififerous Sulphuret of Bismuth*) [ $3(\text{CuS}) + 2\text{Bi}, 3\text{S}$ ]; with Lead, Copper, a little Nickel and Tellurium, in *Siberian Needle Ore*; and with Lead, Silver, a little Iron and Copper, in *Bismuthic Silver*;

(c) As an Oxide, in *Bismuth Ochre* [BiO<sup>3</sup>]; with, probably, traces of Iron and Copper; and also as an Oxide, with Carbonic Acid and Water, mixed with a little Peroxide of Iron, Alumina, and Silica, in *Carbonate of Bismuth*.

This metal sometimes presents itself as a secondary ingredient in many furnace products. It is found, for example, more or less in the metallic state, in many *Cobalt Speisses*, if the Cobalt Ores employed for the preparation of Smalts are not free from Bismuth.\*

*Examination for Bismuth.*

The simplest method for detecting Bismuth in the above-mentioned minerals and cobalt-speiss, is by treating those substances which contain bismuth as a metal, with or without sulphur, combined with a small portion of borax, on charcoal, in the oxidating or reducing flame; and those which contain this metal in an oxidized state, treated with soda, likewise on charcoal in the reducing flame. The bismuth,

\* [Smalts (Silicate of Cobalt) is prepared in a pure state by precipitating Sulphate of Cobalt with Silicate of Potash. It is manufactured in very large quantities in Saxony.]—*Trans.*



which is either already met with in a metallic state, or is reduced by soda, sublimes by degrees, and coats the charcoal with an oxide, which, while hot, appears dark orange-yellow, and on perfectly cooling, citron-yellow.

If the substance at the same time contains much lead, the colour of the deposit is lighter, and very similar to the lead sublimate; this occurs with the Siberian Needle Ore. In such cases, the presence of bismuth cannot be ascertained with certainty by the previous method, and it is necessary to recur to other methods of procedure.

The first is:—The substance is roasted, (if it contains sulphur or arsenic, it is preferable to perform this experiment in a glass tube, to drive these off as much as possible, because it sinters readily on charcoal), dissolved in microcosmic salt by the aid of the oxidating flame, and the glass treated with tin for a short time in the reducing flame. If the quantity of bismuth is not so small that it contains less than a fourth part of the lead present in the substance, the microcosmic salt will be coloured, on perfectly cooling, dark grey, and becomes ultimately opaque. As oxide of antimony produces the same reaction with tin in microcosmic salt, it is necessary that the absence of antimony be ascertained by a preliminary examination of this metal. If the substance, besides bismuth, also contains copper, the microcosmic salt becomes on cooling, brownish-grey, nearly black, and opaque.

During the roasting of a substance, very rich with bismuth, in a glass tube, a yellowish-white sublimate is generally formed close to the assay, and even on the undermost part of the tube, which melts in a strong flame to brownish orbicles; which, on cooling, are transparent and of a yellow colour. This deposit consists of oxide of bismuth.

The second method is the following, if the bismuth present is exceedingly small, and the lead not distinct:—The substance is well roasted in a powdered state, the heated mass fused in a platinum spoon with bisulphate of potash, and the residuary mass treated with water in a porcelain basin, over the flame of the lamp, till solution takes place. By this means the sulphate of potash, and other soluble sulphates



are dissolved, while basic sulphates of lead and bismuth remain. The supernatant liquor is decanted cautiously, the residue again treated with distilled water, a few drops of nitric acid added, and the whole heated. The sulphate of bismuth dissolves, while the sulphate of lead remains behind. If both salts be then separated by filtration, and the oxide of bismuth precipitated from the solution by microcosmic salt, with the application of heat, a white precipitate is obtained, which dissolves either colourless or yellowish in microcosmic salt; but the glass, when treated with tin in the reducing flame, on charcoal, assumes a dark greyish colour on cooling, and behaves precisely like oxide of bismuth.

The artificially-prepared metallic nickel, on a large scale, is not always free from bismuth; should an operator, therefore, wish to determine a small admixture of bismuth in the nickel, before the blowpipe, he must mix one part of the finely divided sample with two parts saltpetre, and treated on a platinum wire in the oxidating flame. The pyrognostic assay, which consists of potash, oxide of nickel, and oxide of bismuth, is detached from the wire, and treated for some time on charcoal in the reducing flame. The oxide of bismuth is very readily reduced by this means; the metallic bismuth sublimes, and coats the charcoal with an oxide.

§ 10. URANIUM—[U].—*Presence in the Mineral Kingdom.*

Uranium, which is an exceedingly rare metal, occurs in nature in an oxidized state:—

(a) As friable and compact Hydrate of the Peroxide of Uranium, the last of which is mixed with Lime and Oxide of Lead; the two varieties are called *Uran Ochre*;

(b) As an Oxide, with Phosphoric Acid, and Phosphate of Lime, in *Uranite*  $[3\text{CaO}, \text{PO}^5 + 2(\text{U}^2\text{O}^3, \text{PO}^5) + 24\text{aq}]$  mixed with  $[3\text{BaO}, \text{PO}^5]$ ; in an oxidized state, with Phosphoric Acid, and Phosphate of Copper, in *Chalkolite* (*Green Uranite*)  $[3\text{CuO}, \text{PO}^5 + 2(\text{U}^2\text{O}^3, \text{PO}^5) + 24\text{aq}]$ ;

(c) As Protoxide, with Titanic Acid, Lime, Oxide of Cerium, Protoxide of Manganese, Peroxide of Iron, Oxide of Tin, Water, and traces of Hydrofluoric Acid and Mag-



nesia, in *Pyrochlore*, from Fredrikswärn, (Wöhler's analysis);

(d) As Peroxide, with Tantallic Acid and Yttria, in *Yellow Ytthro-tantalite*  $[3 \text{Y} \text{O} \{ \frac{\text{Ta} \text{O}^3}{\text{U}_2 \text{O}^3} \}]$ ; and,

(e) As Protoxide, with Silicic Acid, and traces of Protoxide of Iron, Sulphuret of Lead, and Oxide of Cobalt, in *Pitch-Blende (Uran-pecherz)*  $[3 \text{U} \text{O}, 2 \text{Si} \text{O}^3]$ .

*Examination for Uranium.*

In the previously described minerals (with the exception of Chalkolite and Pyrochlore), Uranium is determined by treating them in a powdered state with microcosmic salt, upon platinum wire, as well in the oxidating as in the reducing flame. (See pages 78, 79.)

Pyrochlore, on account of containing a considerable quantity of titanlic acid and iron, destroys the colour given by uranium in the reducing flame; the glass, upon cooling, becoming blood-red. In the oxidating flame, however, the microcosmic salt bead becomes greenish upon refrigeration, but is contaminated with yellow. When the substance contains only a small quantity of uranium, and much iron, the ferruginous reaction is given both with borax and microcosmic salt; therefore, the sample must be fused with bisulphate of potash, the residue dissolved in water, nitric acid added to convert the iron and uranium into peroxides, and then carbonate of ammonia poured into the liquid for their separation,—which is treated of under Iron.

If the mineral contains oxide of copper, as is the case with the Chalkolite, from Cornwall, a green glass is also obtained with borax and microcosmic salt, in the oxidating flame. As minerals containing pro- and per-oxide of iron, and oxide of copper, without uranium, give a similar reaction to the above substances, when examined for this metal, they must be submitted to a different treatment. The substance is to be smelted with soda and borax, with an addition of lead, upon charcoal in the reducing flame, until the whole of the copper is reduced and alloyed with the lead.

The glass, when cold, must be pulverized, treated with hydrochloric acid and water, the protoxides of uranium and



iron, if present, converted into peroxides, by the addition of a few drops of nitric acid, and then carbonate of ammonia added in excess, and the subsequent part of the process conducted in the same manner as mentioned under the head of Iron.\*

§ 11. *COPPER*—[Cu].—*Presence in the Mineral Kingdom, and in the products of Smelting Furnaces.*

This metal is found very abundantly in nature :—

(a) Metallic, as *Native Copper* [Cu];

(b) With Selenium, as well *per se*, as with other Seleniurets, namely; *per se*, in *Seleniuret of Copper* [2Cu, Se]; with Lead, in *Seleniuret of Copper and Lead* and *Seleniuret of Lead and Copper*; and with Silver, &c., in *Eukairite*† [2CuSe+AgSe];

(c) In a sulphuretted state, alone, and in combination with other metallic sulphurets, namely: *per se*, as *Vitreous Copper* (*Kupfer-glanz*) [CuS] (which generally contains traces of Iron and Lead); with Iron, in *Purple Copper* (*Buntkupfererz*) and *Copper Pyrites*; with Arsenic and Iron, in *Tennantite* [ $\frac{4\text{FeS}}{4\text{CuS}}\} 2\text{As}, 3\text{S} + 2(4\text{Cu}, 4\text{S}, 2\text{As}, 3\text{S})$ ]; with Silver and a little Iron, in *Argentiferous Copper Glance* (*Silberkupfer-glanz*) [CuS+AgS]; with Arsenic, Iron, a little Antimony and Silver, in *Copper Blendes*; with Tin, and

\* [According to Liebig, Uranium may be extracted from Pitch-blende (a variety of Uran Ochre) by the following process: after heating the mineral to redness, and reducing it to an impalpable powder, it is digested in pure nitric acid, diluted with four parts of water, taking the precaution to employ a larger quantity of the mineral than the acid added can dissolve. By this process, the protoxide of uranium is converted into peroxide, which unites with the nitric acid, almost to the total exclusion of the iron. A current of hydrosulphuric acid gas is then transmitted through the menstruum, in order to separate lead and copper, the sulphurets of which are always present in Pitch-blende. The solution is boiled, to expel any free acid, and after being concentrated by evaporation, is allowed to repose. The nitrate of peroxide of uranium crystallizes out in flattened four-sided prisms, of a beautiful lemon-yellow colour.

The peroxide of Uranium is employed in the arts, for imparting a fine orange colour to porcelain, &c.]—*Trans.*

† [From the Greek, signifying *opportune*; in allusion to its discovery just as Berzelius had completed his examination of Selenium.—*Phillips.*]—*Trans.*



a little Iron, in *Tin-Pyrites* [ $\text{CuS} + \text{SnS}$ ];\* with Antimony, Silver, Iron, and Zinc, in *Weissgiltigerz* (a mechanical mixture of *Brittle Sulphuret of Silver* with *Grey Antimony*, &c.); with the same Sulphuretted Metals and Sulphuret of Arsenic, in *Grey Copper*; with Antimony and Silver, in *Antimonial Grey Copper*; and with Lead, Antimony, and a little Iron, in *Bournonite*; with Silver, a little Iron and Arsenic, in *Gansekothigerz*; with Bismuth, in *Cupreous Bismuth*; and with Bismuth, Lead, a little Nickel, and Tellurium, in *Siberian Needle Ore*;

(d) In an Oxidized state, either alone or in combination with other Metallic Oxides and Water, namely: as Suboxide, in *Red Oxide of Copper* (*Rothkupfererz*) [ $\text{Cu}_2\text{O}$ ]; as Oxide, with Sesqui-oxide of Manganese and Water, in *Cupreous Manganese* (*Kupfermanganerz*)  $\left[ \begin{array}{c} \text{Cu O} \\ 3 (\text{Mn}^2 \text{O}^3) \\ 9 (\text{H O}) \end{array} \right] + 3 (2 \text{Mn}^2, 2\text{O}^3 + 3 (\text{H O}))$ ; and as an Oxide, with a little Peroxide of Manganese, Iron, and Water, in *Native Oxide of Copper* [ $\text{Cu O}$ ];

(e) As an Oxide, in combination with Chloride of Copper, in *Atacamite* [ $\text{Cu Cl} + 3 (\text{Cu O}) + 6 \text{aq}$ ];

(f) In an Oxidized state, with Acids, either alone or with other Metalline Salts, or Earths and Water, thus:—

(1) With Carbonic Acid and Water, in *Blue Carbonate of Copper* (*Azurite*) [ $2 (\text{Cu O}, \text{CO}^2) + \text{Cu O}, \text{H O}$ ]; and in *Malachite* [ $2 (\text{Cu O}) \text{CO}^2 + \text{H O}$ ];

(2) With Arsenic Acid and Water, in *Condurrite* [ $6 (\text{Cu O}) \text{AsO}^5 + 4 \text{aq}$ ]; in *Fuchroite* [ $4 (\text{Cu O}) \text{AsO}^5 + 7 \text{aq}$ ]; in *Erinite* [ $5 (\text{Cu O}) \text{AsO}^5 + 2 \text{aq}$ ]; in *Prismatic Copper Mica* [ $8 (\text{Cu O}) \text{AsO}^5 + 12 \text{aq}$ ]; and also with Alumina, in *Liroconite* (*Octohedral Arseniate*) [ $2 (\text{Al}^2\text{O}^3, 3 \text{H O}) + 3 (4 \text{Cu O}, \text{AsO}^5, 8 \text{H O})$ ];

(3) With Phosphoric Acid and Water, in *Libethenite* [ $4 \text{Cu O}, \text{PO}^5 + 2 \text{aq}$ ]; and in *Phosphoro-chalcite* (*Pseudo-Malachite*) [ $5 \text{Cu O}, \text{PO}^5 + 5 \text{aq}$ ];

\* [The best formula for *Tin-Pyrites* (*Zinnkies*) is the following:— $\left[ \begin{array}{c} 2 \text{Fe S} \\ 2 \text{Zn S} \end{array} \right] \text{Sn}^2\text{S} + 2 (\text{Cu S}) + \text{Sn}^2\text{S}$ ].



(4) With Sulphuric Acid and Water, in *Native Blue Vitriol* [ $\text{Cu O}, \text{S O}^3 + \text{H O} + 4 \text{ aq}$ ];

(5) With Chromic Acid and Chromate of Lead, in *Vauquelinite*;

(6) With Silicic Acid and Water, in *Diopside* [ $3 \text{ Cu O}, 2 \text{ Si O}^3 + 3 \text{ aq}$ ]; with Silicic Acid, Water, and a little Carbonic Acid, in *Chrysocolla*; and with Silicic Acid, and Silicate of Alumina, in *Allophane*.

Further, Copper is not only found *per se*, in scoriæ, &c., produced in the smelting of Cupreous Ores, but very often as a secondary constituent in the Slags from furnaced Argentiferous and Plumbiferous minerals. It is found:—

(a) Metallic, in pure *Gaarkupfer*,\* and in combination with other metals, in *Schwartzkupfer* (second product), in the *Frischtücken*,† in the *Saigerdörnern*,‡ in the *Darrlingen*,§ and in *Cupriferous Workable Lead*;

(b) With Sulphur, *per se*, and also with Sulphurets, *e. g.*, in *Rohstein*, *Bleistein*, *Kupferstein*, and *Kupferleg*,|| and in different *Tuttys*;

(c) In an Oxidized and Vitreous state, in all the Slags which are obtained in the manufacture of Crude Copper, and in the refinement of Copper for Silver.

#### *Examination for Copper.*

This examination is very simple, and so certain, that its presence or absence in any combination, can be determined in a short time.

The combinations of copper with other metals, as met

\* [The product of the third smelting of Cupriferous Ores.]—*Trans.*

† [*Frischstücken* are the Argentiferous Leads, obtained in the refining of Copper Ores for silver.]—*Trans.*

‡ [The *Saigerdörnern* are the residues which remain after extracting the lead from the *Darrlinge*, and which yield a crude copper on being subjected to a process of smelting.]—*Trans.*

§ [*Darrlingen* are eliquated coppers, from which the silver has been sweated out by lead.]—*Trans.*

|| [*Kupferleg* is one of the products produced by smelting the roasted *stein*, which is obtained by smelting roasted *Bleistein* with Quartzose Copper Ores, with *Bleistein* Slags, and Quartz.]—*Trans.*



with in nature, generally contain selenium; if exposed for some time to the oxidating flame on charcoal, and the metallic globule remaining, treated with borax in a continued oxidating flame, the glass generally exhibits the colour produced by oxide of copper. On melting the cooled glass after separation from the excess of metal, on another part of the charcoal in the reducing flame, it assumes, on cooling, a red colour, and is quite opaque. The latter does not, however, always succeed, because, if exposed too long to the reducing flame, the copper separates, and the glass appears colourless. It succeeds better if a small piece of metallic tin is added, and only treated for a few moments in the reducing flame. A part of the tin oxidizes at the expense of the oxide of copper, and dissolves colourless in the glass, while the copper is reduced to a sub-oxide, which colours the glass red on cooling, and is opaque. The red colour appears fainter in proportion to the other metallic oxides dissolved with it.

Copper is detected in this manner, in most furnace products, consisting only of metallic combinations. Such a combination is treated with borax or microcosmic salt, on charcoal in the reducing flame; the glass pearl, while yet liquid, is removed from the metallic globule by the aid of the forceps, the colour, if any, from oxide of copper observed, and the assay then placed upon another part of the charcoal, and treated as above with tin. If only a trace of copper be present, which can only occur with *Workable Lead*, a red-coloured pearl is not always obtained, and if the metallic compound contain at the same time antimony, the glass on cooling becomes opaque, and is coloured grey or black. In such a case, the metallic mixture must be previously melted *per se* on charcoal, in the oxidating flame, until all the antimony is volatilized, and the greater part of the lead is then dissolved in boracic acid, as will be given with the quantitative examination for Copper, under the process for refining; the globule remaining, is treated first with microcosmic salt in the oxidating flame, and the glass bead is then fused with tin in the reducing flame. If a trace of copper is present, the glass will be coloured distinctly red on cooling, and will be opaque, entirely, or in separate portions.



Should the metallic combination contain much nickel, cobalt, iron, and arsenic, the greater part of the cobalt and iron can be removed by treatment with borax in the reducing flame, on charcoal, and recognized by the colour of the glass, as given with Iron. Lead is then added to that which has been obtained in a molten state in the reducing flame, by which means the greater part of the arsenic volatilizes, and by treatment with boracic acid, the lead, with any remaining cobalt and iron, is dissolved. The remaining cupriferous nickel globule, and probably a part of the arsenic, is treated with microcosmic salt in the oxidating flame, and the observation made as regards the colour which the glass assumes. If copper be present, it appears while hot, dark green, on cooling, clearer, and when perfectly cold, bright green. The latter consists of the light brown of the oxide of nickel, and the blue of the oxide of copper.

The combinations of copper with sulphur and other metallic sulphurets, are either heated, alternately, in the oxidating and reducing flames, on charcoal, till the sulphur is perfectly dissipated; or treated with soda on charcoal, in the reducing flame, by which means the copper is obtained in a metallic state; or dissolved in borax or microcosmic salt, and the glass treated with tin on charcoal, when the presence of copper is recognized by its red tinge. If the roasted substance contains, besides copper, other easily-reducible metallic oxides, no pure copper will be obtained by the reduction with soda, but an admixture with other metals; which, if not obtained in a single globule, must be sifted with water in a mortar, to remove the recrementitious particles, and lead added, if not already present; it is then refined, by treating with boracic acid on charcoal. If convenient to dispense with the refining, the reduced metallic compound may be examined for copper with borax or microcosmic salt, as already mentioned with the metallic combinations. If the roasted assay contains, besides copper, oxide of iron only, a mixture of both metals is not obtained by the reducing process, but particular reguli of copper and iron, which, after purification, are readily distinguished by the lens or magnet. But if it contain oxide



of tin, which occurs with tin pyrites, a white friable metallic mixture is obtained by the reduction; which, if melted for some time with a microcosmic-salt pearl, in the oxidating flame, produces an opaque red glass. If not perfectly roasted, a mixture of sulphuret of copper, and other metallic sulphurets, is very readily obtained.

On treating the roasted substance with borax or microcosmic salt, even if it contains other metallic oxides (with the exception of oxide of bismuth and oxide of antimony), in the oxidating flame, and then in the reducing flame, after the addition of tin; the reaction of copper is always detected, if an appreciable quantity be present. But if it contain at the same time much bismuth or antimony, the glass assumes a dark grey colour on cooling, which destroys the red tinge produced by sub-oxide of copper. If the bismuth or antimony present be small, the glass will often be coloured brownish-grey. If a black or grey pearl is obtained, the roasted substance must be mixed with soda, borax, and proof lead, and this compound melted in the reducing flame. The metallic globule obtained by this means, must first be treated *per se* on charcoal, and then with boracic acid, until either a pure globule of copper is obtained, or till the whole is dissolved, when the copper communicates a blue-green or red colour to the boracic acid: or the globule of copper, freed by boracic acid from the greater part of lead and bismuth, is treated with microcosmic salt and tin, as already mentioned. The latter procedure, which volatilizes the antimony, and separates the bismuth perfectly by boracic acid, is the most certain.

If the copper in a substance is so insignificant, that a portion, dissolved in borax or microcosmic salt, produces with tin no cupreous reaction, a greater quantity (about 100 milligrammes) must be roasted, as with a quantitative copper examination; the roasted substance is then to be mixed with equal parts of soda and borax, and if it contains no easily-reducible metal, 30 to 50 milligrammes proof lead added, and reduced as with a quantitative examination of the copper. The metallic mixture obtained as a globule by this means, in



which all the copper is contained, can be examined further for copper, after treating with boracic acid, and then with microcosmic salt and tin. How to proceed further has already been mentioned. The protoxide and sub-oxide of copper, as met with in nature, and also the latter in combination with other metallic oxides, behave *per se* towards borax and microcosmic salt, with tin in the reducing flame, perfectly similar with oxide of copper, and thus the presence of copper can be readily recognized in the minerals. The copper can be separated also from these minerals by a simple reducing process, with soda and a small portion of borax. The native copper salts, which include silicate of copper, and a compound of this, with silicate of alumina (Allophane), impart to borax and microcosmic salt the colour of protoxide of copper, particularly if treated with tin, on charcoal in the reducing flame. If it be necessary to separate the copper in a metallic state, the sulphate of copper must be previously roasted slightly, in the oxidating and reducing flames alternately; this is reduced like the other salts, with soda and borax on charcoal; the copper now generally coheres, forming a globule, while the difficultly-reducible metallic oxides are dissolved by the borax.

In manipulation with sulphate or arseniate of oxide of copper, nickel, cobalt, or iron, the sulphur volatilizes by roasting, but part of the arsenic remains with the oxide of nickel, as arseniate. If the roasted substance be reduced with soda and borax on charcoal, copper, nickel, or arsenic, produce a fluid metallic globule, and oxide of cobalt and iron are dissolved by the borax. If the reduced metallic bead contains copper, it must impart a green tinge of both nickel and copper to borax or microcosmic salt in the oxidating flame, which becomes somewhat paler on cooling.

The presence of copper can also be determined, if treated with tin, by such a glass losing its transparency on cooling, and assuming a red tinge. If the borax glass, without the addition of tin, be treated in the reducing flame till all the nickel and copper is separated in a metallic state, and if then the glass be observed to appear blue, the roasting will have



occurred imperfectly, and by the reduction, some arseniuret of cobalt has accompanied the metallic button.

It is difficult, by borax or microcosmic salt, to determine the copper in slags as protoxide or sub-oxide, with the exception of that contained in *gaarkupfer*, on account of the small quantity generally present; and, moreover, the other ingredients, which are chiefly silicates of different earths and difficultly-reducible metallic oxides, destroy the reaction of oxide of copper. For this reason, instead of employing the reduction process, the slags must be treated with soda on charcoal. If, even by this method also, copper should not be detected, a greater quantity (about 100 milligrammes) must be reduced with equal parts of soda, the half of borax, and 30 to 50 milligrammes proof lead, and the lead, united to a globule, treated with boracic acid till all is dissolved, or the copper is concentrated. If the slag contains a trace of copper, this becomes reduced and combines with the lead, and in the first case, has coloured the boracic acid red, green, or blue. If the copper present is very minute, the tinge is seen on those parts only where the latter part of the lead containing copper was dissolved. If the slag contain one per cent. of copper, and the glass be treated in the reducing flame, the lead only is dissolved, and the copper remains in a melted state, with its peculiar greenish-blue colour. If the copper be exposed some time to the oxidating flame, it becomes oxidized, and the whole glass is coloured red, by the sub-oxide formed. In the second case, the metallic globule, melted with boracic acid, is treated with microcosmic salt and tin, as above.

A small quantity of copper contained in a substance, can often be detected, if not in combination with sulphuric acid, by one or two drops of hydrochloric acid. It is only necessary to moisten the substance with this acid, and heat it in the forceps, in the apex of the blue flame, when, by this means, the outer flame is coloured greenish blue, and often reddish blue, by the chloride of copper formed. The colouring is more beautiful and stronger, the richer the substance is with oxide of copper. Silicates, *e. g.*, slags, must be pulverized as finely as possible in a mortar, this powder moistened with a



drop of hydrochloric acid in a porcelain basin, dried over the flame of a lamp, and the dried powder kneaded into a granular mass with a drop of water. This grit is placed in the ring of a platinum wire, and melted in the apex of the blue flame. If the silicate contains copper, a blue colouring ensues in the outer flame. If the mineral containing copper be heated, *per se*, in the apex of the blue flame (with the exception of Atachamite), the outer flame is coloured beautifully green. If the mineral, at the same time, contains much lead, a blue flame, with greenish streaks, results.

§ 12. SILVER—[Ag].—*Presence in the Mineral Kingdom, and in the Products of Smelting Furnaces.*

Silver occurs in nature:—

(a) Metallic, both *per se*, and in combination with other metals, namely, as *Native Silver* [Ag], which is often contaminated with minute portions of Antimony, Arsenic, and Iron; in *Native Gold*, which contains more or less Silver; in *Native Amalgam* [Ag, 2 Hg] and [Ag, 3 Hg]; with Antimony, in *Antimonial Silver* [2 Ag, Sb] and [3 Ag, Sb]; with Gold and Tellurium, in *Graphic Tellurium* [Ag, Te + 3 (Au, 3 Te)]; with Iron, Arsenic, and a little Antimony, in *Arsenical Silver*\*; with Gold, Tellurium, and Lead, in *Yellow Tellurium* (*Weiss-Silvanerz*); and with Selenium and Copper, in *Eukairite* (*Seleniuret of Silver and Copper*);

(b) With Sulphur, both *per se*, and in combination with other sulphurets; thus, *per se* in *Sulphuret of Silver* (*Silver Glance*) [Ag S]; with Antimony and a little Copper, in *Brittle Sulphuret of Silver* (*Brittle Silver-Glance*) [6 Ag S + 2 Sb 3 S]; with Arsenic, Copper, and Antimony, in *Eugenglance* or *Polybasite*  $\left\{ \begin{smallmatrix} 2 \text{ Sb, } 3 \text{ S} \\ 2 \text{ As, } 3 \text{ S} \end{smallmatrix} \right\} + 9 \left\{ \begin{smallmatrix} \text{Ag S} \\ 2 \text{ Cu S} \end{smallmatrix} \right\}$ ; with Arsenic, and a very small proportion of Antimony, in *Sulphuret of Silver and Arsenic*, (the light red variety of *Red* or *Ruby Silver*,—it is the *Lichtes Rothgiltigerz* or *Arsensilberblende* of the Germans) [3 (Ag S), 2 As 3 S]; with Antimony, in *Sulphuret of Silver and Antimony*, (the dark red variety of

\* [This is probably a mixture, and not a peculiar species.]—*Trans.*



*Ruby Silver*—*Antimonsilberblende* of the Germans) [ $3(\text{Ag S})$   $2\text{Sb } 3\text{S}$ ]; with Antimony, a little Copper, and Iron, in *Myargyrite* (*Hemi-Prismatic Ruby Blende*) [ $\text{Ag S}, 2\text{Sb } 3\text{S}$ ]; with Copper, and a little Iron, in *Sulphuret of Silver and Copper* (*Argentiferous Copper Glance*); with Antimony, Copper, a little Iron, and Zinc, in *Weissgiltigerz* and *Graugiltigerz*; with Bismuth, Lead, together with a little Iron and Copper, in *Bismuthic Silver*; with Antimony, Arsenic, Copper, Iron, and Zinc, in *Grey Copper*; and as a minute constituent in most Lead and Copper ores, to which belong *Seleniuret of Lead*, *Copper Glance*, *Copper Pyrites*, &c.

(c) In combination with Chlorine, in *Muriate of Silver* (*Hornsilver*) [ $\text{Ag Cl}$ ]; and,

(d) In combination with Iodine, in *Iodic Silver* [ $\text{Ag } 2\text{I}$ ].

Silver occurs in the products of smelting-furnaces:—

(1) Metallic, both *per se*, and in combination with other metals; namely, *per se*, as *Brandsilber*\* and *Amalgamated Silver*†; with a little Lead, and sometimes minute portions of Copper, in *Blicksilber*‡; with Lead, and also with Copper and traces of Antimony, Arsenic, Iron, and Sulphur, in the *Workable Lead*; as a secondary constituent in the *Black Copper* obtained from Copper and Lead ores; and also, in extremely small quantities, in *Abstrich Lead*. §

(2) Combined with Sulphur and other metals, in which, however, it forms only a very humble constituent; namely, in the *Bleistein* obtained from Sulphuret of Lead and Sulphuret of Iron; in the *Kupferstein*, from Sulphuret of Copper, Sulphuret of Lead, and Sulphuret of Iron; in the *Rohstein*, from Sulphuret of Iron, and, sometimes, a little Sulphuret of Lead and Sulphuret of Copper; and in the

\* [*Brandsilber* is *blicksilber* refined by cupellation.]—*Trans.*

† [The amalgam of silver and mercury obtained in extracting silver from its ores by the process of amalgamation; it generally contains copper, antimony, &c.]—*Trans.*

‡ [*Blicksilber* is the crude silver obtained in the refinement of lead for silver, after nearly the whole of the lead has been converted into Litharge.]—*Trans.*

§ [*Abstrich Lead* is the *Workable Lead* from which the *Abstrich* has been raked off.]—*Trans.*



compound *Flue-rakings* from the smelting of different sulphurets which yield Silver; as also, in a fine mechanically-divided state, in the slags obtained by the smelting of Silver Ores, or of the argentiferous products of their reduction.

(3) In an oxidized state, but only in exceedingly small quantities, as, in *Litharge*, in the *Abstrich*, and in the *Cupel Grounds* from which the *Workable Lead* has been separated.

*Examination for Silver.*

Some of the minerals, alloys, and furnace products, previously mentioned, are so constituted as to be readily recognizable by their exterior appearance, for silver. To these belong Native Silver, Native Gold of a very light colour, *Brandsilber*, *Amalgamated Silver*, and *Blicksilber*. Other argentiferous minerals, when combined with volatile ingredients, give pure silver beads when exposed to a strong oxidating flame, and the charcoal becomes coated with a red sublimate. These are Antimonial Silver, Arsenical Silver, natural and artificial amalgams. When the latter are heated in a glass matrass, mercury volatilizes and deposits in orbicles, which may readily be made to cohere by tapping the vessel. The residuum, which is nearly freed from the mercury by this process, gives, when smelted on charcoal, a beautiful white globule. If Graphic or Yellow Tellurium be heated on charcoal, an auriferous yellow globule remains, which is composed of gold and silver: by treating this alloy with nitro-muriatic acid, the gold dissolves and the silver deposits as a chloride. Native gold, containing silver, may also be treated in this manner, when separation is requisite. Those minerals which contain, besides volatile metals, copper, yield a cupreous silver bead when assayed upon charcoal in the oxidating flame. To obtain the silver from these in a pure state, they must be mixed with lead and cupelled. Eukairite belongs to this class. Silver is detected in *Workable Lead*, *Abstrich-Blei*, and in impure *Black Copper*, by cupelling the first two, *per se*, and treating the last with ten times its volume of proof lead on charcoal. If it be requisite to examine at once for silver in minerals and furnace products which consist of metallic



sulphurets, or only contain such, the quantitative silver examination with proof lead and borax is the most satisfactory method for this object. The necessary information upon this treatment will be given under the above-mentioned examination. Native Chloride of Silver (Hornsilver) fuses on charcoal in the oxidating flame, to a brown, grey, or black bead, which, if pure, gives metallic silver in the reducing flame. This decomposition is immediately effected with soda on charcoal. Furnace products which contain only a very small quantity of silver,—for instance, *Litharge*, *Cupel Grounds*, and *Abstrich*, must be assayed according to the process given under the quantitative examination of such substances.

§ 13. *MERCURY*—[Hg].—*Presence in the Mineral Kingdom, and in the products of Smelting Furnaces.*

Mercury occurs in nature:—

(a) Metallic, *per se*, in *Native Mercury* [Hg]; and combined with Silver, in *Native Amalgam*;

(b) In combination with Sulphur, as *Cinnabar*, or *Sulphuret of Mercury*, and in *Lebererz*, or *Hepatic Cinnabar* [Hg, S]; the latter is, however, contaminated with Carbon, Silicic Acid, Oxide of Iron, and other substances;

(c) In combination with Chlorine, as *Muriate of Mercury* or *Horn Quicksilver* [Hg, Cl]; and,

(d) In combination with Iodine, as *Iodic Mercury*.

Mercury also forms a constituent of many products, residues, scorïæ, &c., of the *amalgamation process* for extracting Silver from its ores. To these also belong the products of the amalgamation of Gold and Silver Ores, namely, *Gold Amalgam*, *Silver Amalgam*, and the unwashed residue in which finely divided *Silver Amalgam* is generally present. If the Ores to be amalgamated contain Copper and Lead, these Metals will be found in the residues, when the process is completed.

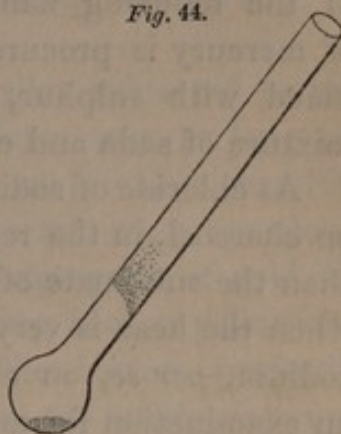
*Examination for Mercury.*

Native Mercury possesses all the properties of the pure metal, and need not therefore be more particularly examined,



unless it be required to detect traces of other metals present.

The combinations of mercury with gold and silver, to which belong both native and artificial amalgams, and also the impure residues obtained by the washing of silver, copper, and lead amalgams, should be ignited in a flask, or bulb tube (see *Fig. 44.*) over the spirit-lamp. The mercury is separated, volatilized, and deposited in the form of small metallic globules in the colder part of the flask or tube, which cannot be confounded with any other metal. In the examination of the compounds



of mercury with sulphur, chlorine, or iodine, to which Native Cinnabar, Hepatic Cinnabar, Horn Quicksilver, and Iodic Silver belong, a portion of the substance should be triturated in an agate mortar, with an equal bulk of soda, the mixture introduced into the small glass vessel, and heated to redness over the spirit-lamp. The sulphur, or chlorine, combines at this temperature with the radical of the soda, forming sulphuret or chloride of sodium, while metallic mercury becomes free, ascends as vapour, and condenses on the upper part of the flask in a greyish sublimate, which coheres into small metallic globules on gently tapping the tube. If the quantity of this metal present be so minute that no metallic sublimate of mercury is obtained, the experiment should be repeated in the same manner, introducing at the same time the end of an iron wire, covered with a piece of gold leaf, into the flask, and holding it a little over the surface of the mixture, when the gold becomes perfectly white, or in a great measure so, even when the quantity of mercury present is extremely small.

According to Wackenroder (*Tabellen zur Analyse der unorganischen Verbindungen*, published 1842), the haloid salts of mercury give a sublimate on charcoal. The oxygen salts also give, with chloride of sodium (sulphuret of mercury with a mixture of soda and chloride of sodium also), a heavy or light white vapour and sublimate of sub-chloride of mercury. If a substance is to be examined for mercury, in



which the latter is not combined with sulphur, and which, *per se*, gives no sublimate, it should be reduced to a fine powder, and fused with dry chloride of sodium on charcoal, in the reducing flame; a white sublimate of sub-chloride of mercury is procured. If the mercury, however, be combined with sulphur, the substance must be fused with a mixture of soda and chloride of sodium.

As chloride of sodium also yields a white sublimate, *per se*, on charcoal, in the reducing flame, but which appears later than the sublimate of sub-chloride of mercury, and then only when the heat is very strong,—the behaviour of chloride of sodium, *per se*, on charcoal, should be first studied, before an examination for mercury in this way be undertaken.

§ 14. PLATINUM—[Pt]. PALLADIUM—[Pd]. RHODIUM—[R]. IRIDIUM—[Ir]. OSMIUM—[Os].—*Presence in the Mineral Kingdom.*

These Metals generally occur united together; sometimes, also, with traces of Iron, Copper, and Lead, in *Native Platinum*, in which Platinum is, however, the principal constituent.

Palladium is likewise found native, combined with a little Platinum and Iridium, along with the *Native Platinum*, in Brazil.

Rhodium occurs only as an extremely small constituent in *Native Platinum*.

Iridium occurs native, combined with a little Osmium, along with *Native Gold*, and *Native Platinum*, in Russia. It also occurs, with a larger proportion of Osmium, in peculiar grains (*Osmium-Iridium*—[Ir Os]), along with *Native Platinum*, in South America. The quantity of Osmium in *Osmium-Iridium*, varies greatly.

Osmium has been found, as yet, only in the last-named compound, and in *Native Platinum*.

*Behaviour of the preceding Native Metals before the Blowpipe.*

These metals cannot be so separated from one another by the blowpipe, that each metal may be recognized when treated with borax or microcosmic salt, on a platinum wire or on



charcoal, as they neither oxidize nor dissolve: beads, more or less coloured, are in this case obtained; but the colour proceeds from the mixed oxidizable metals, namely, from copper, iron, &c., which may be readily detected in this way, in such combinations.

If they be fused with lead, and the alloy subjected to refinement in a cupel, (which operation lasts only as long as it affords the difficultly-fusible metals,) an infusible metallic compound is at length obtained, which contains, proportionably, much lead; but if a sufficiently-large grain of gold be added to it, and then refined in a strong heat, a yellowish-white or even a platinum-grey metallic button, perfectly free from lead, will be obtained, in case too much platinum or iridium is not present in the alloy. If it is not obtained of a *fine* quality from the cupel, it may be, very readily, with boracic acid, on charcoal, in the oxidating flame.

The alloy of gold, platinum, iridium, rhodium, palladium, &c., thus obtained, can only be analyzed in a moist way when it is required to recover the gold. For this purpose it should be dissolved in aqua-regia, the solution partly evaporated, diluted with weak alcohol, and the platinum and iridium thrown down from this solution by chloride of potassium or chloride of ammonium, and the gold, by a fresh-prepared solution of proto-sulphate of iron. The latter is obtained in the metallic state, and requires only to be filtered,edulcorated, and fused with a little borax, on charcoal.

Osmium-Iridium is the only compound which can easily be decomposed, and in which osmium may be recognized. When it is strongly ignited in a glass flask, with saltpetre, oxide of osmium is formed, and may be recognized by its fœtid smell, which is similar to chloride of sulphur.

§ 15. GOLD—[Au].—*Presence in the Mineral Kingdom, and in the Products of Smelting Furnaces.*

Gold always occurs, in nature, in the metallic state, thus:

(a) Mixed with other metals, as with (more or less) Silver, in *Native Gold*; with Tellurium and Silver, in *Graphic Tellurium*; with Tellurium, Lead, and Silver, in *Yellow Tellurium*;



with a large quantity of Tellurium and a little Iron, in *Native Tellurium*; with Lead, Tellurium, a little Copper, Silver, and Sulphuret of Lead, in *Black Tellurium*; in a combination of Seleniuret of Molybdenum, with a little Silver, in *Noble Molybden Glance*.

(b) In metallic Sulphurets; namely, in many Iron and Copper Pyrites, but in small quantities, however. The quantity of Gold in these Pyrites is very different. The Iron Pyrites, so widely diffused in Saxony, which lie in contact with the gangues, and which, at the same time, contain Sulphuret of Antimony and finely-disseminated Ruby Silver, or other Silver Ore, always yield more Gold than those occurring in the same situation, in which no Antimony can be detected: the former contains, in a *cwt.*, owing to the admixture of Silver Ores, from five to ten ounces of Silver, eight ounces of which yield from 0.5 to 0.8 of a grain of gold; and the latter contain, in the *cwt.*, from 0.125 to 0.5 of an ounce of Silver, eight ounces of which yield only from 0.15 to 0.18 of a grain of Gold.

Gold Amalgam is the only product of smelting furnaces, extensively occurring, in which Gold forms the principal ingredient.

However, as auriferous Silver Ores are often smelted, Gold is also found, as a secondary constituent, in many products obtained in the reduction of Silver; more particularly in the following:—*Brandsilber* (if the Gold has not been already separated), *Blicksilber*, *Amalgamated Silver*, *Workable Lead*, *Black Copper*, *Bleistein*, *Kupferstein*, and *Rohstein*.

#### *Examination for Gold.*

Native Gold is easily recognized by its peculiar colour. The remaining auriferous minerals, (Pyrites only excepted,) should be treated, when the volatile metals are also to be detected, on charcoal in the reducing flame, until an unchangeable metallic button is obtained. With some minerals, *e. g.*, Graphic Tellurium, Yellow Tellurium, and Black Tellurium, a button remains, which, on cooling, possesses the colour of gold, and contains the silver present in the mineral.



If it should happen that a pure metallic button is not obtained, a little proof lead and borax must be added to the remaining mass, and the whole treated for some time in the reducing flame. The easily-reducible metals combine with the proof lead, and, after cooling, can be separated from the gold and silver by cupellation on bone ashes. If the button obtained by cupellation, does not possess the colour of gold, but appears white, the quantity of gold is less than that of silver, in which case, the button should be placed in a porcelain capsule, a few drops of nitric acid poured on it, and the capsule heated over the lamp.

If the button does not contain more than a fourth part of its weight of gold, it becomes completely black, and then decomposes, the silver being dissolved, while the gold remains in black flakes. When the silver button contains more than the fourth of its weight of gold, it is blackened, but the silver is not dissolved. It is neither blackened nor dissolved, if the proportion of gold to silver is nearly equal: in this case, the button must be melted with twice its bulk of pure silver, on charcoal, and again treated with nitric acid, by which the mass becomes black, and dissolves; the pure gold being left behind.

Gold Amalgam is treated, first in a flask similar to the Silver Amalgam, in order to drive off the greater part of the mercury, and then on charcoal in the oxidating flame, when a button of pure gold is obtained.

*Brandsilber*, as also *Blicksilber* and *Amalgamated Silver*, are treated, after being refined with a slight addition of proof lead, on a cupel with nitric acid, and examined whether the silver becomes black, and whether black gold flakes, or black particles, remain after the solution of the silver.

*Workable Lead* is refined *per se*, and *Black Copper*, with an addition of proof lead on the cupel, and the resulting metallic button treated with nitric acid. If the silver becomes black, or if black particles remain after the silver is dissolved, the metals contain gold.

Iron and Copper Pyrites, *Bleistein*, *Kupferstein*, and *Rohstein*, must be first examined for silver, in the same



manner as in the quantitative examination. The resulting button of silver is to be placed in a porcelain capsule, containing some hot nitric acid, and *quickly* examined with a lens, whether it dissolves with a black or white colour. In the former case, the substance contains gold; in the latter, it is absent. When a button is not obtained in the examination of Pyrites, it does not follow that gold is not present, as the quantity is often so minute that it cannot be recognized on the cupel with the lens, even from 100 milligrammes of ore. Hence, two or more fragments should be subjected to examination, the resulting *Workable Lead* concentrated by cupellation, mixed with a little pure silver, and again refined, by which means the gold is combined with the silver, and may be recognized as above, by the black colour of the button, when treated with hot nitric acid.

§ 16. *TIN*—[Sn].—*Presence in the Mineral Kingdom, and in the products of smelting furnaces.*

Tin occurs in nature in the following minerals:—

In combination with Sulphur, and Sulphurets of Copper and Iron, in *Tin Pyrites*; as an oxide, in *Tin Stone* (*Pyramidal Tin Ore*) [ $\text{Sn O}^2$ ] which contains traces of Iron, Manganese, Tantallic Acid, and Silica; and as a constituent in most Tantalites, and numerous other minerals, containing Titanium and Uranium.

It very rarely occurs in furnace products, unless Tin Ores have been smelted, and then portions of it will be found in the slags. When Tin Pyrites accompanies an ore of Copper, and cannot be separated in a pure state by reduction, a quantity of Tin will necessarily be detected in the first products of the copper-smelting, particularly in the *Rohstein*.

*Examination for Tin.*

Tin is readily recognized in Tin Pyrites, by exposing a small piece of the mineral to the oxidating flame, on charcoal. The assay at first exhales a sulphurous acid smell, afterwards becomes snow-white on the exterior, and a white coating is perceivable on the support surrounding the speci-



men; this sublimate is so profuse, that the charcoal is not seen in any part between it and the metallic bead. This deposit is not expelled in either flame; in other respects, its comportment is similar to the oxide of tin.

The tin can be separated from this mineral, in the metallic state, by another method; namely,—roast alternately in the oxidating and reducing flame, and then pulverize the mass in an agate mortar, with double its quantity of a mixture consisting of 100 parts soda, 50 parts borax, and 30 parts silica, and heat the whole on charcoal in the reducing flame, until the tin and copper are reduced to a globule. In this treatment the whole of the copper becomes reduced, but only a portion of the tin, the rest remaining dissolved in the glass with protoxide of iron. The cupriforous tin bead, which is friable if too small a quantity of tin is not present, must be separated, and what remains treated with soda in a strong reducing flame, by which means the rest of the tin becomes reduced, and is obtained in particles, by pulverization and sifting with water. The cupreous globule, when heated upon charcoal with microcosmic salt in the oxidating flame, fuses, and the resulting glass, when cold, has a reddish colour, owing to the presence of sub-oxide of copper.

With the stanniferous *Rohstein*, the treatment is the same as above. Tin stone behaves like the oxide of tin, with this difference, that it imparts the colour of iron to borax and microcosmic salt, and often affords the manganese reaction with soda on the platinum wire.

The best method for the detection of tin in Tantalites and Tin Slags is by reduction with soda; but in such a case it is necessary to add a small portion of borax, to dissolve the tantalic combinations, and prevent the reduction of the iron. After the completion of such a process, the tin is obtained by pulverization and sifting. To be convinced that the metallic particles obtained are tin, dissolve protoxide of copper in microcosmic salt, add some of them to the flux, and then heat the whole upon charcoal in the reducing flame. If tin is present, the glass will be coloured reddish on cooling.



§ 17. *ANTIMONY*—[Sb].—*Presence in the Mineral Kingdom, and in the products of smelting furnaces.*

Antimony is found in nature :—

(a) Metallic, combined with other metals ; namely, with a little Silver and Iron, in *Native Antimony* [Sb] ; with Silver, in *Antimonial Silver* ; and with Arsenic, in *Arsenical Antimony* ;

(b) With Sulphur, and other Sulphuretted metals, *e. g.* ; *per se*, in *Grey Antimony*, which generally contains Lead, Copper, Arsenic, and Iron ; with Iron, in *Berthierite* ; with Nickel and Arsenic, in *Nickeliferous Grey Antimony* ; with Lead, and traces of Iron, Copper, Bismuth, and Zinc, in *Jamesonite* ; with Lead, Copper, and Iron, in *Bournonite* ; with Lead, and a trace of Copper, in *Zinkenite* ; with Lead, in *Antimonial Lead-Glance* ; with Silver and Copper, in *Melan-Glance* ; with Silver, Copper, and Iron, in *Miargyrite* ; with Silver, in *Dark Ruby Silver (Rhombohedral Ruby Blende)* ; sometimes with Silver and Arsenic, in *Light Ruby Silver* ; contaminated with Arsenic, Silver, and Iron, in *Arsenical Silver* ; also, more or less, in the following argentiferous minerals : namely, with Silver, Copper, Iron, and Zinc, in *Weissgiltigerz* and *Graugiltigerz* ; with Copper and Silver, in *Antimonial Grey Copper* ; with Copper, Arsenic, Silver, Iron, and Zinc, in *Grey Copper (Tetrahedral Copper Glance)* ; and, in a very minute quantity, with Copper, Iron, Silver, and Arsenic, in *Kupferblende* ;

(c) As an oxide, in *White Antimony* [Sb O<sup>3</sup>] ; which is sometimes contaminated with Peroxide of Iron ; and, with Sulphuret of Antimony, in the *Red Antimonial Ore* [Sb O<sup>3</sup> + 2 (Sb, 3S)].

(d) As Antimonious Acid, in *Antimonial Ochre* [Sb O<sup>4</sup>]. Antimony forms a small ingredient in many Argentiferous and Plumbiferous furnace products, when the smelted or amalgamated ores are not free from Antimonial Silver or Lead Ores. This class includes *Workable Lead*, *Amalgamated Metals*, and the *Abstrichblei*, which hold it in a metallic state ; further, the *Rohstein*, *Bleistein*, *Kupferstein*, and *Lead Rakings*, in which it is found as a Sulphuret ; and the *Abstrich*, in



which it exists as Antimonious Acid, in combination with oxide of lead.

*Examination for Antimony.*

The examination for antimony is not very difficult, as it can be detected in most combinations in which it occurs in a metallic state, by two methods ; namely :—

(a) Upon charcoal ; when the antimony volatilizes, and the support is coated with a white sublimate ; and,—

(b) In an open glass tube ; when antimonial fumes are evolved, consisting of oxide of antimony and antimonious acid, which condense in the upper part. If the treatment occurs with metallic compounds, or metallic sulphurets, in which the antimony is to be sought for ; as, for example, in the minerals above-mentioned, where the antimony is contained as a metal, and in furnace products, the *Workable Lead*, the raw amalgamated metals, the *Abstrichblei*, the *Roh-, Blei-,* and *Kupfer-stein*, and the *Tutty*,—the examination according to the first method must be performed as follows : A fragment of *Workable Lead*, raw amalgamated metal, or *Abstrichblei*, is taken (the other products and minerals are best employed in a powdered state) and placed in a smooth cavity made in the charcoal, and then submitted to a *weak* reducing flame. By holding the support in an horizontal position, if any arsenic be present, it will volatilize, if not in combination with nickel, and coat the charcoal with a white or grey sublimate, at a great distance from the assay. Should no arsenic be present, there will be produced a slight deposit of oxide of antimony. When no more arsenical vapour is evolved, the flame is to be directed upon the sublimate, without igniting the charcoal ; by this procedure the whole of the arsenic is expelled, and a clear surface remains, upon which the antimonial deposit is recognizable.\* This deposit is white, when no lead is present, and can be driven from place to

\* If an operator treats a powdered assay in the reducing flame, it may cohere into a bead ; but this will only occur when the substance is very fusible. When the latter is the case, the oxidating flame must be brought into action.—(*Author.*)



place, either by the oxidating or the reducing flame; if the reducing flame is employed, it assumes a slight dark-blue appearance, which is characteristic of the presence of antimony. If the substance contains lead, a yellow sublimate of the oxide of this metal is also obtained, which rests at a greater distance from the assay than the antimonial one, and wherein thin layers appear bluish, thus resembling a sublimate of antimony in the same disseminated state. When much antimony is present, the lead does not impede the determination, but if the contrary be the case, an operator must not blow for too long a time upon the sample; if this precaution be observed, only a white antimonial sublimate results, as the lead is not expelled unless by a long uninterrupted blast. The deposition of lead may be entirely prevented by the addition of some vitrified boracic acid to the substance under examination; when this mixture is acted on in the reducing flame, the oxide of lead formed is absorbed by the boracic acid, while the greater portion of the antimony volatilizes, and coats the charcoal with its oxide. The antimonial lead globule must not be kept in the centre of the molten mass, but only in contact with it, because the antimony, in the former case, is vaporized with difficulty. If the substance contain zinc, a sublimate of this metal is also obtained, which is readily distinguished from that of antimony, by not being volatilized in the oxidating flame.

Minerals and furnace products containing antimony, as an oxide or acid, can also be partly investigated in the above manner, but a distinct sublimate is not always procured; particularly if the oxide of antimony, or antimonious acid, be disseminated or combined with other bodies. When this occurs, the assay should be mixed with soda, and treated on charcoal, in the reducing flame, when the antimony volatilizes and coats the support with an oxide, which is very distinct, even when a minute quantity of antimony is contained in the sample. If the antimonious acid is in combination with much oxide of lead, which is the case with the *Abstrich*, an operator must not blow for any length of time, so as to prevent the volatilization of much lead. Combinations of oxides of tin



and antimony, or antimonious acid, which do not occur in nature, must be treated with a mixture of soda and borax, on charcoal, in the reducing flame. The oxides are reduced and separated in small limpid metallic globules, which must be sifted from the recrementitious particles, and then heated on charcoal, with three times their volume of proof lead, and a small portion of vitrified boracic acid. If the assay be treated in the reducing flame only, antimony volatilizes, and coats the charcoal with a sublimate; the tin oxidizes, and covers the metallic button with a deposit, which dissolves with a part of the lead in boracic acid. The antimonial sublimate is freed from oxide of lead by this procedure, and can therefore be readily recognized. The oxide of tin which deposits on the molten button, resembles the formation of oxide of nickel upon a nickeliferous lead globule; but as tin and nickel behave perfectly different towards borax, it suffices when the presence of the former is determined.

The second method for the detection of antimony when in a metallic state, in its combinations, is the following:—The substance is heated in a glass tube, whereby the antimony oxidizes and sublimes, forming a white fume, whose behaviour varies, according to the different metals with which the antimony is in combination. If the metals are readily oxidizable, antimonious acid fumes will be expelled, which are very stable, and not acted upon by heat.

The fume which escapes from a silver or copper compound, partly passes off and partly deposits on the upper part of the tube. That which escapes, possesses an acid smell; but, if sulphur happens to be present, the odour of sulphurous acid will predominate.

If the substance contains lead, an exhalation also ensues, which is very dense. The portion which volatilizes may be regarded as pure oxide of antimony; and the non-volatile portion, as antimonite of lead.

That this method is not applicable to the examination of readily-fusible metallic alloys, is easily understood; for, on melting, they flow out from the tube, which is held obliquely.



When the substance is in combination with sulphur, it escapes as sulphurous acid, which is recognized by its characteristic odour; and, in this case, if only a small portion of antimony be contained in the assay, antimonious acid will often be generated, and no oxide of antimony. If the substance contains much arsenic, the antimonial vapour will be very profuse; but, as the crystals of oxide of antimony and arsenious acid combine, they cannot always be distinguished from their appearances. When this ensues, it is preferable to examine on charcoal, as previously described.

Combinations of oxides of antimony, or antimonious acid, and other bodies, cannot always be examined with certainty in an open glass tube: for antimony, it is always better to employ the procedure with soda, on charcoal.

§ 18. *MOLYBDENUM*—[Mo].—*Presence in the Mineral Kingdom.*

It is found—

(a) Metallic, combined with Sulphur, in *Rhombohedral Molybdena Glance* [Mo 2 S];

(b) As Molybdic Acid [Mo O<sup>3</sup>], *per se* as a yellow coating upon the Sulphuret of Molybdenum, and with Oxide of Lead, in *Gelb-Bleierz (Molybdate of Lead)*.\*

*Examination for Molybdenum.*

The molybdenum in Molybdena-Glance is detected by means of microcosmic salt upon the platinum wire. This flux appears at first not to dissolve any of the mineral, but, after a long-continued oxidating-flame, the microcosmic bead assumes a greenish colour, owing to molybdic acid. If the undissolved portion of the assay be removed with a forceps from the molten bead, and what remains treated with a reducing flame, the green colour will be very characteristic. Molybdic acid, before the blowpipe, fuses on charcoal, volatilizes, and imparts to the flame a yellowish-green tinge; and where the

\* [Boussingault found, in *Basic Molybdate of Lead*, from Paramo-Rico, (near Pamplona, in South America), Lead, Molybdic Acid, Carbonic Acid, Hydrochloric Acid, Phosphoric Acid, Chromic Acid, Peroxide of Iron, Alumina, and Silica.]—*Trans.*



assay rested, a reddish shining metallic speck is observed. The acid deposits also a crystalline coating upon the charcoal. Sulphuret of Molybdenum, and also the metal, give, without fusing, the same reaction. The green colour imparted to borax by molybdic acid, remains perfectly transparent after exposure for a very long time to the oxidating flame; therefore the operator may rest satisfied that this reaction proceeds from no other metallic oxide.

According to Berzelius, the Native Molybdic Acid from Bispberg, comports itself in a similar manner to the pure acid, but when treated with soda upon charcoal, it is imbibed, and peroxide of tin remains behind.

Gelb-Bleierz is submitted to the same treatment as the Molybdena Glance, for the detection of the molybdic acid; with this precaution, that too much of the specimen be not added, or otherwise a black opaque glass will result.

Merlet's moist method for the detection of molybdenum in the foregoing minerals, is the following. Powder the specimen, and fuse with nitre in a platinum spoon; molybdate of potash is formed. Dissolve in a porcelain basin with water over a spirit-lamp; decant off the clear solution into another vessel, in order to get rid of the residuary matters that peradventure may occur, and then treat the hot aqueous solution with hydrochloric acid, and immerse in the menstruum a strip of metallic copper. If only a trace of molybdic acid be present, the liquid, where the copper rests, will acquire a beautiful blue colour, which disappears as the solution cools, but is immediately restored by heat.\*

\* [The following is inserted on account of my attention having been directed to it in a communication from the author.]—*Trans.*

“Metallic molybdenum, which is obtained by fusing the oxide in a crucible lined with charcoal, is not fusible before the blowpipe; but when heated upon charcoal, in the oxidating flame, it becomes oxidized; and gradually coating, at no great distance from the assay, the support with a sublimate, which in many places, but particularly nearest the assay, is in transparent silky shining crystalline plates, also in a pulverulent form. The deposit of molybdic acid is, while hot, yellow; and when cold, white. The crystalline plates appear the most beautiful when the assay is kept far from the blowpipe-flame; but the heat must be properly regulated for their



§ 19. TUNGSTEN—[W].—*Presence in the Mineral Kingdom.*

This element exists in nature as an Acid only, either *per se* [ $\text{W O}^3$ ], or in combination with Bases,—namely; with Lime, in *Schwerstein* (*Tungstate of Lime*) [ $\text{Ca O, W O}^3$ ]; with Oxide of Lead, in *Tungstate of Lead* (*Scheelbleispath*); with Iron, and Protoxide of Manganese, in *Wolfram* [ $\text{Mn O, W O}^3 + 3 (\text{Fe O, W O}^3)$  ]—its composition has been represented by Berzelius as follows: [ $\text{Fe}^2 \text{O}^3, 2 \text{W O}^3 + \text{Mn}^2 \text{O}^3, 2 \text{W O}^3$  ] ; further, as a very small ingredient in some varieties of *Tantalite*, and in *Black and Brown Ytthro-Tantalite*.

*Examination for Tungsten.*

Tungstic acid is readily detected in *Schwerstein*, *Wolfram*, and *Tungstate of Lead*, in the following manner: Mix a small portion of powdered mineral with five times its volume of soda, and heat the mass strongly in a platinum spoon. Dissolve the fused assay in boiling water, and decant off the supernatant clear liquor, which contains tungstate of soda. If this solution be treated with hydrochloric acid, a white powder deposits, which, when heated, assumes a beautiful lemon colour. The behaviour of the tungstic acid in *Schwerstein*, towards microcosmic salt, has been previously given, (page 102).

formation. The sublimate can be driven along the charcoal by the oxidating flame; but the place which it leaves appears, when perfectly cold, dark copper-red, and shining oxide of molybdenum remains, which is produced by the molybdic acid coming in contact with the glowing charcoal. Molybdenum remains intact in the reducing flame.

Sulphuret of Molybdenum (*Molybdena Glance*) which also is infusible, affords, in a continued strong oxidating flame, the same sublimate as pure Molybdenum, while its sulphur escapes as sulphurous acid.

Molybdic Acid fuses very readily, volatilizes in the oxidating flame, coating the charcoal with crystalline acid, and copper-red metallic shining oxide. Molybdenum possesses, in an oxidized condition, the property of tinging the outer blowpipe-flame; *e. g.*, if molybdic acid, in a moistened state, be treated on platinum wire with the apex of the blue flame, it volatilizes, colouring the outer flame yellowish-green, analogous to barytes. The same coloration is produced by *Molybdena Glance*, as may be seen by heating a thin fragment of this mineral, held in the platinum forceps, in the apex of the blue flame. No fusion takes place, but the outer flame assumes a yellowish-green colour.”—*Plattner on the behaviour of Molybdenum, Molybdic Acid, and Sulphuret of Molybdenum, per se, in the Blowpipe-flame.*



When the Tantalites are examined in this manner for tungstic acid, tantalic acid (if the tantalium is contained as an acid) will accompany the tungstic acid, which prevents the yellow reaction when heat is applied to the mixture. Should this occur, the white powder must be separated from the solution by filtration,edulcorated, and treated with microcosmic salt, as follows: dissolve in the microcosmic salt upon a platinum wire, so much peroxide of iron that the glass produced will appear colourless or slightly yellow in the oxidating flame, and, when treated in the reducing flame, perfectly colourless when cold. Now add some of the moist precipitate, and submit the whole, first to an oxidating and then to a reducing flame. If tungstic acid be present, the glass will be tinged, either yellow, red, or blood-red,\* according to the quantity of the substance added. This reaction is so exceedingly accurate, that if a *trace* only of tungstic acid be contaminated with the tantalic acid, the ferruginous microcosmic glass will acquire a yellowish hue. The operator must exercise great care in adding the peroxide of iron, to avoid a blue reaction being obtained in the reducing flame. As nearly all Tantalites contain more or less protoxide of iron, this method will very seldom have to be employed, as the reaction is speedily produced by dissolving the pulverized mineral in microcosmic salt, and treating in the reducing flame; if tungstic acid be present, a transparent dark red bead results.† The presence of protoxide of manganese, oxide of tin, yttria, and lime, does not affect the coloration.

\* [As mistakes may arise, on account of titanlic acid affording a similar reaction to tungstic acid, with microcosmic salt, the examination should be extended. According to Rose, tungstic acid gives a yellow glass with borax, and titanlic acid, a violet one, which becomes "turbidly streaked."]—*Trans.*

† [Tungstic acid is most conveniently obtained by decomposing the native Tungstate of Lime, finely pulverized, by hydrochloric acid; chloride of calcium is formed, and tungstic acid precipitates. Dissolved in ammonia, and precipitated again by acid, tungstic acid always forms a compound with the acid employed. It may be obtained in a separate state, by heating the tungstate of ammonia to redness. It is an orange-yellow powder, which becomes dull green when strongly heated. It is quite insoluble in water, or in acids, but dissolves in alkaline solutions.—*Graham.*]—*Trans.*



Ferruginous titanio acid, ferruginous oxide of nickel, and ferruginous antimonious acid, give a similar reaction with microcosmic salt, as ferruginous tungstic acid.

§ 20.—*VANADIUM*—[ V ].—*Presence in the Mineral Kingdom.* \*

It has been found in an *Iron Ore* from Taberg, a *Lead Ore* from Zinnapan in Mexico, also from Warloch-head in Scotland (which is principally *Vanadate of Lead*), in *Volborthite*, *Pea Iron Ore* (from Steinlade, near Goslar), in *Mansfield Copper Slate*, *Hydrophite*, and *Vanadate of Lime*. According to Berzelius, the presence of Vanadium in *Pitchblende* may arise from the presence of *Vanadate of Lime*, the recently-discovered mineral.

Vanadium is a very rare element.

*Examination for Vanadium.*

The pyrognostic properties of Vanadate of Lead, are very characteristic. On charcoal, before the blowpipe, it strongly decrepitates, fuses to a globule, then scintillates, giving a regulus of lead, and the support becomes coated with a yellow sublimate.

With microcosmic salt, it gives, in the oxidating flame, a reddish yellow, and, after cooling, a yellowish green glass; in the reducing flame, it affords a beautiful chrome-green bead.

*Per se*, it fuses when held in the forceps, and presents, on refrigeration, its yellow tinge.

Vanadium, *per se*, on the platinum wire, in the oxidating flame, is converted into vanadic acid, which, with borax, gives, while hot, a dark yellow, and, on cooling, pale yellow glass. In the reducing flame, the glass is, while hot, greenish or brownish, but, on cooling, chrome green,—it is not coloured blue by the addition of tin.

Vanadic acid, with microcosmic salt, dissolves readily, producing a reddish yellow glass, which is, on cooling, pale yellow. It acquires a fine transparent green hue in the re-

\* [The whole of the "Examination for Vanadium" has been introduced by the translator.]



ducing flame, but the dark yellow colour is recalled in the oxidation.

With soda, it easily dissolves, and is absorbed by the charcoal.

On platinum foil, it fuses to a deep yellowish red fluid, which becomes crystalline by refrigeration.

Vanadic acid is distinguished from oxide of chromium by producing a yellow glass on platinum wire in the oxidating flame, which is never the case with the latter.

The following is Seftström's method for obtaining the vanadic acid from slags or the minerals :—Fuse with an equal part of saltpetre, and two parts carbonate of soda,—when the temperature is very high, vanadate of soda is formed,—affuse the resulting mass with boiling water, and neutralize the liquid with nitric acid, then add chloride of barium, or acetate of lead,—a precipitate of vanadate of barytes or lead, is obtained; treat with sulphuric acid, and then separate the red liquid from the sulphate, digest it with alcohol, whereby, under formation of ether, the vanadic acid is resolved into the peroxide of vanadium, and the solution is blue; evaporate to dryness, and then heat the residue to strong redness to expel sulphuric acid,—impure vanadic acid remains; smelt it with nitrate of potash until a portion of the cold sample loses its reddish appearance. Dissolve, filter, and immerse in the liquid a large piece of sal-ammoniac; a white precipitate of vanadate of ammonia will appear, which is insoluble in a saturated solution of the precipitant; filter and edulcorate, first with a solution of chloride of ammonium, and then with spirits of wine of sp. gr. 0.860, dissolve in boiling water, containing a few drops of free ammonia, and, upon cooling, pure vanadate of ammonia is obtained, which is the salt from which all the other compounds are formed.

From the solution of the vanadate of lead in nitric acid, the lead and arsenic can be separated by hydrosulphuric acid; the resulting blue solution of peroxide of vanadium, on evaporation to dryness, yields vanadic acid, with which pure vanadate of ammonia may be formed, by following the foregoing method.



§ 21. *TANTALUM\** (or *COLUMBIUM*)—[Ta].—*Presence in the Mineral Kingdom.*

This metal is found in nature, partly as an acid [Ta O<sup>3</sup>] in combination with bases, and partly as an oxide [Ta O<sup>2</sup>], in union with other metallic oxides; namely,—

(a) As an acid, with Yttria, Lime, and more or less Tungstate of Iron, in *Black and Brown Ytthro-Tantalite*; with Yttria, and Oxide of Uranium, in *Yellow Ytthro-Tantalite*; and with Iron and Protoxide of Manganese, in *Tantalite*. These varieties are found in different places, and often contain Peroxide of Tin, Tungstic Acid, and Lime; *e. g.*, *Kimito-Tantalite* [Fe O, Ta O<sup>3</sup> + Mn O, Ta O<sup>3</sup>]; *Finbo-Tantalite*  $\left\{ \begin{smallmatrix} \text{Mn O} \\ \text{Fe O} \end{smallmatrix} \right\}$   $\left\{ \begin{smallmatrix} \text{Ta O}^3 \\ \text{Sn O}^2 \end{smallmatrix} \right\}$ ; *Broddbo-Tantalite*  $\left\{ \begin{smallmatrix} \text{Mn O} \\ \text{Fe O} \\ \text{Ca O} \end{smallmatrix} \right\}$   $\left\{ \begin{smallmatrix} \text{Ta O}^3 \\ \text{W O}^3 \\ \text{Sn O}^2 \end{smallmatrix} \right\}$ ; and *Bodenmais Tantalite* [3 Mn O, 2 Ta O<sup>3</sup> + 3 Fe O, 2 Ta O<sup>3</sup>];

(b) As an oxide, with Protoxides of Iron and Manganese, in a *Tantalite* from Kimito  $\left[ \begin{smallmatrix} \text{Fe O} \\ \text{Mn O} \end{smallmatrix} \right] \text{Ta O}^2$ ].

*Examination for Tantalum.*

The presence of Tantalum is recognized in the different Tantalites by heating with borax, whereby a chrome or iron-tinged glass is obtained, which may in some cases present a “turbidly streaked” enamel appearance. With microcosmic salt, they either dissolve, leaving a skeleton of tantalic acid, or afford a transparent glass coloured by chromium, iron, or tungsten. These minerals are decomposed, but not dissolved, by soda on charcoal.

The examination for this element is similar to that for Tungsten. It is as follows:—Smelt, in a platinum spoon, the pounded mineral, with three times its volume of soda, and twice its volume of nitrate of potash,—tantalate of potash is formed; dissolve the residuum with water in a porcelain basin, filter, and add to the filtrate a few drops of hydrochloric acid,—

\* [Tantalum was first discovered in an American mineral, whence its name (Columbium); it was subsequently found in some very rare Swedish minerals, but independently, and, from the difficulty of its extraction, the name tantalum was given to it. Metallic columbium or tantalum is a black powder, which, when burnished, appears iron-grey.—*Kane.*]—*Trans.*



tantalic, associated with a large proportion of tungstic acid, is by this means deposited; this precipitate remains white when heated, and is therefore distinguished from the pure tungstic acid, which acquires a yellow colour. This acid is detected in tantalic by its pyrognostic effects with a ferruginous micro-cosmic bead, as was previously given under Tungsten.

The small quantities of tantalic acid which, according to Berzelius, are detectable by moist analyses in a variety of Beryls, are with difficulty recognizable before the blowpipe, owing to the presence of siliceous combinations.

§ 22. *TITANIUM*—[Ti].—*Presence in the Mineral Kingdom, and in the Products of Smelting Furnaces.*

It is found as an acid  $[\text{TiO}^2]$  *per se*, and also in combination with bases, thus:—

(a) *Per se*, in *Anatase* and *Rutile*; the latter generally contains some Protoxides of Iron and Manganese;

(b) In combination with Earths and Metallic Oxides; *e. g.*, with Lime and Silicate of Lime, in *Titanite* and *Sphene*; with Lime, Protoxides of Cerium, Uranium, Iron, Manganese, Tin, &c., in *Pyrochlore*; with Zirconia, Yttria, Oxides of Cerium, Iron, Calcium, and traces of Potash, Magnesia, Silicic Acid, and Oxide of Tin, in *Polymignite*; with Protoxide of Iron, in many rich slags, *e. g.*, in *Titaneisen*, *Crichtonite*, *Menaccanite*, *Nigrin*, *Iserine*, *Ilmenite*, *Volcanic Iron*, *Eisensand*; and in all refractory Iron Ores whose slags are of a vitreous nature;

(c) As a very small ingredient in some minerals, it is found in *Cymophane*, *Kyanite*, *Achmite*, and in some varieties of *Mica*.

When Titaniferous Iron Ores are smelted, the Titanium sometimes separates in the metallic state, and is seen upon the slags in small copper red crystals.\*

*Examination for Titanium.*

The titanium contained in *Anatase*, *Rutile*, *Titanite*, and *Sphene*, can be detected by the behaviour of these minerals

\* [I have seen it in beautiful perfect cubes, upon a slag which Mr. W. K. O'Sullivan obtained from the smelting works near Frankfort-on-the-Maine.]—*Trans.*



with fluxes; *i. e.*, the first two comport themselves with borax, microcosmic salt, and soda, like titanitic acid, the other two afford only the titaniferous colour with microcosmic salt. In other minerals where titanium forms an ingredient, it is somewhat difficult to detect it by the aid of the fluxes, as the other metallic oxides in combination hinder the reaction. If, however, ferruginous titanium ore be dissolved in microcosmic salt, and the glass, which exhibits the colour of peroxide of iron only, treated for a long time in the reducing flame, a more or less brownish red tinge is imparted to the assay, similar to that produced by ferruginous tungstic acid. The intrinsic quantity of titanium present, is deduced from the depth of colour assumed by the glass. If the quantity present be great, the characteristic reaction of oxide of titanium (titanic oxide) will be obtained by treating with tin in microcosmic salt upon charcoal; but if only a small portion be present, this end is not attainable. Titanium, when forming an essential or appreciable ingredient in minerals, may be detected by a very simple method, as follows:—Fuse gradually the finely pulverized mineral in a platinum spoon, with from six to eight times its volume of bisulphate of potash; dissolve out in hot water, (tem. about 200 Fahr.,) and allow the solution to settle; decant off the clear supernatant liquor into a flask containing more hot water, and boil. If the mineral contained even small quantities of titanium, it will be deposited as a white powder, (titanic acid,) provided iron is not present in the state of protoxide, otherwise it will have a yellow tinge, probably from holding some of the iron in combination. This precipitate is to be separated by filtration, and examined (without beingedulcorated) with microcosmic salt, either on a platinum wire or charcoal. If the quantity be so small that no violet colour is imparted to the microcosmic salt, the operator must add to the assay, treated on the wire, a small quantity of peroxide of iron, and, when heated on charcoal, a piece of iron wire, and then heat the glass for some time in the reducing flame; it will appear, while hot, yellow, and upon cooling, red, analogous to oxide of nickel, or ferruginous tungstic acid. This reaction is pro-



duced immediately by the yellow precipitate, when heated with microcosmic salt in the reducing flame.

When such a precipitate is dissolved in borax, the iron has no effect upon the colour produced by the titanous acid.

Should the operator not be perfectly convinced of the absence of tungstic acid, the method with bisulphate of potash will be the most decisive for the detection of the titanium, as the titanous acid is precipitated on boiling the solution, (and any tungstic acid remains dissolved,) and can then be treated with microcosmic salt, as previously given.

If Polymignite be smelted with even ten times its volume of bisulphate of potash, and the fused mass treated with warm water, little or no titanous acid is dissolved; it remains behind, with zirconia and traces of protoxide of iron, as a white voluminous mass, and can readily be recognized by collecting on a filter, and then treating some of the precipitate with microcosmic salt. The metallic titanium found in ferruginous slags dissolves with difficulty in borax, but is pretty readily soluble in microcosmic salt. The crystals which I applied in this research had been examined under the lens and considered as pure, but the yellow microcosmic salt glass was, in the reducing flame, neither *per se* nor with the addition of tin, of a fine violet colour, but somewhat reddish, like the ferruginous titanous acid.

§ 23. CHROMIUM—[Cr].—*Presence in the Mineral Kingdom.*

Chromium occurs only in an oxidized state in nature, thus:—

(a) As Oxide  $[\text{Cr}^2\text{O}^3]$  in a mechanical mixture of  $[\text{Cr}^2\text{O}^3]$  with Quartz and various kinds of primitive rocks, in *Oxide of Chrome*, or *Chrome Ochre*; as Oxide, with Protoxide of Iron, Alumina, and Magnesia, in *Chromate of Iron*, (*Chrome Iron Ore*),  $[\frac{\text{FeO}}{\text{MgO}}] + [\frac{\text{Cr}^2\text{O}^3}{\text{Al}^2\text{O}^3}]$ .

(b) As Chromic Acid  $[\text{CrO}^3]$  with Lead, in *Chromate of Lead*, and with Lead and Oxide of Copper in *Vauquelinite*. In addition to these, Oxide of Chromium and Chromic Acid form an extraneous ingredient in many Siliceous combinations,



to which many of them owe their beautiful green or blood-red colour; *e. g.*, the precious *Emerald*, the *Pyrope*, &c.

Traces of Chromic Acid are also present in *Schiller Spar*, and in some varieties of *Serpentine*.

*Examination for Chromium.*

The presence of chromium in most of the above minerals is readily detected by their behaviour with borax and microcosmic salt before the blowpipe, the bead being of a beautiful green when perfectly cold. This colour is produced by minerals which contain neither lead nor copper, particularly after treatment in the reducing flame. When the minerals contain oxides of lead or of copper, *e. g.*, Chromate of Lead and Vauquelinite, the glass bead affords, in the reducing flame, a grey or red colour, the peculiar green of the Chromium being completely destroyed; in consequence of this, attention should be paid, in the examination of such minerals, to those colours only which they produce in the oxidating flame.

Minerals which contain little chromium, and a large quantity of other metals which yield colouring oxides, and afford no satisfactory chrome reaction with borax and microcosmic salt, can be examined for chromium in the following manner. A small fragment of the mineral is first powdered as fine as possible, the powder mixed with three times its volume of saltpetre, and the mixture fused with the aid of the blowpipe, either on the platinum wire or in a small platinum spoon. Chromate of potash is thus produced, and may be dissolved in water in a porcelain capsule, over the spirit-lamp. If the clear solution be poured off from the insoluble metallic oxides into another capsule, supersaturated with acetic acid, and a crystal of acetate of lead then laid in it; the latter dissolves, and the chromic acid instantly combines with the oxide of lead, forming a beautiful orange-yellow powder, which precipitates, and, after filtration, yields with borax or microcosmic salt, in the oxidating flame, a beautiful green glass. Even small traces of chromium may be detected in this manner. If the substance examined by this method for chromium should contain sulphur or sulphuric acid, (which is



not however present in the natural compound,) the chromate of lead will be contaminated with sulphate, and the yellow colour will be lighter in proportion to the larger or smaller quantity of the sulphate present. Silicates which contain only a little chromium, and much iron or other metals yielding coloured oxides, and which afford with fluxes only the peculiar tinge of iron or of the other metals, cannot be examined for chromium by the foregoing method, siliceous combinations not being decomposed by saltpetre, but requiring a different treatment; as, for example, the following. The finely-pulverized mineral is to be fused to a bead on charcoal, with from 1 to  $1\frac{1}{2}$  parts of soda, and  $\frac{1}{2}$  a part of borax; the bead pulverized, treated with hydrochloric acid, and evaporated to dryness. The resulting dry mass is to be dissolved in water, the silicic acid separated by filtration, a few drops of nitric acid added to the solution, in order to convert the protoxide of iron into the peroxide, and the oxides of chromium, iron, and alumina, &c., precipitated by ammonia. This precipitate, after being collected on a filter, must be fused with saltpetre; chromate of potash is thus obtained, which may be decomposed by acetate of lead, in the manner already described.

The silicates coloured blood-red by chromic acid, namely, the Pyrope and Spinnelle, from Ceylon, possess the property of becoming black and opaque by simple heating, but during the refrigeration they become, by transmitted light, gradually yellowish or chrome-green, then almost colourless; and when perfectly cold they re-exhibit their original colour. Those siliceous combinations which are coloured red both by chromium and iron, are also rendered opaque by calcination, but on cooling, immediately recover their redness and transparency.

§ 24. *TELLURIUM*—[Te].—*Presence in the Mineral Kingdom.*

Tellurium occurs in nature only in the metallic state; thus,—

(a) As *Native Tellurium* [Te], which contains a little Gold and Iron;



(b) With Gold and Silver, in *Graphic Tellurium*, from Nagyag; with Lead, in *Black Tellurium*, mechanically mixed with Telluret of Gold, and sometimes Sulphurets of Silver, Copper, and Antimony; with Bismuth, a little Sulphuret of Bismuth, and Selenium, in *Telluric Bismuth*; with Silver, Lead, and Gold, in *Yellow Tellurium*; and in a compound of Bismuth, Lead, Copper, and a little Nickel, in the *Needle Ore* from Siberia.

*Examination for Tellurium.*

A substance may be examined for tellurium either on charcoal or in a glass tube, open at both ends. In the first case, a white sublimate, with a reddish border, is obtained by heating the mineral in either flame, and which disappears, with a beautiful bluish green tinge, when the reducing flame is directed on it. When the mineral is rich in tellurium, it tinges the flame bluish while subjected to the action of the reducing flame. If the assay, thus treated, gives off the odour of horseradishes, it contains selenium. If the mineral contains lead or bismuth, and the operation be performed on charcoal, the pure sublimate of oxide of tellurium is never obtained when the blast is continued for a few seconds too long, but, on the contrary, will always be contaminated with oxide of lead or bismuth. It will, in consequence, be safer to pulverize the substance, mix it with an equal bulk of glacial boracic acid, place it on a broad charcoal support, and treat it in the reducing flame. The oxides of lead and bismuth thus formed, notwithstanding the action of the reducing flame, will be dissolved by the boracic acid, and will not produce a sublimate; while the tellurium volatilizes and alone coats the charcoal. Should the mineral, at the same time, contain a large quantity of selenium, a portion of it will also be deposited on the charcoal, and the tellurium cannot then be well recognized. In such a case, it is always better to perform the operation in a tube open at both ends.

If the operation is performed in a glass tube in a similar manner to Antimony, a strong white vapour is obtained from



telluriferous minerals, which deposits itself as a pulverulent oxide on the glass, which, by heating, fuses on the same part of the tube on which it is deposited, to clear colourless drops, which may be more closely examined with a lens. Should, however, selenium be present, a trace of selenite of tellurium will be formed, which also melts into clear colourless drops, incapable of being distinguished from pure oxide of tellurium by the unaided eye.

When the telluriferous metal contains much lead, a sublimate is obtained which appears grey close to the assay and white at a distance from it. The white portion can be fused to colourless drops, and is oxide of tellurium; the grey, on the contrary, does not fuse like oxide of tellurium, but exhibits a somewhat changed appearance, and forms only a semi-fused greyish coating on the glass. According to Berzelius, this substance is tellurate of oxide of lead.

If bismuth be also present in the telluriferous metal, it remains behind, while the tellurium volatilizes and deposits in the tube. By continued treatment in the flame, it oxidizes, but the surface of the bead is coated with fused brown oxide of bismuth.

§ 25. *ARSENIC*—[As].—*Presence in the Mineral Kingdom, and in the Products of Smelting Furnaces.*

Arsenic is found very abundantly in nature. It occurs:—

(a) Metallic, either *per se* or in combination with other metals: namely, as *Native Arsenic* [As], which generally contains traces of Antimony and Iron; with Cobalt, in *Speisscobalt*, which often contains traces of Iron, Copper, and Sulphur; with Nickel, in *Arsenical Nickel* (*Kupfernickel*), which sometimes contains traces of Iron, Lead, Cobalt, Antimony, and Sulphur; with a lesser proportion of Nickel, in *Weissnickelkies*, from Schneeberg; with Antimony, in *Arsenical Antimony* (*Arsenik-Speisglanz*); with Bismuth, in *Bismuth Blende*; and with Silver, Iron, and Antimony, in *Arsenical Antimonial Silver*.

(b) With Sulphur, both *per se* and combined with other Sulphurets: namely, *per se*, as *Sulphuret of Arsenic*, red



[ $\text{AsS}^2$ ] (*Realgar*); yellow [ $\text{AsS}^3$ ] (*Orpiment*); with Iron, in *Arsenical Pyrites*; with Cobalt and a little Iron, in *Bright White Cobalt*; with Nickel and Iron, in *Nickel Glanz*; with Copper, Antimony, Iron, and Silver, in *Grey Copper* (*Fahlerz*); with Silver and a little Antimony, in the light red variety of *Ruby Silver* (*Argent Rouge Arsenié*, Necker); with Nickel and Antimony, in *Antimonial Nickel* (*Nickelspeissglanzerz*); and with Copper and Iron, in *Tennantite*.

(c) As an acid; thus,—as Arsenious Acid *per se*, in *Oxide of Arsenic* (*Arsenikblüthe*) [ $\text{AsO}^3$ ]; and as Arsenic Acid and Arsenious Acid, combined with other bodies; namely, as Arsenic Acid, with Lime and Water, in *Pharmacolite*; with Oxide of Cobalt and Water, in *Cobalt Bloom*; with Oxide of Copper and Water, in *Condurrite*, *Euchroite*, *Erinite*, *Rhombohedral Arseniate*, and, accompanied by Alumina, in *Liroconite*; with Oxide of Nickel and Water, in *Nickel Ochre*, which often contains traces of Oxides of Cobalt and Iron, and Sulphuric Acid; with Protoxide of Iron and Water, in *Scorodite*; with Protoxide of Iron, Peroxide of Iron, and Water, in *Cube Ore*, or *Arseniate of Iron*, from Cornwall; with Oxide and Chloride of Lead, in *Arseniate of Lead*, from Johann Georgenstadt and Cornwall, which also contains traces of Iron and Phosphoric Acid; and with Oxide and Basic Sulphate of the Oxide of Iron, together with Constitutional Water, in *Pitchy Iron Ore*, or *Eisensinter*; lastly, as Arsenious Acid, combined with Oxide of Cobalt, in a pulverulent substance from Schneeberg.

As many of the above-mentioned Arsenic combinations partly *per se*, and partly in combination with other substances, are subjected, on a great scale, to operations for the reduction of the metals which they contain, and as Arsenic is a metal which is very difficultly separated in the dry way from a number of other metals, it must form more or less a constituent of the "*Dressed Ores*," and of the various intermediate products of the smelting operation. To the latter belong especially the products which are obtained in Arsenic-Smelting Works, as well as many of those which are obtained in Silver Works, as *Rohstein*, *Bleistein*, *Tutty*, *Abstrich*, &c.,



and also the *Speiss* produced in Cobalt-Smelting Works, and often, even the *Smalts*.

*Examination for Arsenic.*

The examination for arsenic is in most cases very simple. It should, however, be ascertained whether the arsenic exists in the compound in a metallic state or as an acid. Native Arsenic is instantly recognized, by the alliaceous odour which it exhales in volatilizing when treated on charcoal before the blowpipe. In a glass matrass, it sublimes in a metallic state, leaving a non-volatile metallic mass behind, which, by fusion with lead and cupellation on bone ashes, sometimes yields a button of silver. The combinations of arsenic with sulphur, as Realgar and Orpiment, burn with a whitish yellow flame when ignited with the aid of the blowpipe on charcoal, and give off a dense greyish white vapour, which smells like garlic, and deposits itself in part on the charcoal. It fuses in a flask, boils, and sublimes. The sublimate is transparent, and of a dark yellow or red colour. Heated in an open glass tube, it burns and deposits white arsenic on the upper side of the tube; at the end of the tube, held upwards, the smell of sulphurous acid is readily recognized. Metallic arsenic is best obtained from sulphuret of arsenic by the following method:—The compound is first decomposed in an open glass tube into arsenious and sulphurous acids, the former of which deposits itself in a crystalline form in the tube, and the latter escapes. For this purpose, the tube must be held obliquely, and heated a very little above the specimen under examination, in order that the ascending vapour may pass over the hottest part of the tube, and be thus perfectly burned. The glass tube is then to be drawn out close to where the arsenious acid has collected, the latter forced into the part thus retracted, and reduced, with charcoal, by the method which will be more fully treated of hereafter, for the reduction of pure arsenious acid to the metallic state. The greater part of the combinations of arsenic with other metals, possess this property, that when a small portion of them is placed on charcoal and heated in the reducing flame, a part of the arsenic



is immediately volatilized, and the charcoal covered with a white coating by it. When the quantity of arsenic present is considerable, a dense greyish white smoke ascends from the assay, which diffuses itself, and may be readily recognized, even at a considerable distance, by its alliaceous smell; but if the quantity is inconsiderable, the above vapour is not always seen during the blowing, and the arsenical smell experienced. In this case, the red-hot assay should be brought close to the nose, in order that the arsenic escaping may be recognized by its peculiar smell. If a small quantity of arsenic is combined with a metal from which it is with difficulty separated, *e. g.*, with nickel, the compound should be melted with proof lead in the oxidating flame, and the smell observed to ascertain if arsenic is volatilized.

Many compounds of arsenic with metals afford metallic arsenic when heated in a glass flask; *e. g.*, some varieties of Speisscobalt, Weissnickelkies, Arsenical Antimony, &c.; while others yield none, *e. g.*, Arsenical Nickel (Kupfernichel). All the metallic arseniurets which occur in nature, when heated in an open glass tube, yield arsenious acid, which sublimes in combination with oxide of antimony, when the latter metal is present in the substance under examination.

If a metallic compound yields no arsenical smell when heated on charcoal *per se*, or with proof lead, and gives no sublimate of arsenious acid in an open tube, the presence of a minute trace of arsenic may still be detected by the following method:—

Friable metallic compounds, and such as can be reduced to powder, should be pulverized, and of the non-pulverizable, a portion sufficient for an assay should be filed off. Artificial nickeliferous alloys, prepared on the great scale, do not require reduction to a finely divided state, as they are obtained either in a fine powder, or as a slightly adhering easily pulverizable mass; melted nickel, on the other hand, German silver, and similar compositions, must be divided in this way. From 75 to 100 milligrammes of the metallic powder are then to be mixed in an agate mortar, with from five to six times as much saltpetre, and the mixture strongly ignited on a pla-



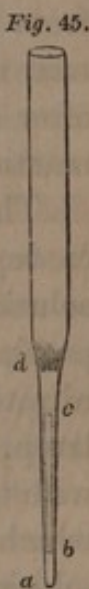
tinum spoon in the oxidating flame, (see p. 69,) by which means the metals are oxidized, and the arsenic acid formed, is combined with the free potash of the saltpetre. The fused mass is then removed, the spoon placed in a porcelain cup, water poured on it, and the whole warmed over the spirit-lamp, by which treatment the arsenic acid formed, the carbonate of potash, and the remaining undecomposed nitrate of potash are dissolved. If the metal to be examined contains zinc, lead, tin, or bismuth, slight traces of these metals will be present in the solution, as free potash dissolves the oxides of these metals; these traces do not, however, hinder the recognition of the arsenic. The solution, which should be somewhat concentrated, is then diluted, without being separated from the residue, with spirits of wine, and powdered tartaric acid dissolved by a moderate heat in the alcoholic solution, after decantation from the residue, until it affords the acid reaction on litmus paper.

The bitartrate of potash formed, should then be allowed to deposit from the liquid in which it is insoluble. If the solution appears clear, it is to be carefully poured off from the precipitated salt into a porcelain basin, a small crystal of nitrate of silver placed in it, and the whole heated over the lamp. Should the solution contain arsenic acid, it combines with the oxide of silver, forming an insoluble arseniate of silver, which falls to the bottom as a brownish red powder, but if the solution is free from arsenic, the nitrate of silver dissolves without producing any precipitate. Arsenical sulphurets, when heated on charcoal in the reducing flame, readily yield a strong or a weak arsenical smell. If the quantity of arsenic contained in a sulphuret be extremely minute, neither the vapour nor the smell of arsenic can be perceived. Such substances (to which, *e. g.*, *Rohstein*, *Bleistein*, *Flue-rakings*, &c., belong), must be pulverized, the powder mixed with soda, and the mixture fused on charcoal in the reducing flame. The sulphur is by this means separated by combining with the radical of the soda, while the arsenic is volatilized and may be recognized by its characteristic smell. The native arsenical sulphurets, according to their various constituents, when heated in a bulb



tube, sometimes yield a little sulphuret of arsenic, with much metallic arsenic, *e. g.*, Arsenical Iron; sometimes only sulphuret of arsenic, *e. g.*, Nickel Glance; and sometimes no sublimate, *e. g.*, Bright White Cobalt. In a tube open at both ends, however, they all yield arsenious and sulphurous acids.

Pure arsenious acid may be tested before the blowpipe, either on charcoal with soda, or by a method which Berzelius has given. The latter method is decidedly the safest, and most accurate when small quantities are to be examined. The following is the mode of procedure:—A glass tube is to be drawn out so that the diameter of the part drawn out is only about the thickness of a strong knitting-needle, and the end then sealed up. In order to detect the arsenious acid (which may be even less than one milligramme), it is to be introduced into the elongated part, forced into the extreme end (*a*, *Fig. 45*), and a fragment of charcoal placed over it, about the size represented by *b c*; the part of the tube occupied by the charcoal is then heated in the flame of the spirit-lamp, until the charcoal becomes ignited, and the part containing the arsenious acid gradually introduced into the flame. The arsenious acid is volatilized, and on coming in contact with the ignited charcoal, is reduced, forming a deposit in the cold wide part of the tube of metallic arsenic. When the quantity of arsenious acid under investigation is extremely minute, a black trace of metallic arsenic is only obtained between *c* and *d*. If the part of the tube drawn out be gradually heated nearer and nearer the black trace, the latter may be made to form a ring on the glass, and if the part retracted be cut off at *c*, and the part *d* held in the flame of the spirit-lamp, the arsenic may be volatilized, and the peculiar alliaceous smell recognized.\*



When the arsenic is present as an acid, combined with earths, or metallic oxides, three modes can be employed for its detection:—

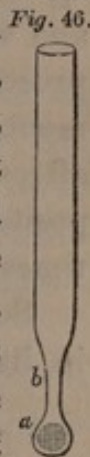
\* [This method is far more delicate if cyanide of potassium be substituted for charcoal. In this case, also, other compounds, besides arsenious acid, can be examined.]—*Trans.*



The first and simplest is, to mix the substance with soda, then treat it on charcoal in the reducing flame, and examine whether the smell of arsenic is evolved. This test is not, however, delicate enough in all cases, especially when the arsenical acids are combined with metallic oxides, which are easily reduced, and form, with metallic arsenic, readily-fusible arsenical alloys, which yield with difficulty the smell of arsenic. This is more particularly the case with oxide of nickel.

The second method for examining compounds for arsenical acids, combined with earths, is to mix the substance with charcoal powder, and heat the mixture in a small tube having a bulb blown on one end of it, by which the arsenical acids are reduced, and metallic arsenic sublimed. Metallic arseniates and arsenites, with the exception of arsenite of copper (known in commerce under the name of Scheel's Green), cannot be examined for arsenic in this way, because arsenical alloys are often formed by the bases accompanying the acid being reduced with it, from which arsenic cannot be sublimed.

To the compounds of the arsenical acids with earths, which admit of being examined in this way, belongs, in particular, arsenite of lime, which is often obtained only in exceedingly small quantities, in medico chemical examinations for legal purposes. Berzelius, in his *Lehrbuch der Chemie*, gives the subjoined method of procedure in such a case:—The arsenite of lime is gently ignited, then mixed with fresh ignited charcoal, and introduced into the end of a sealed tube, (Fig. 46), a part of the closed end of which is elongated, so that the mixture fills it up to (a). The tube is next heated, gently at first, in order to drive off moisture, and then the part *a*, containing the substance, heated in the blowpipe flame until near the fusing point of the glass. The arsenic is by this means reduced, and collects in the narrow part (b) of the tube, where, from the very small surface over which it is spread, the most minute traces may be detected.



The third method for the detection of arsenic in arsenites



and arseniates having earths or metallic oxides for their bases, is to mix the substance with three times its bulk of saltpetre, and proceed in the manner already described for the examination of arseniferous metals, in which the arsenic cannot be detected by treating on charcoal in the reducing flame.

§ 26. *SELENIUM*—[Se].—*Presence in the Mineral Kingdom.*

It occurs most frequently in the following minerals:—

With Lead, in *Seleniuret of Lead*, or *Selen Blei*, which often affords traces of Cobalt and Iron; with Copper, in *Seleniuret of Copper*; with Silver and Copper, with some earthy matters, in *Eukaerite*; with Lead and Copper, in *Seleniuret of Lead and Copper*; with Lead and Cobalt, in *Seleniuret of Cobalt and Lead*;\* with Lead and Mercury, in *Seleniuret of Lead and Mercury*; and as a minute constituent in *Telluric Bismuth*, and in some varieties of *Swedish Lead Glances*.

*Examination for Selenium.*

The examination for selenium is so simple, that even a minute trace of it in any metallic compound may be easily recognized by the following procedure:—

A small fragment of the compound is to be ignited on charcoal in the oxidating flame, and immediately held under the nose. If the substance contains selenium, a strong disagreeable smell is given off, similar to decaying horseradishes, and which is peculiar to this metal. If much selenium is present, a reddish vapour is evolved before ignition commences, which consists of finely-divided selenium; but afterwards, a steel-grey metallic glistening sublimate is deposited on the charcoal, which, on the exterior edges, sometimes passes into violet, and in thin layers appears blue.

Selenium may also be readily separated from its compounds in its simple form, by merely roasting the assay in an open

\* [*Seleniuret of Cobalt and Lead* has much the aspect of *Seleniuret of Lead*. It gives off in the closed tube a sublimation of Selenium, and exhibits with the fluxes the reaction of Cobalt, by colouring them blue.—*Phillips.*]*—Trans.*



glass tube (see page 52), and, during the operation, inclining the tube, in order to oxidize the other elements, when this metal will sublime with a reddish colour. If the quantity of selenium present be pretty large, the part of the sublimate nearest the assay appears almost of a steel-grey; small crystals of selenic acid are sometimes deposited before the red sublimate, but they again disappear on the application of a very gentle heat. If sulphur be also present, which is the case with the seleniferous lead glances, sulphurous acid will be evolved, and may be recognized at the end of the tube either by the smell, or by moistened litmus.

If selenium occurs with tellurium, *e. g.*, Telluric Bismuth from Tellemarken, which contains seleniuret of bismuth, and if the specimen be subjected to experiment in a glass tube, oxide of tellurium is first given off, and, by continuing the blast, the selenium deposits as a red coating between the oxide of tellurium and the heated assay. As sulphuret of arsenic, however, sometimes sublimes in an open tube, like selenium, this experiment alone does not suffice; the substance should therefore be treated on charcoal, by which means it can be instantly decided whether the sublimate is selenium or arsenic.

#### (γ) EXAMINATION FOR NON-METALLIC BODIES AND ACIDS.

##### § 1. OXYGEN [O], and HYDROGEN [H], in combination as WATER [H O].—*Presence in the Mineral Kingdom.*

Under this head no regard is paid to the Water occurring in nature, as Rain, Snow, Spring, Lake, and Sea Water, &c., but to the presence of the same in Minerals, as it often forms an essential constituent.\* It also occurs in large quantities in some minerals and hydrates: it may likewise be contained in a mechanical state.

It would be superfluous here to specify all those minerals which contain Water, as this information will be found under each described metal.

\* [It is termed by Graham, Constitutional Water.]—*Trans.*



*Examination for Water.*

This examination is very simple, and is to be performed in the subjoined manner:—The substance is to be placed in a small perfectly dry glass vessel (*see Fig. 8, page 11*), and then heated over a spirit lamp. If it contains mechanically-combined water, or be a salt containing chemically combined water, it will in the former instance be entirely eliminated, and in the latter only partly. The aqueous vapour will deposit in drops on the upper part of the vessel, where it can be readily seen. If a substance, insoluble in water, contain its water chemically combined, an evolution of vapour seldom occurs on the first application of the heat; but if the vessel be introduced further into the flame, and strongly heated, the water will be liberated as in the above case.

During the examination for water, the operator should observe all phenomena that take place; because by so doing he may distinguish various substances which are analogous to others in their outward appearances, *e. g.*, many substances containing sulphuric, phosphoric, or hydrofluoric acids, give off water, when strongly heated, which reacts acid with litmus or Brazil-wood paper; and sulphates often generate so much sulphurous acid, that it can be readily recognized by its characteristic odour.

Fluorides evolve, when strongly heated, hydrofluoric acid, which attacks the glass vessel,—this occurs with fluoride of cerium.

After the water has been expelled from the arseniates, arsenious acid may sublime, as with Scorodite.

An alkaline reaction with turmeric paper, or reddened litmus paper, indicates the presence of ammonia.

Decrepitation and change of colour must also be carefully observed by the experimenter, in order to discriminate with certainty the substance under examination.

§ 2. *NITROGEN* [N], and *OXYGEN* [O], in combination as *NITRIC ACID* [NO<sup>5</sup>].—*Presence in the Mineral Kingdom.*

This acid occurs in *Native Saltpetre*, which is generally contaminated with Sulphate, Nitrate, and Carbonate of Lime,



and some Iron and Chloride of Calcium ; and in *Nitrate of Soda*, which generally contains seven to eight per cent. of Chloride of Sodium.

*Examination for Nitric Acid.*

The native salts which contain this acid, are readily recognized by detonating when ignited upon charcoal.

Substances which are infusible, containing nitric acid, must be heated to redness, after being perfectly desiccated, in a glass tube, by which means dark yellow fumes (nitrous gas) are eliminated, and are recognized by their peculiar colour and smell. If the nitric acid contained in a substance be small, and firmly united to a base, so that no characteristic fumes are obtained, a portion of the substance must be triturated in a mortar, with about an equal weight of bisulphate of potash, and the mixture then treated as previously given. The free sulphuric acid combines with the base, liberating nitric acid, and the tube becomes filled with nitrous gas, whose brownish-red, or yellow colour, is best perceived by looking perpendicularly into the mouth of the tube.

§ 3. *SULPHUR* [S] and *SULPHURIC ACID* [SO<sup>3</sup>].—

*Presence in the Mineral Kingdom and furnace products.*

Sulphur is found beautifully crystallized in nature, but is often contaminated with Bitumen, Silica, Lime, Iron, Carbon, Water, &c. It occurs most frequently in combination with Metals.

Sulphuric Acid is only met with in nature, in combination with Alkalies, Earths, and Metallic Oxides, which combinations have been previously given. All dressed ores, containing an admixture of metallic sulphurets, which are not perfectly smelted before roasting, give, when re-fused, sulphuretted metals, which are called *Stein*,—and often a small portion of such metallic sulphurets, is mechanically contained in many products which ought to be perfectly free from sulphur. Sulphur also forms partly an essential, and partly a secondary ingredient in many furnace products. It



may be considered an essential ingredient, in *Rohstein*, *Bleistein*, *Kupferstein*, *Tutty*, &c., and a secondary one, in *Workable Lead*, *Schwartz-Kupfer*, *Slags*, &c.

Sulphuric Acid occurs in many roasted ores which had previously contained Sulphur.

*Examination for Sulphur, or Sulphuric Acid.*

Native sulphur is easily recognized by submitting it to the flame of a lamp upon charcoal. It burns with a fine bluish colour, generating sulphurous acid.

Sulphuretted combinations may be detected by different methods, viz.:—

(a) Sometimes by heating the substance strongly in a glass tube. Some metallic sulphurets possess a higher degree of sulphuration; *e. g.*, Iron Pyrites give off sulphur, which deposits in the upper part of the tube. If such a combination contains at the same time arsenic, *e. g.*, Mispickel and Nickel-glance, no yellow sublimate of sulphur will be obtained, but a yellowish brown one, consisting of sulphuret of arsenic;

(b) By roasting the substance in an open glass tube. If only a small trace of sulphur is present, no sulphurous-acid smell will be perceived, but its presence may be recognized by holding a stripe of moistened litmus paper in the tube,—it will be immediately reddened;

(c) By heating the substance in the oxidating flame upon charcoal. If an appreciable quantity of sulphur be present, the sulphurous-acid smell will be immediately recognized;

(d) The most accurate method, when no selenium is present, is, melting the powdered assay (those substances that cannot be reduced readily to powder may be applied in small particles,) with two parts soda and one part borax, upon charcoal. In this treatment, sulphuret of sodium is formed, which, when moistened and applied to a piece of bright silver, blackens it. The addition of the borax is to prevent the sulphuret of sodium formed from being absorbed by the charcoal.

As the seleniurets behave exactly like the sulphurets towards metallic silver, the substance must previously be heated



*per se* on charcoal, in order to ascertain whether the characteristic horseradish odour is exhaled. When the two occur together in a substance, the sulphur examination must be first performed in the open tube.

Sulphuric acid can be detected when in combination with alkalies, earths, or metallic oxides:—

(<sup>1</sup>) By melting the substance with silicate of soda in the reducing flame. The sulphuric acid at first combines with a portion of soda, which becomes reduced to sulphuret of sodium, and the glass subsequently assumes a red or dark yellow colour, according to the proportion of sulphuric acid present. If the quantity of sulphuric acid be very minute, the colour appears only after the glass has perfectly cooled. The above method is only suitable for *colourless* sulphuric acid compounds: coloured sulphates must be submitted to the following treatment:—

(<sup>2</sup>) Smelted with two parts soda, and one part borax, on charcoal in the reducing flame, and the resulting mass moistened with water and placed on silver; a tarnishing of the metal will indicate the presence of sulphur.

§ 4. PHOSPHORIC ACID—[ $\text{PO}^5$ ].—*Presence in the Mineral Kingdom.*

This acid is always met with in nature in a combined state. The minerals in which it forms an ingredient have been mentioned under Lime, Alumina, Yttria, Copper, Uranium, Lead, Iron, and Manganese.

*Examination for Phosphoric Acid.*

If a substance contains more than from 4 to 5 per cent. of phosphoric acid, the annexed examination, (Berzelius's,) is the best. Dissolve some of the substance in boracic acid upon charcoal in the oxidating flame, and then introduce into the molten bead a piece of fine iron wire, and expose the whole to a strong reducing flame. The iron oxidizes, at the expense of the phosphoric acid, and borate and phosphate of iron are produced, the latter of which fuses in a strong heat. As the globule cools, a phosphorescence is generally remarked



on various parts of the charcoal, which proceeds from the crystallization of the phosphuret of iron. When perfectly cold, the glass must be removed from the charcoal, and broken into particles between folds of paper, upon an anvil. By this means the phosphuret of iron will separate as a globular metallic button, and can be submitted to a further examination. It should be attractable by the magnet, brittle under the hammer, and ferruginous-coloured when broken. If the substance contained only a small quantity of this acid, the globule containing the phosphuret of iron will be sensibly ductile when struck. When very little or no phosphoric acid is present, on breaking the button the iron wire will be found intact, except where it protruded beyond the assay.

If the substance under examination contained other ingredients reducible by iron, and capable of melting with it to a globule, *e. g.*, sulphuric or arsenic acid, &c., it must be examined for these, before being subjected to the above treatment.

According to Fuchs and Erdmann, phosphoric acid may be readily detected in minerals, if only three per cent. be present, in the following manner:—A splinter of the mineral is placed between the platinum points of the forceps, dipped into sulphuric acid, and then heated in a dark room with the apex of the blue flame, when the outer flame will be immediately coloured bluish green, of a perfectly different shade to that produced by boracic acid, barytes, or copper. Many phosphoric minerals, when treated *per se* in the blue flame, colour the outer flame green; *e. g.*, Wavellite.

When reactions are not obtained by the foregoing methods, it is better to have recourse to the moist way. The substance free from sulphuric acid and alumina must be fused in the oxidating flame, with four times its volume of soda, in the platinum spoon, and the residuum dissolved with water in a porcelain basin over a spirit-lamp, and then allowed to settle. Some of the clear supernatant liquor must then be decanted off, mixed with acetic acid, re-heated over the lamp, and a small crystal of nitrate of silver immersed in the menstruum. If the solution contains only a trace of phosphoric acid, a lemon-



yellow precipitate of tri-phosphate of silver subsides. Should the mixture not have been sufficiently heated, previous to the addition of the silver salt, a white precipitate of di-pyrophosphate of silver will occur, which cannot be readily distinguished from other compounds.

If the substance contains alumina, *e. g.*, Wavellite, Amblygonite, Lazulite, &c., the phosphoric acid cannot be readily detected in the manner last mentioned, because on melting the substance with soda, phosphate of alumina combines with it, and this combination dissolves out on heating with water, and, on the addition of acetic acid, deposits. The subjoined method (Berzelius's) will detect a trace of phosphoric acid in an aluminous compound. Triturate the powdered substance in an agate mortar, with a mixture consisting of 6 parts soda and  $1\frac{1}{2}$  parts silica, then melt the whole mass in the oxidating flame upon charcoal. Treat the residuum in a porcelain basin with boiling water:—the phosphate and excess of carbonate of soda is dissolved; leaving silicate of alumina and soda. If the substance contained only a little alumina, some silicate of soda will also dissolve, but this will have no detrimental influence on the detection of the phosphoric acid. The clear liquor is treated with acetic acid, &c., as recently given. If silicate of soda be contained in the solution, it precipitates on the addition of acetic acid, but will not in the slightest degree prevent the yellow reaction of the phosphoric acid with the silver salt.

When examining for phosphorus in metallic phosphurets before the blowpipe, the pulverized assay must be smelted with nitrate of potash on the platinum wire, and the residuary mass treated with soda, similar to a substance containing phosphoric acid. The phosphorus and the metal oxidize, phosphate of potash is formed, and finally, the metallic oxide separates.

§ 5. *CHLORINE*—[Cl].—*Presence in the Mineral Kingdom.*

Chlorine never occurs in a free state in nature, but in combination with other bodies.

The minerals containing Chlorine have been previously



given under Potash, Soda, Ammonia, Lime, Magnesia, Silver, Mercury, Copper, and Lead.

*Examination for Chlorine.*

According to Berzelius, chlorine is detected in metallic chlorides, in the following manner. Dissolve oxide of copper in microcosmic salt, on the platinum wire, until a diaphanous bead is obtained in the oxidating flame. The substance under examination is now to be added, and heated. If chlorine is present, the assay will be surrounded by a beautiful blue-coloured flame, inclining to purple, which disappears after some time, but can be reproduced by adding a fresh supply of the sample. Chlorine therefore cannot be confounded with any of the acids which occur in the mineral kingdom, for none of them afford the foregoing reaction; and even those that impart a colour to the flame when mixed with a copper salt, give no tinge when operated upon in a cupri-ferous microcosmic glass. A second method, which is annexed, is also given by Berzelius, for the detection of chlorine in a compound soluble in water. A small quantity of sulphate of iron or copper must be dissolved, a few drops of the solution placed upon a bright piece of silver, and the metallic chloride added; by this procedure, the silver will be blackened,—provided chlorine is present. According to Merlet, when the chloride is insoluble in water, it must, previously to being added to the solution, be fused with soda on a platinum wire. When a small quantity of chlorine is present, the spot produced upon the silver will be greyish black.

If the operator is not fortunate in obtaining a satisfactory result by the above methods, he must resort to the moist way, which will detect the *minutest* trace of chlorine. When the substance is insoluble, it must be fused with soda, as above-stated; when soluble, it is only requisite to dissolve it in *distilled water*, and to add a drop of a solution of nitrate of silver. If there be only 1 part of chlorine in 10,000 parts of the solution, a milkiness ensues.\*

\* [The last test is always preferable, unless a chemist is on a tour, and therefore has only a few common re-agents in his box].—*Trans.*



§ 6. *BROMINE*—[Br].—*Presence in the Mineral Kingdom.*

Bromine occurs as a small ingredient in nearly all *Salt Springs*, as Bromide of Sodium and Magnesium.

*Examination for Bromine.*

According to Berzelius, metallic bromides give, with a cupreous microcosmic bead, and also with sulphate of copper on a silver plate, the same reactions as metallic chlorides; but the blue colour which the flame assumes does not incline to purple, but becomes green, especially on the edges. To distinguish the two with certainty, the substance under examination must be fused with bisulphate of potash in a small glass flask, with a long neck, over the spirit-lamp. Bromine and sulphurous acid will be eliminated; the former fills the vessel with a yellow gas, readily recognized by its characteristic odour, although sulphurous acid be present.

In examining for bromine in salt springs, a considerable quantity of the liquid must be evaporated, until nearly the whole of the chloride of sodium is separated. The mother liquor is then poured off, evaporated to dryness, and the residue examined with bisulphate of potash in a glass flask. In this case, however, iodine is generally liberated, and therefore the pure yellow of bromine is not obtained, as it acquires a somewhat violet tinge, from the presence of iodine.\*

§ 7. *IODINE*—[I].—*Presence in the Mineral Kingdom.*

Iodine occurs in *Rock Salt* (also in *Salt Springs*) in very small quantities, and generally in combination with *Sodium*. It has also been detected in a Mexican mineral, combined with Silver, and in a Silesian one, in combination with Zinc.

Its presence in the vegetable kingdom does not belong to

\* [It requires a very scientific operator to detect, by the preceding methods, the presence of bromine, when combined either with chlorine or iodine; I should, therefore, recommend those who wish to arrive at an accurate result, to consult Fresenius's work, and proceed according to the moist methods which are there given.]—*Trans.*



this work; but I may mention that it is likewise found, in combination with Sodium, in most kinds of *Sea-weed*, &c.\*

*Examination for Iodine.*

According to Berzelius, the metallic iodides, when treated with cupriferos microcosmic salt, impart a beautiful and deep green tinge to the blowpipe-flame, which cannot be confounded with the colour given when the bead, *per se*, is fused in either flame. Metallic iodides, melted with bisulphate of potash in a glass tube, eliminate iodine vapour, which displays a beautiful purple colour. Sulphurous acid is also set free.

Mineral waters can be examined for iodine in the same manner as for bromine; the presence of the former is indicated by the violet-coloured vapour. If an appreciable quantity of bromine be present, the gas evolved will be of a darkish yellow colour.†

§ 8. *FLUORINE*—[F].—*Presence in the Mineral Kingdom.*

Fluorine is always found in combination. The minerals in which it forms an essential or secondary ingredient, have been cited under Soda, Lithia, Lime, Magnesia, Alumina, Yttria, and Cerium.‡

*Examination for Fluorine, or for Hydrofluoric Acid.*

Hydrofluoric acid is not readily eliminated by heat, from substances in which fluorine forms an essential ingredient;

\* [In some of these productions, such as the *Fucus Serratus* and *Fucus Digitatus*, it exists ready formed, and, according to Fyfe, (*Edin. Philos. Journal*, i. 254), may be separated by the action of water; but in others it can only be detected after incineration.—*Liebig's Turner.*]—*Trans.*

† [A good test for Iodine is nitrate of silver. The iodide of silver is of a pale yellow colour, (the chloride is white,) and is *very difficultly* soluble in ammonia. The chloride dissolves in ammonia with great facility. The moist way must never be resorted to, unless the dry one proves unsatisfactory.]—*Trans.*

‡ [Traces of Fluorine have been found in the teeth, and also in the bones, of different animals.]—*Trans.*



*e. g.*, Fluor-Spar, Topaz, Cryolite, &c., as from those in which it sometimes occurs as a contingent one; *e. g.*, in Mica and Hornblende, when it is expelled generally as fluo-silicic acid.

If a small quantity of hydrofluoric acid be held in feeble combination, in a mineral containing water, it is necessary to heat it in a test tube, and apply a strip of moistened Brazil-wood paper to its mouth. As the temperature augments, fluo-silicic acid will be evolved, a siliceous circle formed at a short distance from the assay, and the Brazil-wood paper tinged straw-yellow, by the hydrofluoric acid.

According to the experiments of Bonsdorf, Brazil-wood paper is likewise coloured straw-yellow, by phosphoric and oxalic acids, but not by sulphuric, nitric, arsenic, or boracic acids. As phosphoric acid, however, is not volatile in such an examination, the tinge acquired by the paper will indicate the presence of fluorine. This test is so delicate, that it is only necessary, at times, to moisten the compound fluoride with hydrochloric acid, on a watch glass, and then to apply the reagent-paper. When a substance under examination for fluorine, produces no etching upon the glass, and affords no reaction with the Brazil-wood paper, the following method of Berzelius's must be adopted:—Mix the assay with metaphosphate of soda, (formed by heating the micro-cosmic salt to incipient redness,) and heat the mass in the end of an open glass tube, in such a position that there will be an access of hot air from the flame; by this procedure aqueous hydrofluoric acid is formed, which can be recognized by its smell, being more suffocating than chlorine, and also by the etching produced by the condensation of vapour in the tube. By applying moist Brazil-wood paper to the extremity of the tube, it will be instantly coloured.

The subjoined is Merlet's method for the detection of this acid:—Pulverize the substance for examination, then triturate it to an impalpable powder, and mix it with an equal part of bisulphate of potash. Heat the mass gradually in a moderately-wide dry test tube. The judicious application of heat must be strictly observed, for if the operator heats first the part of the tube where the assay rests, the whole may be



lost, on account of the glass being shattered. The spirit-flame must first be applied to the fore-part of the tube, and then made to recede slowly, until it fuses the assay. After the mixture has been for some time kept in a molten state, the lamp must be withdrawn, and the part containing the assay severed with a file. The fore-part of the tube must then be well washed, and afterwards dried with bibulous paper. Should the fluorine contained in the substance be significant, the glass tube, when held up to the light, will be found to have lost its transparency, and to be very rough to the touch.\*

§ 9. *CARBON*—[C]—and *CARBONIC ACID*—[CO<sup>2</sup>].—*Presence in the Mineral Kingdom.*

It occurs in nature in a pure state, as *Diamond*; and, in combination with other bodies, in *Anthracite*, *Graphite*, *Stone Coal*, *Brown Coal*, &c.

Carbonic acid occurs in minerals combined with bases which have been given in former sections of this work; it is also met with in the gaseous form, in mines and volcanic regions, and places where the earth contains large quantities of Carbonate of Lime; but as the examination of free gases does not come within the province of the Blowpipe, I need not enumerate the different places where this gas is exhaled.

*Examination for Carbon and Carbonic Acid.*

The diamond is so difficult to burn, that the ordinary blowpipe is not sufficiently powerful.† Some varieties of charcoal,

\* [Of all known substances, hydrofluoric acid is the most destructive to animal matter. When a drop of the concentrated acid comes in contact with the skin, instantaneous disorganization ensues, and deep ulceration of a malignant character is produced. On this account, the greatest care is requisite in its preparation. It acts energetically on glass; its transparency is instantly destroyed, heat is evolved, the acid boils, and in a short time entirely disappears. A colourless gas, commonly known by the name of *fluo-silicic acid gas*, is the sole product. This compound is always formed when hydrofluoric acid comes in contact with a siliceous substance.—*Liebig's Turner, page 376.*]—*Trans.*

† [The diamond is remarkably indestructible, and may be heated to whiteness, in a covered crucible, without injury; but it begins to burn, in



when ignited with anhydrous alum in the oxidating flame, leave a recementitious mass, consisting of ferruginous and earthy particles. When they are pulverized, mixed with nitrate of potash, and heated strongly in a platinum spoon, they detonate, and afford carbonate of potash.

The presence of carbonic acid is detected by the gas being eliminated with effervescence on heating a mineral containing it before the blowpipe. It is also readily detected by moistening the substance with a few drops of hydrochloric acid—effervescence likewise ensues.

§ 10. *BORACIC ACID*—[ $\text{B O}^3$ ].—*Presence in the Mineral Kingdom.*

This Acid occurs combined with Water in *Native Boracic Acid*; also in several minerals in combination with various bases. Those in which it forms an essential or secondary ingredient have been already adverted to under Soda, Lime, Magnesia, and Alumina.

*Examination for Boracic Acid.*

According to Berzelius, Native Boracic Acid of Tuscany produces the following reactions:—Moistened with water and placed on Brazil-wood paper, it bleaches it in about half an hour; a solution in alcohol applied to curcuma paper, imparts a brown tint.

*Per se*, on charcoal, it melts to a clear glass; should it contain gypsum, the assay will be opaque when cold.

Turner has recommended the following method for the detection of boracic acid in salts and minerals. Reduce the substance to an impalpable powder, and mix it with a flux consisting of  $4\frac{1}{2}$  parts of bisulphate of potash, and 1 part of finely-powdered fluor spar. Knead the whole into a paste, with a few drops of water; then place the moist assay on the

the open air, at about the melting point of silver ( $1873^{\circ}$ ), and is entirely converted into carbonic acid gas. It is more quickly consumed in fused nitre, when the carbonic acid is retained by the potash: this is a simple mode of analyzing the diamond, by which it has been proved to be perfectly pure carbon.—*Graham.*—*Trans.*



platinum ring, and fuse the mass with the apex of the blue flame. During the fusion of the assay, fluo-boric acid gas is liberated, which communicates a pure green colour to the outer flame. Great care must be taken, when only a small quantity of boracic acid is present, for the colouring will be momentary. Merlet remarks that the reaction will be more certain, when 3 to 4 parts of the flux are used with 1 of the substance.

§ 11. *SILICIC ACID*—[Si O<sup>3</sup>].—*Presence in the Mineral Kingdom, and in Furnace Products.*

Silicic acid forms a principal ingredient of the solid parts of our globe. It is found in nearly a pure state, in *Berg-Crystal, Quartz, Chalcedony, Hornstone, Cornelian, Flint, &c.* When any of the preceding minerals are coloured, it is generally owing to the presence of Iron or Manganese. The native silicates have been cited under the Earths and Metals. Lastly, it is considered as an essential ingredient of dressed Ores and Slags.

*Examination for Silicic Acid.*

The Berg-Crystal and Milk-Quartz comport themselves with the fluxes, like pure silicic acid. The other varieties of Quartz, as regards their solubility in the fluxes, behave in the same manner; but the colours imparted to the borax or microcosmic glasses by the metallic oxides, are more or less visible, according to the one contained.

The silicates are recognized by means of microcosmic salt and soda. They are decomposed in such a manner by the former, that their bases combine only with the free acid in the flux, and the silicic acid separates. This examination is generally performed on a platinum wire. The microcosmic salt must first be fused into a bead, the silicate added, and the whole treated in the oxidating flame. The glass bead, while hot, is clear, and the separated silica floats through it in a collected state. As glasses are obtained by numerous silicates, which are perfectly clear while hot, but opaline on cooling, the operator must carefully observe the flux in its



molten state, for the presence of silica. The siliceous appearance is generally observable with those compounds of silicic acid which have lime, magnesia, glucina, yttria, or oxide of zinc, for the base.

Silicates of Zirconia, as Hyacinth and Zircon, are with great difficulty acted upon by microcosmic salt. The preferable method for the detection of the acid in these minerals has been given under Zirconia. If a substance contains a *small* quantity only of silicate, (or be intermixed with some quartz,) a glass is obtained in which no skeleton of silica appears, owing to the solution of the silicic acid; it can be detected, however, by the aid of the moist way, proceeding in the manner given in the examination of the different Earthy Silicates. The silicic acid is separated in such a condition by this method, that it can afterwards be recognized with a little microcosmic salt on a platinum wire, or with soda on charcoal.

The silicates dissolve imperfectly in soda on a platinum wire or charcoal, with effervescence. If it be a compound in which the oxygen of the silicic acid is at least double that of the base, a clear glass will be obtained by the judicious application of soda, which will remain the same when cold, because the glass of silicate of soda acts as a solvent on the free bases.

A silicate containing an infusible base, gives a clear glass with *a little* soda. With a slight addition more, the glass is opaque; and with another supply, the assay becomes intact.

By the first addition of soda, a part of the base separates, which is re-dissolved, and the glass is clear; but when a large addition of the flux is supplied, so much of the base separates that the whole assay becomes infusible.

Substances in which only a small quantity of a silicate is contained, which is not at all detectible by soda, must be treated in the moist way. This method also must be resorted to in the examination of dressed ores (often containing metallic sulphurets and arseniurets) for silicic acid.



¶ *Examples of the Method of Procedure employed in  
examining different Compounds for all their  
Constituents, with the aid of the Blowpipe.*

(a) SALTS.

Salts come under the following heads:—

- (a) Those which have Alkalies, for their bases;
- (b) Those which have Earths;
- (c) Those which have Earths and Alkalies together;
- (d) Those which have Metallic Oxides; and
- (e) Those which have Metallic Oxides and Earths together.

These different salts can often be distinguished from each other by their external characters, and thus immediately recognized; cases, however, occur which cannot be so easily determined, and recourse therefore must be had to chemical analysis.

Such an examination can be easily performed with the blowpipe, and the whole operation completed in a short time, if the proper method be undertaken; which will be shewn in succeeding examples.

1. *Sulphate of Potash.*

If this salt be in the state of powder, it is not possible to determine from external appearances whether the base is an earth, an alkali, or a metallic oxide; the examination should therefore be conducted in the following manner:—A small portion of the salt is heated in a bulb tube, and examined in order to see if it yields water; if it be employed in a crystalline state, it decrepitates, but yields none. The pulverized salt is next mixed with soda, this mixture fused on platinum foil in the oxidating flame, and examined during the fusion, in order to see if the whole mass fuses clear, or if any thing is precipitated. Sulphate of potash fuses with soda on platinum foil to a perfectly clear glass; the base, therefore, is an alkali. The particular alkali is determined by examining how the exterior flame is coloured when a small fragment



adhering to the moistened loop of the platinum wire, is fused at the point of the blue flame. In this case, it is at once seen that the exterior flame exhibits a violet tinge, which shews the presence of potash.

If the acid also is to be determined, a small portion must be treated on charcoal in the reducing flame. The salt under examination fuses, is absorbed by the charcoal, which it coats with a white substance, formed of a number of orbicles, similar to that produced by sulphate of potash, or a combination of potassium, with chlorine, bromine, or iodine. If the mass absorbed by the charcoal be moistened with a drop of water, it produces a strong hepatic odour and becomes black, when removed from the charcoal and placed on a slip of silver-foil moistened with water.

By this simple examination, it is ascertained that the constituents of this salt are sulphuric acid and potash.

### 2. *Nitrate of Potash (Saltpetre).*

If the salt cannot be recognized by its external characters, the examination is conducted in the same manner as the preceding.

Heated in a bulb tube, it fuses readily, giving a clear glass, and yields no water. By this test it is already known that the base is an alkali; the test with soda on platinum foil may consequently be omitted.

Kept in a state of fusion on platinum wire, it colours the exterior flame violet. The base is consequently potash.

Ignited on charcoal, its surface becomes exceedingly vesicular. The acid is therefore nitric acid.

### 3. *Carbonate of Soda.*

In the examination of this salt, the procedure in the commencement is similar; but it must be further treated, because the acid cannot be thus recognized.

The hydrated salt yields water in a bulb tube, which reacts neither acid nor alkaline.

It fuses to a clear glass with soda on charcoal. The base is consequently an alkali.



It fuses readily on platinum wire, and tinges the exterior flame strongly yellow. The base is consequently soda.

It melts on charcoal without detonating, is absorbed by the charcoal, but gives no sublimate. It is consequently not in combination with nitric, sulphuric, or muriatic acids.

It reacts alkaline with turmeric, or with reddened litmus paper.

It is now to be determined whether the soda is combined with carbonic acid. For this purpose, a small specimen of the salt should be fused with silicic acid on charcoal, and examined in order to see if the two substances combine to a clear glass with effervescence. This is the case with carbonate of soda, because its acid is evolved while the soda combines with the silicic acid. If the glass becomes of a yellow or a red colour on cooling, the carbonate of soda is not free from an admixture of sulphuric acid.

#### 4. *Sal Ammoniac (Chloride of Ammonium).*

In testing for water in a bulb tube, if the salt sublimes without leaving a residue, it must be an ammonia salt. If it be then tested with soda, for ammonia, and with a micro-cosmic salt bead saturated with oxide of copper, for chlorine, results are obtained, which can be obtained with chloride of ammonium only.

#### 5. *Potasso-Alum.*

When this salt is pulverized, it cannot be recognized as alum, unless from the taste; nor can we determine, from its external character, whether the base is an alkali or an earth, or whether both act as a base. The operation must therefore be proceeded with in a manner similar to that given for sulphate of potash.

In a bulb tube, alum fuses, tumefies, and yields water, which reacts neither acid nor alkaline. The dry mass ignited, yields an acid which reddens moistened litmus paper placed in the upper part of the tube. By further examination of the salt, this acid is determined.

It forms an infusible compound with soda on platinum



foil, when its water is driven off. The base, therefore, appears to be an earth. But if a small portion be treated *per se* on a platinum wire at the point of the blue flame, the exterior flame will be coloured deeply violet, from potash. So far it is thus known that the salt has two bases, of which the earthy one remains to be ascertained.

As the salt appears perfectly white after the removal of its water, it should be tested with solution of cobalt; when it becomes coloured of a beautiful blue, if, in separating the water from the substance on charcoal, a pure oxidating flame has been employed. This shews the base to be alumina.

The acid only remains now to be determined. It is first tested for sulphuric acid, because in testing the salt in the bulb tube, litmus paper was reddened. When fused with soda on charcoal in the reducing flame, the fused mass, when laid on silver and moistened with water, produces a black spot of sulphuret of silver. The acid is consequently sulphuric: separate tests for other acids produce no reaction; the salt, therefore, consists of sulphate of alumina and sulphate of potash.

#### 6. *Epsom Salt (Sulphate of Magnesia).*

The constituents of this salt are recognized in the same manner as those of alum.

In a bulb tube, it yields a large quantity of water, which does not react acid.

It is infusible with soda on platinum foil; the base, therefore, appears not to be an alkali.

The anhydrous salt, held with the forceps in the blue flame, tinges the outer flame, sometimes feebly, but evidently yellow, from an admixture of soda.

Solution of cobalt communicates to it a beautiful, but feeble rose tint. The base is therefore magnesia.

When the mass is fused with soda, on charcoal in the reducing flame, it produces, when laid on silver foil and moistened with water, a black stain of sulphuret of silver. It produces with soda and silicic acid, the peculiar hepatic tint. The acid is therefore sulphuric, and the salt consists of sul-



phate of magnesia, which is sometimes contaminated with a little sulphate of soda.

#### 7. *Celestine.*

Crystallized Celestine decrepitates strongly, when heated in a bulb tube, but yields no water. The test with soda on platinum foil, may be dispensed with, as this substance is instantly recognized as an earthy salt. It should be proved, however, whether it tinges the outer flame. A fragment held in the forceps, and treated with the point of the blue flame, fuses, and communicates a reddish tint to the exterior flame. The colour (which is much clearer than that given by a salt of lime,) is similar to that produced by lithia, but not so beautiful. This shews the presence, therefore, of lithia or strontia.

A specimen is next treated with soda on charcoal, in order to ascertain if it is absorbed with the flux by the charcoal. It fuses with it, the compound undergoes a strong ebullition, and sinks into the charcoal, as a strong hepatic mass. By this means it is at once determined, that the salt is either sulphate of barytes, or sulphate of strontian; but as it has been already found to tinge the outer flame red, and also that it is not very difficultly-fusible, it may be considered as almost certain, that it is not Heavy-Spar, but Celestine.

If a specimen be ignited for a long time, on charcoal in the reducing flame, the sulphuret of strontium so formed, treated with hydrochloric acid, and the resulting salt evaporated to dryness, and treated with alcohol, (as has been already more fully given under the head of Examination for Strontian,) for the presence of strontia, is recognized with certainty, by the red colour of the alcoholic flame.

#### 8. *Calcareous Spar.*

This salt is readily recognized from its external character. The method of procedure is similar to the foregoing.

Heated in a bulb tube, it does not yield water.

Held in the forceps, and strongly heated in the apex of the blue flame, it does not melt, but becomes luminous, and tinges the outer flame a feeble carmine red. During the refriger-



ation, it generally becomes of a blackish-green colour, which shews the presence of iron or manganese.

The ignited fragment, when moistened, and laid upon litmus paper, reacts alkaline.

From this behaviour, the conclusion may be drawn, that the substance under examination is an alkaline earth, and this earth, lime, in combination with carbonic acid.

A fragment should be dissolved, next, on the platinum wire, with borax; to ascertain if it effervesces, or not, during solution, and to notice the colour communicated to the borax glass. Calcareous Spar dissolves with effervescence, the carbonic acid escaping, and the glass has generally a feeble iron tinge. If a larger proportion be dissolved, the glass crystallizes during the refrigeration, presenting a number of sharp edges. By this means, it has now been discovered, that the salt is a carbonate, containing a little oxide of iron, and that the base apparently is lime. A separate test for manganese, determines whether the salt under investigation may not probably contain this metal also.

When a portion is treated with soda on charcoal, it is not dissolved; the soda is absorbed by the charcoal, and an infusible mass remains, which may be considered as lime.

#### 9. *A doubtful Mineral.*

This mineral occasionally occurs in a locality not far from Freiberg, on the sides of small lodes, accompanying various silver ores, particularly Dark Ruby Silver, and also with Iron Pyrites, in druses on gangues,—as an apparently changed natural product, of an hexagonal prismatic form, and a colour partly yellowish-white, partly reddish-yellow; its fracture is almost earthy. Breithaupt calls it *Pseudo-Apatite*.

Many of the crystals, particularly the dark coloured, when ignited in a bulb tube, decrepitate, and yield a small quantity of water only.

The fragments remaining in the bulb tube, vitrify on the edges only, when intensely heated at the apex of the blue flame,—strongly phosphorescing at the same time, and when cold, appearing of a lighter colour. During the heating, the



exterior flame is coloured deeply yellow, from the presence of soda; with careful observation, however, a dark-green tinge may be evidently observed, shewing the presence of phosphoric acid. If the fragment held in the forceps, be moistened with sulphuric acid, and then heated, the green tinge of the exterior flame becomes more decided, and, notwithstanding the yellow colour of the soda, may be recognized as the reaction of phosphoric acid. It yields a regulus of phosphuret of iron, with boracic acid and iron.

From the preceding investigation, it may be seen that the salt is a phosphate. Its behaviour to vitreous fluxes and cobalt solution, should now be examined, in order to be able to come to a conclusion as to the remaining constituents, particularly the bases.

This mineral dissolves slowly in borax, forming a glass, which sometimes becomes streaked with opalescent lines, when a moderate quantity of the mineral is present; with a still larger quantity, the glass becomes turbid during refrigeration. The reddish-yellow crystals give with borax a yellowish tinge, from oxide of iron.

It readily dissolves, with a slight effervescence, in microcosmic salt, and yields a glass coloured by oxide of iron, which becomes turbidly streaked only when a large quantity of the substance is added; when nearly saturated it becomes, *per se*, during refrigeration, turbid, and exhibits imperfect facets. When perfectly saturated, an almost milk-white bead is obtained.

With an equal weight of soda, the powdered mineral fuses on charcoal to a bead which, under a continued blast, becomes infusible, and loses its orbicular form. A larger addition of soda is absorbed by the charcoal. If another portion of the powdered mineral be fused with soda on charcoal in the reducing flame, and the fused mass laid on a moistened silver plate, a black stain of sulphuret of silver is produced.

When fused with soda and saltpetre on a platinum foil, it gives no reaction; manganese, therefore, is absent.

Cobalt solution communicates a grey colour to the powdered mineral.



From the behaviour to borax, microcosmic salt, soda, and solution of cobalt, it can be seen that the mineral contains, besides phosphoric acid, carbonic acid (because it dissolves in microcosmic salt with effervescence), and sulphuric acid (because, when fused with soda, it blackens silver). It may also be seen that the colouring ingredient is oxide of iron, and that the base is not alumina, as it gives a grey colour instead of a blue, with solution of cobalt.

If these blowpipe reactions be compared with those of other combinations of phosphoric acid which occur in nature, it will be found that they resemble most those of *apatite*.

We know, however, that *apatite* is a combination of phosphate of lime and chloride of lime; the latter being often replaced by fluoride of calcium. It is therefore necessary that the mineral should be directly examined for chlorine, hydrofluoric acid, lime, and other bases.

As we have already seen, (under Examination for Chlorine), no certain reaction for chlorine can be produced in the dry way. If, however, it be treated with nitric acid, it readily dissolves, with effervescence; if this solution be diluted with distilled water, it will be rendered slightly turbid on the addition of nitrate of silver; consequently, it is not perfectly free from chlorine.

When fused in a glass tube, closed at one end, with an equal quantity of bisulphate of potash, faint marks are produced on the surface of the glass, at a small distance from the mixture, which remain when the closed end is cut off, the tube washed with water, and dried. The mineral must therefore contain hydrofluoric acid.

The test for lime and other earths must be performed with the help of the moist way. It, may, however, be considered nearly certain, that the principal constituent is lime, as the mineral forms during refrigeration, when fused with microcosmic salt, imperfect facets; an admixture of other earths can only be detected, however, by recourse to the moist way.

For this purpose, a small portion must be treated with hydrochloric acid, and the earths precipitated as described under



Lime, and more especially under Apatite. The mineral readily dissolves with the production of heat and evolution of gas, (from the escape of carbonic acid); the solution, when diluted with water and spirits of wine, gives, with dilute sulphuric acid, an evident precipitate, which affords the reaction of gypsum before the blowpipe, after beingedulcorated; the solution, filtered from the gypsum, gives, with ammonia, a slight gelatinous precipitate, which, on further examination, behaves as ferruginous alumina.

The mineral contains, therefore,—

Phosphoric acid, a large quantity;

Carbonic acid, in smaller quantity;

Hydrofluoric acid, }  
Sulphuric acid, } in still smaller proportions;

Chlorine, very little;

Lime, a large quantity;

Alumina, in small quantity;

Soda, apparently only in small quantity;

Oxide of iron, as colouring matter;

Water, a little, (probably only mechanical).

These constituents consequently shew that the problematical mineral is a combination of phosphate of lime with phosphate of alumina, carbonate of lime, sulphate of lime, fluoride of calcium, and chloride of calcium.

#### 10. *Phosphate of Lead.*

This mineral is readily distinguished, when in the solid state, by its external characters. In this case, it is only necessary to subject it to direct examination for its known constituents. If it be in the state of powder, however, it may be easily confounded with other minerals of the same colour, and it should therefore be examined in the following manner:—

It should be first tested on charcoal in both flames, and its fusibility, and other characters from which a conclusion may be drawn, examined. The powder fuses to a bead on charcoal in the oxidating flame, which crystallizes during refrigeration, and becomes of a dark colour. If any substance



is observed to volatilize during the fusion, and deposit itself in part on the charcoal, as a white sublimate which admits of being driven farther from the assay on the application of either flame, antimony is probably present. A separate test for antimony need not, however, be undertaken on this account, as other compounds may be present which would yield a white sublimate, and which may be accidentally discovered during the course of investigation. If the bead be treated long enough with a strong blast in the reducing flame, the charcoal will be seen to be yellow, as, when coated with oxide of lead, and if the blast be discontinued, the bead crystallizes almost instantaneously, during the refrigeration, large facets of an almost white colour, similar in brilliancy to mother-of-pearl, being formed. As this appearance is peculiar to phosphate of lead, and as a sublimate of lead was already obtained on the charcoal, it is not necessary to test farther for the presence of phosphoric acid and lead. It may however be done, in order to be perfectly convinced.

A globule of phosphuret of iron and metallic lead may be readily obtained with boracic acid and iron, and a button of lead with soda on charcoal, which may be tested for silver by cupellation on bone ashes.

The appearance of a white sublimate when the mineral is treated *per se* on charcoal, is readily explained by the fact, that the natural combinations of phosphoric acid with oxide of lead, always contain chloride of lead, which volatilizes. A separate test for chlorine with a microcosmic salt bead saturated with oxide of copper, shews very clearly that the mineral contains chlorine.

#### 11. *Nickel Ochre.*

This mineral may be easily determined to be a metallic salt. Its constituents are also readily recognized during the course of the examination.

It yields some water when heated *per se* in a bulb tube, its colour becoming darker. Its behaviour *per se* on charcoal is then to be examined. It sometimes diffuses an evident alliaceous odour when treated in the oxidating flame, and by



treatment in the reducing flame it fuses, with the escape of some arsenic, to a black brittle metallic button, which is readily attracted by the magnet.

From this property, and from the mineral possessing a green colour, it may be considered with a degree of certainty that it principally consists of arseniate of oxide of nickel.

But as natural arseniate of nickel is not always free from oxide of cobalt, oxide of iron, and sulphuric acid, it should be tested with borax, and a separate examination made for sulphuric acid.

It dissolves readily in borax, and yields a glass which is violet while hot, and reddish brown when cold, (oxide of nickel). If this glass be treated on charcoal in the reducing flame until all the nickel is precipitated, the glass will appear perfectly colourless when the mineral is free from cobalt and iron; if not free from these, it will appear blue from cobalt, or green from iron. The blue glass may, however, contain iron also; it is therefore necessary to fuse it, after being freed from reduced metallic particles, in the loop of a platinum wire, treat it for some time in the oxidating flame, and carefully observe whether it appears green or blue while hot; if the former is the case, it is not free from iron.

It is exceedingly difficult to separate completely particles of reduced nickel from a glass from which the nickel has been thrown down in the metallic state; this is, however, easily effected if the precipitated nickel be melted with a particle of lead, and the glass treated in the reducing flame only. The nickel thus combines with the lead, forming a readily-fusible alloy, from which the glass may be removed while in the molten state.

The examination for sulphuric acid is made by fusion with soda and borax on charcoal in the reducing flame, and the fused assay treated on a moist piece of silver.

#### 12. *Wolfram.*

The behaviour of this mineral before the blowpipe, as well *per se* on charcoal, as with fluxes, must be first determined, in order to be able to recognize the principal constituents,



unless the mineral is recognized from its physical character as wolfram.

It decrepitates when heated in a bulb tube, and gives very little water.

Its fusibility must then be examined. This operation is most conveniently performed in the forceps, in order to be able to observe at the same time, whether the exterior flame is coloured. It fuses with difficulty to a bead, but does not tinge the exterior flame in the slightest degree. The surface of the fused parts appear crystalline iron-grey, and of a metallic lustre. It fuses difficultly to a bead, on charcoal, without producing a sublimate; the surface of the bead presents small eminences, from a number of considerable-sized laminated crystals.

The mineral should now be tested with borax and microcosmic salt.

Borax dissolves it in the oxidating flame, without difficulty, to a clear glass, which is reddish-yellow while hot, when the quantity present is extremely minute, and pure yellow during the refrigeration, as when coloured with oxide of iron; from a larger quantity, it appears blood-red while hot, and reddish-yellow under refrigeration. Treated for a few moments in the reducing flame, the tint becomes lighter, showing the colour to be derived from oxide of iron only.

It is readily dissolved by microcosmic salt in the oxidating flame, to a clear glass, which appears reddish-yellow while hot, and somewhat lighter during the refrigeration; its appearance, however, shows only the presence of oxide of iron. If the glass be treated in the reducing flame, it becomes dark red; even when the quantity present is moderate, it becomes opaque. If a small quantity of the saturated glass be treated with tin on charcoal, for a few moments, in the reducing flame, it will assume a green colour when cold. If such a glass be exposed for some time to a strong blast in the reducing flame, the green colour disappears, and a slight reddish-yellow tint remains behind, which undergoes no further change.

From these reactions with borax and microcosmic salt, we



may conclude that, as the borax glass assumes a redder tint after being treated with the oxidating flame, and a more or less yellow colour, when treated for a short time in the reducing flame, the mineral must contain manganese as well as iron; and as the microcosmic salt bead becomes dark red, when treated in the reducing flame, and assumes a green colour on the addition of tin, which becomes reddish-yellow when exposed for some time to the reducing flame, tungsten must be present, as well as iron and manganese.

A test with soda on platinum foil, shews the presence of a considerable quantity of manganese.

If the powdered mineral be fused with soda in a platinum spoon, the fused mass dissolved in water, and hydrochloric acid added to the clear solution, a white precipitate is produced, which, on heating the whole, becomes of a beautiful lemon-yellow colour, and may be recognized as tungstic acid.

The constituents of Wolfram, therefore, are; tungstic acid, protoxides of iron and manganese, and a little water. The colour of the mineral shows that the iron and manganese are present as protoxides, and not as peroxides.

#### ( $\beta$ ) ALUMINATES.

As the number of aluminates which occur in nature is very limited, and as we have already treated of the most of them, when speaking of the examination for Magnesia and Alumina, a single example will suffice.

##### *Spinelle (red) from Ceylon.*

The first thing to be done, is to ascertain its behaviour before the blowpipe, both *per se*, and with fluxes.

Heated strongly in the apex of the blue flame while held in the forceps, it loses its form, but communicates no colour to the exterior flame. If the thoroughly-ignited fragment be viewed with reflected sun-light, it appears almost black and opaque, while hot, but becomes chrome-green during refrigeration, then almost colourless, and when perfectly cold, it assumes its red colour, unaltered. This change of colour shows the presence of chromium.



With borax it dissolves with difficulty, even in a powdered state, to a clear, slightly yellowish, green-coloured glass, which does not become "turbidly streaked."

It is dissolved by microcosmic salt with difficulty when in fragments, but fuses readily and perfectly when it is employed in the state of a powder, to a clear glass, which has a reddish tint while hot, but during the refrigeration it becomes evidently, though slightly, of a chrome-green.

It is not dissolved by soda, but fuses to a vesicular mass without depositing a sublimate on the charcoal. Fused with soda on platinum foil, the presence of traces of manganese may be detected.

Solution of cobalt communicates a blue colour to the fine powder.

From this behaviour, we may conclude that, as the mineral dissolves with difficulty in borax, without producing a glass streaked with turbid lines; as, by solution in microcosmic salt, no skeleton of silicic acid remains behind; as it does not dissolve when treated with soda, no sublimate being at the same time produced; and as the powder receives a blue colour from cobalt solution;—the mineral under examination is an aluminate, not a silicate, which contains neither oxide of lead nor zinc.

As the aluminates readily admit of decomposition, by fusion with soda and borax on charcoal, (as has been mentioned when speaking of the examination of such combinations for Magnesia,) the alumina and magnesia, and an unimportant constituent, the silicic acid, may be recognized with certainty, in the spinelle from Ceylon, by further treatment of the fused mass.

#### ( $\gamma$ ) SILICATES.

The Silicates can be divided into:—

- (*a*) Monobasic; and,
- (*b*) Polybasic Silicates.

The former may be readily recognised as silicates before the blowpipe; and the base, as it consists of one earth or of one metallic oxide, may often be recognized at the same time.



The latter can also be recognized as silicates by their behaviour before the blowpipe; but the bases do not always admit of being so readily recognized.

In cases where the bases do not admit of being ascertained with accuracy during reactions before the blowpipe, recourse must be had to the moist way, and the compound decomposed either by fusion with bisulphate of potash in a platinum spoon, or by fusion with soda and borax on charcoal. The first method answers very well with monobasic silicates, (silicates of alumina excepted); the second may be employed with more advantage, with polybasic silicates.

#### 1. *Tabular Spar.*

Heated on a bulb tube, it undergoes no change; it yields, however, a little water.

Held in the forceps, it communicates no tinge to the exterior flame, and fuses on the edges only to a semi-clear glass.

It is readily dissolved in large quantities to a clear glass, which does not become streaked with turbid lines.

It is dissolved by microcosmic salt to a clear glass, leaving a siliceous skeleton behind. This glass, when moderately saturated, becomes, during refrigeration, opalescent.

With an equal weight of soda, the powder fuses to a vesicular glass, which, on the addition of more soda, tumefies and becomes infusible.

Solution of cobalt renders it difficultly fusible, producing a blue colour on the fused edges only.

From these results, we may come to the conclusion that Tabular Spar, as it leaves behind a skeleton of silica when treated with microcosmic salt, is a silicate; and that the base, as the mineral readily dissolves in borax, and as solution of cobalt communicates a blue colour to it only where it fuses, must be lime.

To prove this fact more fully, it is only necessary to fuse the powdered silicate with bisulphate of potash, and treat the fused mass in the manner already described under Lime.



2. *Soap Stone.*

Heated *per se* in a bulb tube, it yields little water, gives off an empyreumatic odour, and becomes black.

Heated in the oxidating flame with the forceps, it becomes white, contracts slightly, and fuses on the thinnest edges to a white enamel. It does not tinge the exterior flame.

Borax readily dissolves it to a clear glass, which generally possesses a slight ferruginous tinge.

Microcosmic salt decomposes it, leaving a residue of silica. The clear colourless glass, thus produced, crystallizes on cooling.

With a certain quantity of soda, it fuses to a clear glass, which becomes turbid when either too much or too little is employed.

Cobalt solution communicates to it a reddish colour, when it is exposed for a sufficient time in the state of powder to the oxidating flame.

From these reactions it is quite apparent that Soap Stone is a silicate of magnesia.

As this mineral, however, sometimes contains a little alumina, which cannot be detected by the aid of the dry way alone, the finely-pulverized soap stone should be fused with bisulphate of potash, in order to be certain that alumina is present, and the fused mass decomposed in the manner given under the head "Examination for Magnesia."

3. *Pyrosmalite.*

Heated *per se*, in a bulb tube, it undergoes no change, but yields a little water.

In the forceps it fuses on the edges only, to a black metallic shining slag, which becomes red in the reducing flame.

It is dissolved by borax in the oxidating flame, with a slight effervescence, to a clear glass, of an amethystine colour. This colour disappears in the reducing flame, and the glass, unless saturated, becomes colourless.

It is with difficulty decomposed by microcosmic salt.



When treated in the oxidating flame, it affords a glass of an amethyst colour—losing it in the reducing flame, and which contains a skeleton of silica.

With a little soda, it fuses to a black glass; if the flux be increased, a difficultly-fusible black slag is formed.

Pyrosmalite is therefore a silicate, whose base is oxide of manganese. But as it dissolves with effervescence in borax, carbonic acid is present, combined with the oxide.

#### 4. *Felspar.*

*Per se*, in a bulb tube, it undergoes no change, and when perfectly transparent, yields no water.

In the forceps it is very difficultly fusible, melting only on the edges to a semi-clear vesicular glass. Like soda, it tinges the exterior flame yellow.

It is dissolved to a clear glass by borax, very slowly, and without effervescence.

It is perfectly decomposed by microcosmic salt, only when in the state of powder; a skeleton of silica being left behind. The glass becomes opalescent on cooling.

The powder fuses with soda very slowly, and with effervescence, producing a very difficultly-fusible clear glass, perfectly free from vesicles.

Solution of cobalt communicates a blue colour to the fine powder, on the fused edges only.

Felspar is thus a silicate; and as it is very difficultly soluble in borax, colouring the exterior flame yellow, the base must consist principally of alumina, with a little soda: but it cannot be determined, by the blowpipe alone, whether or not this silicate is combined with small quantities of other silicates.

About 75 milligrammes of the finely powdered mineral should therefore be melted with soda and borax, on charcoal, to a clear bead, which should be pulverized,—the powder treated with hydrochloric acid, and the solution evaporated to dryness; the chloride formed, dissolved in water; the clear solution separated by filtration from the insoluble silicic acid, and the filtrate examined for the different earths, in the



manner given under the Examination for Barytes, Lime, and Alumina. In this way it will be found, that the solution contains a large quantity of alumina, with a trace of lime.

The constituents thus found, shew that the silicate must be a felspar; but whether it contains soda, or potash with an accidental trace of soda, can be ascertained only by making a separate test for potash, in the way given at page 87. Potash-felspar appears to contain a trace of soda, as it generally colours the exterior flame more or less yellow. If a considerable precipitate of chloride of platinum and potash is formed, in an alcoholic solution of the alkaline portions of the mineral, it is a potash-felspar, which contains only a trace of soda; but if no precipitate is obtained, it is a soda-felspar. A portion of the hydrochloric acid solution, prepared to examine for the earths, may be employed in testing for potash, so as to avoid a second fusion of the substance with soda. An experiment may also be made, in order to ascertain if fluorine is present.

5. *Rohslag, from the Freyberg Smelting Works.*

*Per se*, in the forceps, it readily fuses, retaining its black-green colour, and tinges the exterior flame slightly yellow, from traces of soda.

It dissolves easily in borax to a clear glass, which is strongly coloured by iron.

Microcosmic salt dissolves it to a clear glass, which is likewise tinged yellow, leaving behind a siliceous skeleton.

It readily dissolves with soda on charcoal, with effervescence, to a black bead, which deposits no sublimate on the charcoal, when treated in the reducing flame, but frequently produces, after some time, a black spot on moistened metallic silver.

It gives the reaction of manganese, when fused on platinum foil, with soda and saltpetre.

From these blowpipe reactions, *Rohslag* would appear to be a silicate, whose principal base is protoxide of iron. But as it is known that such a slag also contains different earths, which cannot be detected with the blowpipe, about 100 milli-



grammes, in a pulverized state, should be taken, fused with soda and borax on charcoal in the oxidating flame, and the fused bead decomposed in the manner given under Barytes. Sulphuric acid, or bi-sulphate of potash, produces an exceedingly small precipitate of sulphate of barytes, in the diluted solution of the chlorides. When the protoxide of iron is converted by the addition of a few drops of nitric acid, into the peroxide, and a little chloride of ammonium added to the solution filtered from the barytes-precipitate, ammonia throws down peroxide of iron and alumina: oxalate of ammonia produces, in the solution filtered from these substances, a slight precipitate of oxalate of lime: and, lastly, microcosmic salt gives a slight precipitate, in the solution filtered from the oxalate of lime, of magnesia, and protoxide of manganese, combined with phosphoric acid and ammonia.

If these different precipitates, after edulcoration, be treated before the blowpipe, in the manner given already in speaking of the earths, it will be found that *Rohslag* consists of,—

Silica,  
Protoxide of Iron,  
Alumina,  
Lime,  
Magnesia,  
Barytes,

and traces of manganese, sulphur, and soda.

As the fragment of slag employed was perfectly vitreous, and free from admixed particles of *Rohstein*, the minute quantities of sulphur present may be considered as combined with barytes or lime.

6. *Black Copper Slag, from the Freyberg Smelting Works.*

It fuses readily in the forceps, but does not colour the exterior flame; but when moistened with muriatic acid, it colours the exterior flame bluish green, from the formed chloride of copper.

It behaves to borax and microcosmic salt like *Rohslag*, but the beads appear, with the same quantity of substance,



more intensely coloured, and the microcosmic salt does not contain so large a skeleton of silica. The iron present is therefore greater, and the silicic acid less.

With soda it fuses on charcoal, with effervescence, to a black lead. If this be treated for some time in the reducing flame, a white sublimate of oxide of lead is deposited on the charcoal.

By a reduction-test with a large quantity of soda, metallic particles are obtained, which behave to boracic acid like a mixture of lead and copper.

It shews the presence of a slight trace of manganese, when fused with saltpetre and soda on platinum foil.

It may be seen from these reactions, that *Black Copper Slag* is apparently a silicate of protoxide of iron, containing slight traces of oxides of lead and copper, and protoxide of manganese. The presence of earthy bases must be ascertained by the moist way.

For this purpose, about 100 milligrammes of the finely-pulverized slag should be fused to a bead in the reducing flame with soda and borax, and a button of pure silver, of about 80 milligrammes in weight; and the melted bead further treated in the way described for analyzing the *Rohslag*. In this way, it will be found that *Black Copper Slag* contains alumina and a trace of lime, and both must be considered as bases.

If the silver globule, free from slag, be treated with microcosmic salt for a short time on charcoal in the oxidating flame, and the resulting bead re-fused with a little tin in the reducing flame, it will become brownish red and opaque during refrigeration, from the presence of protoxide of copper.

*Black Copper Slag* therefore consists of,—

Silica,

Protoxide of Iron,

Alumina,

Lime,

and traces of oxides of lead and copper, with protoxide of manganese.



## (δ) COMPOUNDS OF METALLIC OXIDES.

The compounds of metallic oxides occurring in nature, which cannot be considered as Salts, are either oxides or hydrates. Some of these exist *per se*, while others are in combination with different simple minerals. Those which can be submitted to ignition in a bulb tube without yielding water, are oxides; and those which yield water, are either hydrates, or oxides containing hydrates.

Metallic oxides occurring in the products of smelting-works are often contaminated with sulphuric acid, arsenical acids, antimonious acid, and a portion of the oxides combined with these acids; but they never contain chemically-combined water.

The method of procedure to be employed in the investigation of such compounds of metallic oxides, may be gathered from the two following examples.

1. *Brown Earthy Cobalt.*

Ignited in a bulb tube, it yields water of an empyreumatic odour, which does not affect the colour of litmus-paper.

It suffers no change on charcoal in the oxidating flame; in the reducing flame, it gives off a slight alliaceous odour, but does not melt or yield a sublimate.

Borax, and microcosmic salt, dissolve it in the oxidating flame to a clear dark violet glass, which appears, after treatment in the reducing flame, greenish while hot, and pure blue when cold.

Soda does not dissolve it. It yields a mass coloured deep green, from the presence of manganese, when they are fused together on platinum foil in the oxidating flame.

When reduced with soda, and the resulting mass levigated, a metallic powder is obtained, which is attracted by the magnet, and which, when dissolved in microcosmic salt, appears green while hot, and blue when cold (cobalt and iron).

If a borax bead, saturated with this mineral, be treated for some time with a little lead on charcoal in the reducing



flame, the lead will congeal, on cooling, with a clear surface ; consequently, no nickel is present.

From these reactions, we may conclude,—as the brown earthy cobalt gives no water in a bulb tube, dissolves in borax and microcosmic salt, in the oxidating flame, with a dark violet colour, which changes in the reducing flame to blue, and produces, with soda on platinum foil, a green colour,—that it consists of hydrates of the oxides of cobalt and manganese: and, as it gives off a slight odour of arsenic, when heated on charcoal, and as the borax bead exhibits, when treated for a short time in the reducing flame (by which the manganese is reduced to the state of protoxide), a green colour while hot, and only blue when cold,—that it must be contaminated with arsenic acid and oxide of iron.

2. *Abstrich, from the Freyberg Smelting Works.*

It undergoes no change when heated in a bulb tube.

In a tube open at both ends, it behaves itself similarly.

It readily fuses on charcoal, spreads itself, and is reduced with effervescence to a very fluid metallic button, which gives off a strong alliaceous odour when kept in a state of fusion in the oxidating flame, coats the charcoal with a sublimate of oxide of antimony and oxide of lead, and finally behaves itself like pure oxide of lead.

Borax readily dissolves it in the oxidating flame to a clear green glass, which retains its green colour when cold. In the reducing flame, on charcoal, the bead spreads, and a number of lead globules are reduced, which have a strong alliaceous odour, and which coat the charcoal with oxide of antimony and lead.

If the small lead globules be combined and removed from the glass, and the latter again fused to a bead by a continued application of the reducing flame, it becomes colourless, and remains so when cold. But if a fragment of *Abstrich* be reduced in the reducing flame on charcoal, with a borax bead of half its size, the reduced lead removed, and the borax glass melted to a bead, it will appear greenish, and, when further treated with tin, of a pure vitriol-green, from iron.



Microcosmic salt dissolves it also, in the oxidating flame, to a clear green glass, which retains its green colour when cold. The colour of the glass undergoes no change on charcoal in the reducing flame while hot, but during the refrigeration it becomes turbid, and of a greenish yellow colour. If this glass be treated for a short time with tin, it becomes, during refrigeration, of a perfect blackish grey colour, from reduced antimony; if, however, it be kept long enough fluid in the reducing flame, the antimony will be volatilized, and a glass obtained, which becomes perfectly red during the refrigeration, from protoxide of copper.

It is instantly reduced with soda to a grey, somewhat brittle, metallic button.

From these reactions, it may be seen that *Abstrich* is an oxide of lead, containing a little oxide of copper, and a still smaller quantity of oxide of iron; and that a portion of the oxide of lead is combined with an arsenical and antimonial acid, as no volatile body is set free, when heated in an open tube.

#### ( $\epsilon$ ) SULPHURETTED, ARSENIURETTED, & SELENIURETTED METALS.

The system to be followed in these researches is similar to the one given under Metallic Oxides; but in many cases, where numerous metals are present, producing with the fluxes a confused coloration, different steps are to be taken. In most instances, before the fluxes are applied, the assay should be freed from sulphur and arsenic, by roasting it upon charcoal. The following examples may probably be of great service to the experimentalist.

##### 1. *Zinc Blende, from Przibram.*

When heated in a matrass, it falls to powder, and when this powder is heated to redness, no volatile ingredients are eliminated. If heat be applied to the powder in an open vessel, its colour changes to a lighter hue, and sulphurous acid gas is evolved, which reddens moistened litmus-paper.

On charcoal, in the reducing flame, the colour of the



powder likewise assumes a lighter tinge, and traces of sulphurous acid are evolved. In the reducing flame, *sinteration* ensues, and the charcoal becomes coated with a yellowish sublimate, which is white when cold. When treated with a solution of cobalt, it assumes a beautiful green colour (oxide of zinc). The fine powder dissolves copiously in borax and microcosmic salt, if it be heated with these fluxes on charcoal in the oxidating flame. The glass obtained is clear, and presents the ferruginous tint only; but when the glass is over-saturated, it appears turbidly streaked.

The roasted, as well as the unroasted powder, dissolves with effervescence in soda on charcoal, forming an opaline mass. After continued blowing with the reducing flame, the charcoal is at first covered with a reddish-brown sublimate, the colour of which is most distinct on refrigeration, and immediately recognized as oxide of cadmium; a large sublimate of zinc then occurs, and the soda is imbibed by the charcoal. If the unroasted substance is operated upon, a strong hepatic smell results, when the spot where the soda has been absorbed is moistened with water.

From the preceding reactions, we may infer that the ingredients of Zinc Blende\* are,—

Zinc, and

Sulphur,

With portions of Cadmium and Iron.

## 2. *Cupriferous Sulphuret of Bismuth.*

It decrepitates slightly when heated in a flask, ultimately melting, but volatile ingredients are not evolved.

When heated in an open glass vessel, sulphur sublimes, and in the under part of the tube a deposit appears, which, when strongly heated, melts to brownish drops; and the substance after cooling, is yellowish by transmitted light (oxide of bismuth.)

It melts readily upon charcoal, with ebullition and spirting. When the blowing is uninterrupted, nearly the whole vola-

\* [The Blende mentioned above is probably the *Black Jack* of English miners,—found in several of the Saxon localities.]—*Trans.*



tilizes, and the charcoal presents an orange-yellow coating. This deposit becomes lemon-yellow on cooling, and appears to be pure oxide of bismuth. If the residuary matter be melted with borax in the oxidating flame, a beautiful green glass is obtained, which will remain unaltered when cold, (iron and oxide of copper.) On treating this bead with tin, the assay will be of an opalestic red colour when cold, (sub-oxide of copper.)

If the roasted mineral is melted on charcoal, with bisulphate of potash, in the oxidating flame, and the fused assay be treated according to the method given under the examination for Bismuth, the operator will not find lead. When the mineral is melted with borax, on charcoal in the reducing flame, a greenish glass is obtained, which acquires a vitriol-green tinge when treated with tin, (protoxide of iron.) When a small portion of the mineral is purified with proof lead, on charcoal, and submitted to the oxidating flame, till the sulphur is eliminated, then melted with a little boracic acid, a button of copper remains behind, which forms about five per cent. of the quantity taken. The mineral, therefore, consists of,—

Bismuth,  
Sulphur,  
Copper, and  
Iron (a trace.)

3. *Bournonite, from Kuprinz, near Freyberg.*

When heated *per se* in a flask, it decrepitates, giving off volatile ingredients.

Heated in an open vessel, it evolves a large portion of sulphurous acid, and also a dense white vapour, which deposits partly on the upper, and partly on the lower side of the flask. The upper portion is volatile, (oxide of antimony) while the under is not, and being present in large quantities, appears to consist of antimoniuuret of lead.

It melts readily on charcoal, coating it with oxide of antimony, which congeals to a black bead, possessing a rough surface. By continued blowing, the bead re-melts, and the



charcoal becomes thickly coated with oxide of lead. If the globule, greatly reduced in volume, be treated with borax in the reducing flame, a colourless glass is formed, (no iron present,) but when subjected to the oxidating flame, a red cupriforous glass is obtained, (sub-oxide of copper,) and a cupreous button, which is brittle, from a small trace of sulphur, but when treated with a little soda, it becomes perfectly malleable.

The cupreous globule, when cupellated with lead, gives a button of silver.

This Bournonite, therefore, is composed of,—

Sulphur,  
Antimony,  
Lead,  
Copper,  
Silver (0.1 per cent.)

#### 4. *Nickel-Glance,*

decrepitates when heated *per se* in a flask, evolving a large quantity of sulphuret of arsenic; as the heat augments it melts. When heated in an open glass tube, arsenious and sulphurous acids are eliminated.

Heated on charcoal, a dense arsenical vapour is evolved, part of which coats the charcoal, and is readily expelled. If the greater part of the arsenic be volatilized, the assay fuses readily to a globule, quite brittle on cooling. If this be melted with borax on charcoal in the reducing flame, the glass appears, after perfect refrigeration, greenish blue. When the glass is separated from the metal, and heated on the ring of the platinum wire in the oxidating flame, it appears while hot, green, from cobalt and oxide of iron, and after cooling only slightly blue, from oxide of cobalt. On charcoal with tin, the glass assumes a vitriol-green colour, mingled with much blue. The metallic globule, separated from the borax-glass, and re-treated in the oxidating flame with borax on charcoal, gives the tinge from oxide of nickel only; the nickel separates in the metallic state, in the reducing flame, and the glass appears colourless.



From the above reactions, we may conclude that Nickel-Glance contains,—

Arsenic,

Nickel,

Sulphur,

Iron, and

Cobalt.

5. *Grey Copper, from Freyberg.*

It decrepitates in a flask, and gives, if melted, a red sublimate of sulphuret of arsenic.

The powdered mineral fumes pretty strongly, in an open glass tube. This vapour deposits a white sublimate on the glass, and appears to be a mixture of arsenious acid and oxide of antimony. At the upper end of the tube, a strong smell of sulphurous acid will be recognized.

The powder melts readily, *per se*, on charcoal, to a globule which fumes strongly. The vapour deposits partly on the charcoal, and consists of oxide of antimony. If too little has not been applied, a second sublimate is obtained, nearer to the assay than the sublimate of antimony; while hot, it is yellowish, on cooling, white, and assumes a green colour with a solution of cobalt, (oxide of zinc).

On account of the large quantity of sulphur present, no arsenical smell is perceived when the assay is treated *per se* on charcoal; but if another portion of the mineral be treated with soda on charcoal, the sulphur separates, the arsenic volatilizes, and is distinctly perceptible from its alliaceous smell. If a portion of the mineral be gradually roasted on charcoal, and treated with borax on the same in the reducing flame, a bottle-green glass is obtained, which becomes vitriol-green after dressing with tin, (iron,) and leaves a somewhat liquid metallic globule, which has a light copper colour, and therefore appears to consist of copper and a little antimony. If this globule be treated for some time with borax in the oxidating flame, the glass will be coloured red, from suboxide of copper, and the globule assumes a pure copper colour. If the cupriferous button be cupellated with lead, a small silver globule is procured.



*Grey Copper*, therefore, consists of—

Sulphur,  
Antimony,  
Arsenic,  
Copper,  
Silver,  
Iron, and  
Zinc.

6. *Rohstein*, from the *Freyberg Furnaces*.

Heated strongly in a matrass, it gives off no volatile particles, but assumes a blackish colour.

Heated in an open glass tube, it evolves sulphurous acid, recognisable by moistened litmus paper, or by its odour.

On the lower side of the tube, near the assay, a thin white coating is formed, not volatile, and appearing to be antimonious acid.

It melts very readily *per se* on charcoal; and, after a continued exposure to the reducing flame, coats the support with three different metallic oxides:—The sublimate which is at first produced, is deposited at some distance from the assay; it is white, and allows of being driven from one side to the other in the oxidating flame (oxide of antimony). The sublimate produced later, is immediately in contact with the antimonial sublimate; it is, while hot, of a lemon-yellow, and, on cooling, of a sulphur-yellow colour (oxide of lead). The third deposit, which is near the assay, appears only in very thin white laminæ; if it be moistened with a solution of cobalt, and heated strongly but carefully in the oxidating flame, it becomes green (oxide of zinc). *Rohstein* emits arsenical vapour, when heated with soda on charcoal.

When this substance, in a finely powdered state, is roasted carefully on charcoal, and a part of the roasted assay dissolved in borax on a platinum wire in the oxidating flame, a clear glass is obtained, which presents the colour of oxide of iron only. If this glass be treated on charcoal with tin for a short time, it becomes reddish on cooling, from copper; after a longer reducing flame, the copper is separated, the glass



remains clear when cold, and of a pure vitriol-green hue, from the presence of iron.

If another portion of the roasted *Rohstein* be dressed with soda, borax, and lead, and heated on charcoal in the reducing flame, those metallic oxides are reduced, combining with the lead.

The plumbiferous alloy, while yet held in a molten state in the blowpipe flame, evolves antimony; when the blowing is discontinued, it cools with a clear surface,—(no nickel). If the plumbiferous globule be melted with boracic acid till the greater part of the lead is separated, and it be then melted with microcosmic salt on charcoal in the oxidating flame, and the resulting glass bead treated with tin, a very distinct reaction of copper is also produced.

The constituents of this *Rohstein* are, therefore,—

Sulphur,

Iron,

Antimony,

Lead,

Copper,

Zinc,

Arsenic,

—and, according to a quantitative silver assay, also,

Silver (0.18 per cent.)

#### 7. *Lead-Speiss, from the Freyberg Furnaces.*

Heated to redness in a glass tube, it liquefies, forming a black mass, but evolving nothing volatile.

A little arsenious acid is eliminated in an open glass tube; near the assay it exhibits a white non-volatile deposit of antimonious acid (probably), and, on the upper part of the glass tube, a smell of sulphurous acid is distinctly observed.

On charcoal *per se* in the reducing flame, it melts at the commencement to a globule, and evolves arsenical vapour; but after continued blowing, a crust forms on the surface and the globule becomes infusible. But if another splinter of the mineral be melted with twice its volume of lead and boracic acid on charcoal in the reducing flame, a sublimate of oxide of



antimony, sufficiently distinct, is obtained, and a dense arsenical vapour is observed.

If a part of this Lead-Speiss, in a finely powdered state, be roasted on charcoal, (by which means the sulphur and a part of the arsenic volatilizes, but the other part of the arsenic is converted into arsenic acid, and, without being capable of separation, combines with the metallic oxides formed,) and the roasted assay melted with borax on charcoal in the reducing flame, a black opaque glass and a ramose white metallic globule are obtained; a portion of the glass diluted with borax on charcoal, and treated for some time in the oxidating flame, exhibits a greenish blue colour; melted in the ring of a platinum wire, and treated with the oxidating flame, it assumes a reddish yellow colour, which, on refrigeration, becomes yellowish green,—it is therefore coloured by iron and cobalt. If the metallic globule be melted on charcoal with microcosmic salt in the oxidating flame, a green glass results, coloured by nickel and oxide of copper, which, when treated with tin, is, on refrigeration, red and opaque, owing to the presence of suboxide of copper.

According to the above reactions, Lead-Speiss consists of—

Arsenic,  
Sulphur,  
Nickel,  
Iron,  
Cobalt,  
Antimony,  
Copper;—and gives, on cupellation,  
Silver, (0.048 per cent.)

If convenient to ascertain the presence of nickel, iron, and cobalt, with greater certainty, and to observe the colours of the metallic oxides separately in the fluxes, rather a circuitous method, similar to the following, must be pursued.

Roast about 75 milligrammes of finely divided Lead-Speiss, on charcoal in the reducing flame, to volatilize the sulphur and the other metals, so as to form arseniates of nickel and cobalt.

Melt the roasted assay with equal parts of soda and borax, on charcoal in the reducing flame. By this treatment, nickel,



copper, and the greater portion of the cobalt, antimony, and arsenic, are reduced, forming a limpid metallic globule, while the iron, as protoxide, and the irreducible oxide of cobalt, remain dissolved in the flame. If the glass flows readily, and is free from metallic particles, the blast must be discontinued, and the arsenical metals separated from the glass.

Pound the glass, mix it with twice its volume of soda, and expose the assay on charcoal to the reducing flame. Then sift the grey metallic powder, attractable by the magnet, from the slaggy matters, dissolve it in borax, and either the colour from iron, or that from iron with a little cobalt, will be the result.

When the metallic particles are treated with borax on charcoal for a long time in the oxidating flame, and a portion of the resulting glass bead dressed with more borax and some lead, and then exposed on charcoal to the reducing flame, the characteristic cobalt-blue tinge will be communicated to the flux.

The assay, freed from cobalt by the assaying with borax, must be re-treated with this flux on charcoal in the oxidating flame, and the glass bead, while hot, separated from the metallic assay. It exhibits the colour from nickel only. When placed on another part of the charcoal, and treated in the reducing flame, the nickel is separated in a metallic state, the glass assumes on cooling a brown-red colour, and is opaque from suboxide of copper. After long blowing, the copper is also separated in a metallic state, and the glass has a tinge only of blue, from a trace of remaining cobalt. (The copper does not appear to be reduced before the nickel is separated.) If the borax glass, perfectly free from metallic globules, be melted on charcoal with microcosmic salt in the oxidating flame, a glass darkly coloured is attained; if it is melted in a similar manner on a platinum wire in the oxidating flame, it is infusible, and appears coloured beautifully green by oxide of nickel and copper.

8. *Cuprififerous Seleniuret of Lead—Seleniuret of Copper and Lead.*

Decrepitates strongly when heated in a flask, but otherwise does not change. Gives a sublimate in an open glass



tube, which, at the greatest distance from the assay, is red, and nearer to it steel-grey (selenium). No sulphurous acid is detectable either by the smell at the upper end of the tube, or by moistened litmus paper. Fumes on charcoal, smells strongly of selenium, melts on the surface only, and coats the charcoal at the commencement with selenium, which appears grey and of a shining metallic lustre, and afterwards also with oxide of lead. After continued blowing, a black slaggy mass remains, which melts readily to a globule, and imparts a bottle-green tinge to the glass, owing to the presence of an inconsiderable quantity of iron. The separated metallic globule is somewhat malleable *per se* on charcoal, produces a strong sublimate of oxide of lead, and, if treated with boric acid on charcoal, gives a pure cupriferosus globule.

The constituents therefore are :—

Selenium,

Lead,

Copper, and

Iron (a trace).

(§) COMBINATIONS OF METALS WHICH CONTAIN  
LITTLE OR NO ARSENIC.

The system to be followed in such researches is very simple, as may be deduced from the following examples.

1. *Native Amalgam.*

Heated strongly *per se* in a matrass, metallic orbicles deposit in its neck, which cohere on shaking the vessel. If the porous residue be first treated in the oxidating flame, and then in the reducing flame on charcoal with borax, the operator will obtain a colourless glass, and a metallic globule which has the appearance of silver, and remains unchanged by cupellation. It is composed of,—

Silver, and

Mercury.



### 2. *Workable Lead from the Freyberg Furnaces.*

If convinced that mercury is absent, a glass alembic is not necessary. It can also be dispensed with in the case of *Workable Lead*.

In the open glass tube, it melts to a bead, which is coated with oxide, and gives nothing volatile. It melts readily on charcoal; has a moderately strong smell of arsenic; at the commencement it coats the charcoal with oxide of antimony; and also with oxide of lead, after continued blowing.

Melted with borax on charcoal, in the reducing flame, a clear colourless glass is obtained, remaining so when melted in the ring of a platinum wire, and treated for some time in the oxidating flame; it is therefore free from iron and cobalt. Melted with soda and borax, on charcoal in the reducing flame, and the glass placed on a moistened silver plate, a black stain of sulphuret of silver is often produced; it is therefore not always free from sulphur.

Treated with boracic acid, on charcoal in the reducing flame, till only a small metallic particle remains, and this globule melted with microcosmic salt again, on charcoal, in the oxidating flame, a greenish glass is obtained, which, if treated with tin, appears on cooling opaque red, from copper. If the metallic globule be cupellated, after melting with microcosmic salt, a globule of silver remains behind.

According to the foregoing reactions, *Workable Lead* consists of,—

Lead,  
Silver,  
Copper (traces),  
Arsenic,  
Antimony, and sometimes traces of  
Sulphur.

### 3. *Impure Tin.*

On charcoal in the oxidating flame, it behaves, as regards its easy oxidation, like pure tin. In the reducing flame, it gives a coating of oxide of tin, mixed with a dark yellow



powder, which exhibits a lemon-yellow colour on cooling, and appears therefore to be oxide of bismuth. As a confirmation, another part of this tin was melted with microcosmic salt on charcoal, first in the oxidating flame and then in the reducing flame. The glass bead was, while hot, transparent, but, on cooling, became black and opaque, which indicated the presence of bismuth. Melted with borax on charcoal in the reducing flame, a glass was produced which was coloured pure vitriol-green, by protoxide of iron.

The tin was contaminated with bismuth and iron.

#### 4. *Black Copper, very Impure.*

Heated strongly in the open glass tube, it eliminates a little sulphurous acid, recognized by the introduction of moistened litmus paper. At some distance from the assay, a very slight white deposit is formed, having the appearance of oxide of antimony. On charcoal, *per se*, it melts with difficulty, yielding a sublimate of oxide of lead only. Melted together with proof lead and boracic acid, in such a manner that one side remains free, while the lead dissolves, a distinct sublimate of oxide of antimony is produced. The remaining part of the metallic globule, which is free from lead, has a greyish white colour, and is scopiform.

The black copper, melted with borax on charcoal in the reducing flame, produces a glass coloured blue by cobalt, which melts on a platinum wire in the oxidating flame, appearing while hot, green, and, on cooling, blue (cobalt and oxide of iron). The metallic globule remaining, after treatment with boracic acid, produces, with microcosmic salt on charcoal in the oxidating flame, a glass which appears of a beautiful green both when hot and cold, and becomes opaquish red with tin (nickel and copper). The remaining undissolved metallic globule still appears greyish white, and is extremely dendritic. This appearance indicates the presence of arsenic, which is combined with nickel, and cannot be separated from this metal either by boracic acid or microcosmic salt. A separate examination for arsenic, according to page 208, indicates the presence of this metal.



This black copper consists of—

Lead,		Iron,
Copper,		Antimony,
Nickel,		Arsenic,
Cobalt,		Sulphur ;

and, (according to a separate examination,) a little silver.

5. *German Silver, or White Copper (Argentan).*

No change ensues when it is heated in a glass tube.

Melted on charcoal in the reducing flame, it affords a sublimate which is yellow, while hot, and white when cold ; with solution of cobalt it assumes a beautiful green colour, and therefore may be regarded as oxide of zinc.

The globule, melted *per se* on charcoal, and treated with borax in the outer flame, till those metallic oxides, irreducible in the inner flame, are dissolved, should be re-melted in the reducing flame to separate the metals. The glass thus obtained is blue, and does not change its colour when fused on a platinum wire in the oxidating flame. Cobalt is therefore only dissolved.

The metallic globule, freed from cobalt and melted with microcosmic salt on charcoal in the oxidating flame, gives a glass coloured dark green. A portion of this vitreous mass, treated with more microcosmic salt on platinum wire in the oxidating flame, affords a bead which, on cooling, remains of a beautiful green (copper and nickel). This, removed, and treated on charcoal with tin, becomes opaque-red on cooling, owing to the presence of copper.

The globule remaining undissolved, after treatment with microcosmic salt, is perfectly malleable, appears reddish-white, and consists, as it indicates a trace of silver only on cupellation, of copper and nickel.

The composition of this alloy, is therefore,—

Copper, with a trace of silver,  
Nickel, with some cobalt, and  
Zinc.



### SECTION III.







## QUANTITATIVE ANALYSES

### WITH THE BLOWPIPE.

*Preparation of the Substance whose Metallic Constituents  
are to be ascertained.*

Before it is possible to proceed with the investigation of a mineral, ore, furnace product, &c., in order to ascertain the different metals present, they must undergo a certain treatment, which is termed "Preparation of the Assay."

The antecedent labours are :—desiccation of those substances containing mechanically-mixed water ; and pulverization, when required in a finely-divided state. Brittle substances, difficultly-reducible to powder, may be crushed under the hammer upon an anvil : but malleable bodies can be first laminated, and then cut into shreds with a strong scissors.

It frequently occurs that ores, prepared on a larger scale, are apparently dry, although some per cents. of water may be mechanically held between their layers ; in other cases, ores kept, after desiccation, in damp apartments, or in open vessels, re-absorb moisture from the air. Therefore, if the substance under examination contains mechanical moisture, a quantity greater than is requisite for two assays, should be heated in a porcelain basin over a spirit lamp, to expel the water, and the desiccated mass then triturated. During the desiccation, care must be taken not to raise the temperature so high as to roast the specimen.

Minerals and furnace products received for investigation in a dry but not pulverized state, may be broken into fragments upon the anvil, and afterwards, if they be fit for powdering, triturated in the agate mortar. Great exactness in the results cannot be expected, unless a portion eight or ten times greater than is required for one experiment be pre-



pared, (except pure crystals and homogeneous specimens of minerals). It is impossible to represent the average constituents of any substance, *e. g.*, a rich metallic ore associated with extraneous non-metallic constituents: for by taking a small portion for investigation, it is probable that either too small or too large a per-centage of the metals is obtained, according to the excess of the metallic or non-metallic ingredients present. Therefore, of minerals dressed on a larger scale, an ounce should be selected from various parts of the mass, pulverized in an iron mortar, and, if possible, reduced to powder. A satisfactory result will thus be obtained, if eight to ten decigrammes (the quantity necessary for an experiment) be employed.

### DESCRIPTION OF THE VARIOUS QUANTITATIVE ANALYSES.

All minerals, ores, and furnace products, must be specified under various heads, viz. :—

¶ Ores, Minerals, and Products of Smelting-Works,—whose subdivisions are :—

- (a) Such as contain volatile ingredients;
- (b) Such as contain no volatile ingredients except chlorine;
- (c) Compounds of metallic oxides reducible upon charcoal; and,
- (d) Such as are irreducible with borax and *workable lead*.

¶ Metallic compounds, the principal ingredients of which are :—

- (a) Silver.
- (b) Gold.
- (c) Copper or Nickel.
- (d) Lead.
- (e) Antimony or Zinc.
- (f) Tin.
- (g) Mercury.
- (h) Iron or Steel.



¶A *Ores, Minerals, and Furnace Products.*

## 1. THE SILVER ASSAY.

The silver assay with the blowpipe, which Harkort has published, is one of the most important quantitative analyses that can be performed with this instrument. It affords the means not only of ascertaining the proportion of silver in any ore, mineral, or production of smelting-works, &c., but also of determining its quantity with sufficient accuracy. However, to obtain satisfactory results, it must be considered what ingredients besides silver are combined in the body under examination.

## (a) EXAMINATION FOR SILVER IN THOSE CONTAINING VOLATILE INGREDIENTS.

To this class belong, besides the ores prepared on a greater scale, and containing large quantities of Sulphurets of Iron, Copper, Arsenic, Antimony, and Zinc, the following minerals: Vitreous Silver (Sulphuret of Silver), Antimonial Silver, Melan Glance, light and dark Ruby Silver, Telluric Silver, Arsenical Silver, Argentiferous Sulphuret of Copper, Miargyrite, Fahl Glance, Bismuthic Lead Ore, Vitreous Copper, Bournonite, Tin Pyrites, Galena, &c.; and of the furnace products, *Rohstein*, *Bleistein*, *Kupferstein*, *Kupferleg*, *Tutty*,\* Lead Speiss, Cobalt Speiss, &c.

After preparation in the manner given at pages 263, 264, the following procedure is to be undertaken:—

*Weighing and Dressing of the Assay.*

The weight is to be ascertained either on a balance, or upon the scales, described after the gold assay, and at the end of this Section.

Of rich silver ores associated with non-metallic constituents, and therefore prone to represent very varying contents, two, or if necessary, three samples of 1 decigramme, ought to

\* [Explained in notes, at pages 143-169.]—*Trans.*



be weighed twice; but poor silver ores, crystallized minerals, and also products of smelting-works, which generally form a homogeneous mass, may be weighed once only. However, if the experimenter be not accustomed to blowpipe examinations, even ores which generally do not materially differ, should be weighed and examined twice.

A sample of 1 decigramme being weighed, it is to be poured from the basin of the balance (the adhering particles can be removed with the hair-brush, so as to incur no loss,) into the mixing capsule, and dressed with borax and proof lead. The requisite proportion of borax varies according to the quantity and fusibility of the substance. In most cases, 1 decigramme is sufficient even for a difficultly-fusible alloy; if, however, during the operation, the mass with this quantity appears intact when submitted to a strong heat, another small portion of borax should be added. For ores easily fusible, particularly for such as are not associated with earths, and consist of sulphurets which unite quickly with lead, but do not oxidize so readily as this metal, a small quantity only of borax (50 to 75 milligrammes) ought to be taken. However, if argillaceous constituents be in excess, or the assay contains much iron, cobalt, or tin, one decigramme of the flux will be necessary.

With regard to the lead, it should first be considered what other metals besides silver are extant in the alloy, for if an ore, mineral, or furnace product contains no more than 7 per cent. of copper, or 10 per cent. of nickel, 5 decigrammes of lead will be sufficient for 1 decigramme of the ground ore; but, on the contrary, if the substance contains more than the above-cited per-centage of copper or nickel, the quantity of lead must be increased. As it is impossible always to have prescience of the per-centage of these metals in a mineral or ore, the safest plan to adopt, is to have a slight excess of lead, otherwise the copper cannot be perfectly detached from the silver, and also, a fusion of a nickeliferous *workable lead* is nearly impossible.

The annexed table shews the proportions of lead to the substance in some of the most common minerals and furnace products, consisting partly of copper and nickel.



	NAMES OF THE MINERALS.	PER CENTS. OF METAL.	DECIGRAMM <sup>s</sup> OF LEAD.
ONE DECIGRAMME OF	Copper Glance . . . . .	·84 Copper.	15 Decigra <sup>ms</sup>
	Purple Copper . . . . .	·63 „	12 „
	Tennantite . . . . .	·45 „	10 „
	Copper Blende . . . . .	·42 „	10 „
	Grey Copper . . . . .	·40 „	10 „
	Cupreous Bismuth . . . . .	·35 „	10 „
	Copper Pyrites . . . . .	·34 „	10 „
	Argentiferous Copper Glance .	·30 „	10 „
	Tin Pyrites . . . . .	·28 „	7 „
	Bournonite . . . . .	·12 „	7 „
	Kupferstein, obtained from the fusion of roasted Cupriferous Bleistein . . . . .	·45 „	10 „
	Kupferleg . . . . .	·50 „	10 „
	Lead-speiss . . . . .	·50 Nickel, Cobalt, and Copper . .	10 „
	Cobalt-speiss . . . . .	·55 Nickel and Cobalt.	10 „

Having added the necessary proportion of borax and lead to the ground ore, the whole assay is to be mixed, the handle of the spoon serving as a spatula. A cornet of soda-paper is now held carefully between the thumb and forefinger of one hand, the capsule with the assay in the other; its contents are carefully placed in the cornet, and the adhering particles detached by the small brush, and added to the main mixture in the recipients. When this is accomplished, the sides of the upper part of the cornet must be brought together, folded over about a quarter of an inch, and pressed; care must, however, be used in closing it, lest the under part be broken, and particles of ore be lost. This accident will be avoided



by keeping the cornet upon the long side of the middle-finger, during the filling process.

The cornet, filled with the assay, is next placed upon a porcelain basin, where it is to remain until required for the experiment,—and if the same substance is to be investigated several times, or various ores are to be examined for silver, they must be weighed and dressed similarly. The operator should remember, that the weighing of the assay must be followed immediately by the dressing with borax and lead, and should different substances be examined, a superscription on the cornets will prevent any mistake. The weighing and dressing is succeeded by,—

*The Fusion of the Assay.*

This is effected upon charcoal, in the reducing flame. First, a cylindrical cavity, the size of the filled cornet, is bored (with the instrument described in the first Section,—page 19, *Fig. 23*,) into a good piece of charcoal, upon its diametrical section, close to one of the edges. In this deepening the cornet is placed, and carefully adjusted, so that it touches on all parts.

A strong reducing flame is now to be directed upon the whole surface nearly of the assay. Although the soda-paper becomes carbonized, still this carbon is not destroyed, until the upper part of the borax, together with the particles of ore, are fused; and when this is accomplished, it is impossible to remove them by the current of air. If, after the combustion of the carbon, portions of the assay appear as liquid scoriæ, mingled with lead globules, the whole must be enveloped in a strong and pure reducing flame. During this operation, a small portion of sulphur, arsenic, antimony, zinc, &c., volatilize, but the greater portion fuses, and coalesces into a bead with the lead, &c.; while the argillaceous ingredients, with small quantities of the non-volatile metals, oxidized, unite with the borax, forming scoriæ. Sometimes, when the alloy consists of difficultly-fusible metals, the scoria appears to be perfectly free from lead, although, in its under part, the substance remains intact; to submit this to the action of the



flame, the charcoal must be inclined to the other side, and thus a further flowing action takes place in the cavity, and the sample is reversed.

By this turning, which cannot be spared even with the most easily-fusible mixture, the bottom of the soda-paper is brought to the upper part of the charcoal, and as it will be difficultly consumed by a pure reducing flame, the assay should be placed in such a position to the flame, that the recrements are only covered by it where they are not in contact with the soda-paper; by observing these directions, atmospheric air accedes, and the cornet is speedily consumed. When this occurs, the whole sample must be re-covered by the flame, in order to reduce and reunite with the main bead all traces of lead, which, during the antecedent process, might have oxidized and combined with the recrementitious matter.

If the scorice which have been enveloped in the reducing flame appear orbicular, perfectly fluid, and free from lead, although their position near the bead of lead had changed several times, the operator may infer that they are free from silver also.

During the operation, it is not indispensable to keep the leaden bead wholly covered, while the scorice are subjected to the reducing flame, but the temperature must always be sufficiently high to keep the plumbiferous globule fluid. However, if, owing to an imperfect reducing flame, it happens that the recrementitious particles spread over the charcoal, presenting small globules of lead, the main bead must be covered wholly by the flame, and, by inclining the charcoal, brought to those parts of the support where the small ones are visible, in order to combine with them. The assay being treated, as before mentioned, so far that the scorice are in full fusion, and free from lead, the reducing must be substituted for an oxidating flame, which must be kept at a somewhat greater distance from the lead globule. By the judicious application of the flame, the volatile metals, together with the sulphur, separate from the lead, and the readily-oxidizable ones, as iron, tin, cobalt, likewise a small portion of nickel and



copper, combine with the scoriæ, partly as protoxides, and partly as peroxides, whereas the greater portions of the nickel, copper, and silver, remain with the lead.

After elimination of nearly all the volatile constituents, the lead oxidizes rapidly, and also traces of silver,—the latter, even in rich ores, is almost imperceptible, and becomes still more insignificant, when the scoriæ containing the oxide of silver touch the hot charcoal, for the greater portion of it is again reduced, and can be made to re-combine, by the movement of the scoriæ with the argentiferous lead globule.

So soon as all volatile ingredients are vaporized, the motion and oxidation of the lead increase rapidly, attended with considerable ebullition of the scoriæ. By inclining the support, the button, which is generally surrounded with recrements, is brought to a free place, and allowed to refrigerate. After cooling, if the argentiferous lead (*workable lead*) obtained be of a white colour, the operation is completed; but if it appears dull or blackish, traces of sulphur are present, and these must be expelled by repeated oxidation. Great care must be taken, in the expulsion of the volatile ingredients; for, in the first place, the brittleness of an impure *workable lead* might prevent it from being removed from the scoriæ, without loss of some of the particles; and, secondly, not only because it can with difficulty be cupelled, but particularly, if sulphur remains, a violent motion ensues in the operation, occasioning the projection of some of the metal from the cupel.

If the regulations here laid down be strictly followed, the small lead globules remaining in the scoriæ will not be perceptibly argentiferous, as they result only from the oxidation of the main bead, and reduction of the oxide from the silverless recrements by the carbon. Although the small quantities of silver oxidizing in company with the lead are to be calculated as loss, still they, in comparison to the oxidized quantity of lead, do not surpass the proportion of silver lost in the beginning of the oxidation with the same quantity of lead; as I shall shew after describing this operation.

Of substances treated in this manner, the most difficult of



fusion are the following:—sulphurets of iron, arsenic, some ores of nickel, cobalt, and a species of *rohstein*, principally consisting of sulphuret of iron; whereas other substances appertaining to the same class, generally melt readily, even when they contain difficulty-fusible earths.

The fusion of these substances is greatly facilitated by roasting on charcoal, a process to which the lead assays are generally submitted, with subsequent addition of the borax and lead necessary. In this operation, the greater part of the arsenic and sulphur volatilizes, and the remainder becomes acidified, and combines with the newly-formed oxides of cobalt and nickel. By submitting this assay to the inner flame, the oxide of nickel is reduced, the peroxide of iron becomes protoxide, and the acids are reduced to sulphur and arsenic. The metallic nickel combines with the lead, together with the silver and traces of sulphur and arsenic, forming an easily-fusible alloy; the free portions of sulphur and arsenic volatilize, and the peroxide of iron and oxide of cobalt dissolve in the borax.

When the scorïæ appear perfectly molten, and free from lead, it is only necessary to submit the alloy for some moments to the outer flame, when the last traces of sulphur and arsenic will be expelled.

The fusion of argentiferous minerals should always be accomplished in the reducing flame, because, if the assay be treated with the oxidating flame, exact results cannot be expected: a considerable portion of the lead would oxidize at the outset, dissolve in the borax, and, coming in contact with the carbon, be reduced by it, forming globules with small particles of silver, which are intermingled with the recrements; and even if these reunited with the main bead, they would soon be replaced by others newly-formed, undistinguishable from the argentiferous lead.

The time necessary for fusing a non-previously roasted alloy varies from five to eight minutes, according to its fusibility and the quantity of volatile and argillaceous constituents.

The refrigerated globule must be separated from the scorïæ in the following manner:—The whole mass should be removed



with the forceps, wrapped in paper, and placed upon the steel anvil; by a few gentle strokes of the hammer, the recrementitious particles are perfectly detached.

*Treatment of the Workable Lead obtained by the Fusion.*

The separation of lead from silver contained in *workable lead*, is effected by oxidation at a red heat, with access of atmospheric air, and is based upon the property of lead to oxidize at such a temperature, while the silver remains unaltered. Harkort divides this process into two stages, the first termed "Oxidation," the second, "Cupellation."

*The Oxidation.*

This operation is a very simple process. A cupel of *sieved* bone-ashes is prepared by means of the instruments described at page 23, placed upon the mould, and submitted to a red heat, in the oxidating flame, to remove all hygroscopic moisture. By this precaution the experimenter will avoid a loss of *workable lead*.

The cupel being desiccated, is charged with the *workable lead*, and smelted in a strong outer flame until the surface of the assay brightens, and oxidation commences. If the *workable lead* contains large portions of copper or nickel, the fusion requires more time, owing to the nickel separating from the lead at the commencement, covering its exterior with an infusible layer, which prevents access of atmospheric air, and makes the oxidation impossible,—the copper renders the alloy but difficultly fusible. If, therefore, much nickel is present, a small quantity of pure lead (2 to 4 decigrammes) should be added to the assay. When the oxidation begins, the point of the blowpipe must be advanced so as to produce a fine blue flame, which must be directed upon the sides of the cupel, in order to continue the fusion without immediate contact; by this procedure the surrounding air has access to the bead, the lead and copper of which oxidize, forming scoriæ, which are conveyed on the liquid mass to the sides. If the assay does not contain much silver, the scoria iridesces beautifully, and remains upon the sides of the cupel, forming



a solid mass (*litharge*); on the contrary, if the alloy be rich in silver, no prismatic colours appear, so that from this phenomenon, a conclusion is arrived at with regard to the richness or poorness of the ore. The presence of copper renders the *litharge* almost black; but if the assay be free from this metal, the dross is of a reddish-yellow colour.

Care must be taken during this process to preserve an appropriate temperature, for if the heat be too high, portions of lead volatilize, particularly in rich assays, entraining a small portion of silver, and the *litharge*, instead of congealing, remains fluid, and is absorbed by the bone-ashes of the cupel, thus causing a new loss of silver. On the other hand, if the temperature be not sufficient for keeping up the oxidation, a coating of *litharge* covers the exterior of the alloy, and checks oxidation, &c. The loss of silver caused by this oversight is nearly imperceptible, as the oxidation again proceeds when the temperature augments; but this accident should nevertheless be avoided.

If the oxidation be effected at the temperature, the *litharge* surrounding the bead solidifies, and accumulates in such a manner, that it covers the greater part of the assay, obstructing the free access of air. When this is the case, the cupel must be inclined, that the molten lead may flow by its own gravity to the sides,—thus acquiring a greater oxidating surface. Its volume now diminishes rapidly, and when it is in a poor assay, of about double the size represented in *Fig. 6 (c)*, page 7, or in a rich ore, the operator must carefully regulate the distance between the cupel and the flame, for the gradual cooling of the assay. The slow refrigeration is necessary to obtain the globule in a regular form, and prevent any loss of silver, for by too sudden a decrease of temperature, the lead generally detaches itself at once from the scoriæ, often occasioning the projection of particles of silver from the cupel.

I cannot omit mentioning here, a phenomenon peculiar to rich alloys, which ensues, if a sample containing upwards of 50 per cent. of silver, be treated so far that the proportion of silver to lead is about seven to one. In such a case, the congealing globule acquires a greyish-white coating, contain-



ing a large proportion of silver.—(I am inclined to consider this coating as a sub-oxide of lead, with metallic silver). The greater portion of this pellicle remains with the *litharge*, causing a considerable loss of the noble metal. If this inconvenience occurs, the assay ought to be fused again with the reducing, and afterwards submitted to the oxidating, flame, in order to change the above given proportions. If, therefore, rich substances are to be examined, the process must be pursued until the globule consists of pure silver nearly, as the previously mentioned phenomenon only manifests itself in alloys of the composition one part lead to seven of silver.

The operation being regularly terminated, the cupel is removed from its support, put upon the anvil, and the *litharge*, with the argentiferous globule, placed in a porcelain basin. During the refrigeration, the operator can prepare the cupel for the succeeding process.

The separation of the metallic globule from the dross, is most easily accomplished by placing the mass upon the anvil in a strong paper, and striking it rather sharply with the hammer. In this process also, the globule acquires an indentation, which admits of its being placed in the cupel, so that it cannot move from place to place.

#### *The Cupellation.*

The cupellation is the most difficult part of the whole experiment, and, for obtaining exact results, requires great care, and also long practice. One of the principal requisites is a good cupel, possessing a smooth surface, no fissures in the interior, and not too dense, as it is necessary in this operation that the bone-ashes absorb all the *litharge* generated. For this reason, bone-ashes not only sifted, but also levigated, ought to be employed, the first not affording so dense a mass, whereas the latter by themselves are apt to give a soft surface.

The preparation of the cupel is conducted in the following manner:—The oxidation process being finished, and the argentiferous lead, &c., removed from the bone-ashes, the cupel not spoiled by *litharge* is to be comminuted with the iron



spatula, and then placed in the iron stand, the cavity filled with levigated bone-ashes, and the new cupel formed by means of the bolt; the cupel is then submitted to a red heat. If any crevices, owing to moisture, open during the process, they are readily filled up by applying, with some force, the warm clean bolt, upon the interior.

When the cupel is finished, the argentiferous globule must be placed close to its border, and the support then approached near the flame. An oxidizing flame must then be directed horizontally upon the button until fusion ensues; the support must then be brought gradually to a perpendicular position, by which motion the globule leaves its place and rolls to the centre of the cupel. At this moment the flame is withdrawn from the bead, and directed upon the cupel only, surrounding it, which must be kept up at a red heat during the experiment. If the temperature be sufficiently high, the bead will remain molten without being touched by the flame; but if not, the assay must be again fused with the open flame, and afterwards subjected to the other treatment.

The surest guarantee for the success of the cupellation is the dryness of the bone-ashes and the absorption of the *litharge*, for when the bone-ashes are not sufficiently heated, the cupel acquires a coating of *litharge*, upon which the globule oscillates, and if the assay be not altogether spoiled, at least the results will be uncertain. For the completion of the operation, the heat is augmented for the purpose of expelling entirely the last coating of *litharge*; the globule must then be withdrawn gradually from the flame and allowed to cool. An inspection through the lens will then convince the operator whether the surface is bright, and if not, a new cupellation must be undertaken. In rich assays, five to ten seconds before the brightening,\* a beautiful iridescence ensues, which disappears with the remainder of the *litharge*. It is, however, necessary to continue the blast, and the touching the button on all parts with the apex of the blue flame, until the silver

\* [A beautiful play of colours appear momentarily on the surface of the globule, which, after a short time, appear lustrous and white,—this phenomenon is denominated “the brightening.”]—*Trans.*



exhibits a pure bright colour. In the heating of a greater globule, small asperities sometimes appear on the exterior, which might be taken for extraneous matters, but after refrigeration, they will be recognized as silver.\*

To prevent a "spitting" of the metal, the globule must, as previously remarked, be cooled very gradually.

The cause of this spitting is, according to Lucas and Gay-Lussac, owing to silver in the molten state being capable of absorbing large portions of oxygen gas from the air, which is rapidly discharged during the refrigeration of the metal, and sometimes occasions the projection of small portions of silver from the cupel.†

Should the assay contain copper, at the moment of brightening the silver, it generally dilates upon the cupel, and although it appears white after cooling, this is no proof of its purity. Such a button ought to be fused and cupellated a second time, with 1 decigramme of pure lead, if it is large enough for weighing, and if not, 50 milligrammes, in order to obtain a pure and splendid globule. The cupellation of a cupriferous alloy in this manner is of better effect, than by the direct addition at the outset of a quantity of lead sufficient for the separation of the copper, as this in many cases requires the double quantum of lead, impeding the oxidation as well as the cupellation; the reason is, that in the oxidation much less copper oxidizes, comparatively, than in the cupellation.

In the cupellation of poor assays, inconveniences often arise, which appear to be of very little importance, but if attention be not paid to them, exact results will not be obtained. I shall therefore advert to them.

1. It often happens that the small globule adheres to the cupel, and when the process is pursued, the form becomes

\* When silver obtained from chloride of silver is treated with the oxidating flame, after the lapse of a few seconds, asperities present themselves, which, however, cannot be removed either by the reducing flame or upon charcoal. A partial oxidation of the silver appears to take place in this instance (?)—*Author*.

† [Graham remarks that this property is possessed by pure silver only, and that it does not appear at all in silver containing 1 or 2 per cent. of copper.]—*Trans*.



so irregular that a determination of its weight is very difficult. When this occurs, a small portion of pure lead should be added, to increase the weight of the assay, and the whole re-cupellated. If then the cupel be slightly inclined, the gravity of the button is sufficient to segregate it from the impediment, and conduct it to another place, where the process can be terminated.

2. Sometimes, if the operator does not possess sufficient practice in cupellation, the *litharge*, instead of being absorbed by the bone-ashes, surrounds the metallic mass. Should this take place, the operation must be suspended, and the button, if large enough, removed by the forceps from the recrementitious matters; if, on the contrary, it be too small, a portion of pure lead is to be added, and fused with it. In both cases, the operation must be performed upon a newly-prepared cupel.

3. Often, after brightening, a coating of *litharge* remains upon the silver, and, although the metal appears to be pure, cannot be entirely separated. Its expulsion and imbibition are, however, effected, by placing it at a convenient distance from the blowpipe, and submitting it to a powerful oxidating flame. If all the directions given in the preceding pages are exactly complied with, it is possible to obtain accurate results, even from substances containing 0.000752 per cent. of silver.

In cases where it is necessary to find out the quantitative contents of alloys still poorer, several assays of one decigramme should be weighed with the requisite quantity of borax and lead, fused in the previously-described manner, and cupellated like *workable lead*, two or three globules at a time. When this is finished, all the buttons must be united, and again submitted to a new oxidation and cupellation. By this process, a determination of the silver contained in the whole substance is effected, so that, by a simple division of the weight of the button by the number of decigrammes employed, the per-centage of silver in each assay may be found.



*Determination of the Weight of the Silver Globules obtained.*

In order to determine the weight of the pure silver globule, it must be removed from the cupel by the forceps, and freed, as formerly taught, from any adhering *litharge*, then weighed upon the balance. Should it be so small that its gravity can be ascertained with greater exactness upon the scale, it must be very carefully removed from the cupel, that its form may not alter, and measured on the instrument described after the Gold Assay.

As, in the cupellation of a larger quantity of argentiferous lead, a small portion of silver oxidizes, which, combining chemically with the *litharge*, is absorbed by the bone-ashes, I deemed it necessary to ascertain this loss, to ensure an accurate result.

Although this oxidation of the silver takes place, not only in the cupellation, but also in the first process, the loss sustained is much inferior to that occurring in the mercantile assayings, in which all the *litharge* is imbibed by the cupel.

In an assay containing 1 per cent. of silver, it is almost impossible to determine this loss upon the balance; it, however, increases in a relative proportion with the size and nature of the globule. It also varies according to the quantity of lead employed; but remains constant for each per-centage of silver, if the proportions of the lead, and the temperature, are not changed.

The following will show the justness of these observations, in contradiction to those chemists, who suppose that a mechanical separation of the silver is effected.

1. If a button of pure silver, of known weight, be fused in the oxidating flame, with a portion of *litharge* upon a clay basin, and after refrigeration and separation from the scoriæ, re-weighed, a loss of silver will be obvious, although no traces of metallic silver are perceptible in the *litharge*, or upon the basin. If the *litharge* is mixed with a small quantity of soda, and treated in the reducing flame, a plumbiferous globule will be obtained, which, when cupelled, affords a silver button. Gold, when submitted to the preceding treatment,



suffers no loss, and, therefore, no trace of the noble metal is obtained on cupelling the *litharge*.

2. If a weighed silver globule be heated with borax upon charcoal, in the oxidating flame, the borax glass assumes an enamel aspect, after the lapse of some moments, owing to the solution of some oxide of silver. When this glass is heated in the reducing flame, small particles of metallic silver appear upon its exterior. Though this last example does not prove that the loss of silver sustained in the cupellation is chemical, it shows the capacity of this metal to oxidize at a high temperature.

All these reasons induced me to form a table for the use of the assayer, that he might readily detect the quantity of silver lost in any of the alloys. This table is not only adapted for the determination of the loss sustained in the cupellation of substances free from copper, but also for cupriferous alloys, which are not obtained in a pure state by the first addition of lead. I give the proportions, with the requisite quantities of lead for cupellation; viz., instead of 5, 7, 10, 12, and 15 decigrammes (see pages 266, 267),—6, 8, 11, 13, and 16 should be taken (see page 280). The following suggests itself here: whether, in a cupriferous silver ore, requiring, *e. g.*, 15 decigrammes of proof lead, and treated, according to the quantity of sulphur, in the oxidating flame, (by which a part of the lead oxidizes, combining with the scoræ, and another part volatilizes), the loss in the cupellation is the same as if no lead was lost in the fusion? Experience affords the following answer:

If an argentiferous lead be treated with borax on charcoal in the oxidating flame, a small portion of silver oxidizes with the lead, and this loss of silver being in an exact ratio to the loss sustained by "oxidation," where the *litharge* remains on the cupel, this loss of lead must therefore be taken into account. So far as concerns the loss of lead by volatilization, which sometimes amounts to  $\frac{1}{2}$  or 1 decigramme, this need not, however, be considered, as the volatilized portion causes no appreciable change in the cupel loss, on account of the difference produced in such cases, even where the per-centage of silver is very large, not exceeding 0.01, or, at the utmost, 0.05 milligrammes.



*Compensation Table for the Loss of Silver sustained in Cupellation.*

Weight in <i>Milli- grammes</i> of the silver globule obtained by cupel- lation.	If the assay contains, of copper,					If the assay contains under 7 per cent. of copper, or does not contain this metal at all, it must be dressed with,— ( <i>Decigrammes of lead.</i> )				
	80 to 90 ℥ Cent.	60 to 79 ℥ Cent.	30 to 59 ℥ Cent.	10 to 29 ℥ Cent.	7 to 9 ℥ Cent.					
	It is to be dressed and oxidized with, ( <i>Decigrammes of lead.</i> )									
	16	13	11	8	6	5	4	3	2	1
The loss of Silver by absorption amounts, in <i>Milligrammes</i> , to										
99.75 to										
99.5 . .	..	..	..	..	..	0.50	0.45	0.39	0.32	0.25
90 . .	..	..	..	0.83	0.69	0.47	0.42	0.36	0.29	0.22
80 . .	..	..	..	0.75	0.64	0.44	0.39	0.33	0.26	0.20
70 . .	..	..	0.82	0.68	0.58	0.40	0.35	0.29	0.23	0.18
60 . .	..	..	0.74	0.61	0.52	0.36	0.30	0.26	0.20	0.16
50 . .	..	..	0.65	0.54	0.46	0.32	0.26	0.23	0.17	0.14
40 . .	..	0.62	0.55	0.46	0.39	0.27	0.22	0.20	0.15	0.12
35 . .	..	0.57	0.50	0.42	0.36	0.25	0.20	0.18	0.13	0.11
30 . .	..	0.51	0.45	0.38	0.32	0.22	0.18	0.16	0.12	0.10
25 . .	..	0.45	0.40	0.34	0.29	0.20	0.16	0.14	0.10	&c.
20 . .	0.45	0.39	0.35	0.29	0.25	0.17	0.14	0.12	&c.	
15 . .	0.37	0.32	0.28	0.23	0.20	0.15	0.12	0.10		
12 . .	0.32	0.26	0.23	0.19	0.17	0.13	0.11	&c.		
10 . .	0.27	0.23	0.20	0.17	0.15	0.11	0.10			
9 . .	0.25	0.21	0.18	0.16	0.14	0.10	&c.			
8 . .	0.22	0.18	0.16	0.15	0.13	0.09				
7 . .	0.20	0.16	0.14	0.13	0.12	0.08				
6 . .	0.17	0.14	0.12	0.11	0.10	0.07				
5 . .	0.14	0.12	0.11	0.10	0.09	0.06				
4 . .	0.11	0.10	0.09	0.08	0.07	0.05				
3 . .	0.09	0.08	0.07	0.06	0.05	0.04				
2 . .	0.07	0.06	0.05	0.04	0.04	0.03				
1 . .	0.05	0.04	0.04	0.03	0.03	0.02				



Here it is scarcely necessary to observe, that if the operator be not accustomed to blowpipe operations, the cupellation is generally effected at too high a temperature, in which case, even if account be kept in mind of the cupellation-loss, too low a per-centage of the noble metal ensues. It is therefore of the greatest importance to ascertain the proper degree of heat required for the experiment, and the best way to arrive at it is by repeated analyses. I shall give the following as an example:—

A globule of fine silver is exactly weighed, dressed with 5 decigrammes of lead and the requisite proportion of borax, fused in the reducing flame, and the *workable lead* obtained cupellated. If no mechanical loss ensues, and by weighing the button a greater deficiency is indicated than given in the table, the temperature is too high; but if the loss corresponds with that noted in the table, the necessary heat has been employed. The greatest loss is generally sustained in cupellation.

If the balance is so sensible that it indicates a weight of 0.05 milligrammes, the “cupellation-loss” may be carried to the second place of decimals, and calculated for a silver button whose weight is *between* 90 and 100, 80 and 90, or 70 and 80, &c., milligrammes, from the difference of the same; *e. g.*, if, from an assay dressed with 5 decigrammes of lead, a silver button is obtained whose weight is 53.45 milligrammes, as the difference between 50 and 60 is 10, and as 53.45 makes the third part of this difference, so the “cupel loss” for the per centage of silver would be  $0.32 + \frac{0.36 - 0.32}{3} = 0.32 + 0.01 = 0.33$  milligrammes.

If however, the balance is only sufficiently delicate to turn with 0.1, the cupel loss should not be extended to the second place of decimals,—when the number in the second place surmounts 5, it is to be reckoned as 0.1.

It is obvious that the cupellation-loss is not to be reckoned in experiments serving as a control in mercantile examinations, because a larger deficiency of silver results in such operations; nor is it necessary with globules whose weights



are ascertained upon the scale,—the loss sustained in them being so minute that its results may be exceeded by mistakes committed in measuring their diameters.

(b) EXAMINATION OF ORES, MINERALS, AND FURNACE PRODUCTS CONTAINING NO VOLATILE INGREDIENTS EXCEPT CHLORINE.

To this class belong ores consisting principally of earthy matters, and poor in silver,—roasted argentiferous ores,—all silver ores roasted with chloride of sodium, which are to be amalgamated, and the residues of the amalgamated process; Horn-Silver, Earthy Silver Glance,\* argentiferous slags, and also the dross of gold- and silver-smiths.

Of the desiccated and pulverized substance,  $\frac{2}{3}$  of a decigramme are weighed, and dressed with 1 decigramme of borax and 5 decigrammes of proof lead. If, however, the assay contains copper, the proportion of lead must be augmented in proportion to the quantity of the former metal. The whole is then enveloped in a “soda-paper cornet,” and pressed into a prepared charcoal cavity.

As in this operation few constituents are present which are to be combined with and then separated from the lead, and as the process consists principally in forming an argentiferous button, and vitrifying the earths and difficultly-reducible metallic oxides by means of the borax, the assaying is effected without the slightest inconvenience. The sample is only to be submitted to the reducing flame until the lead has completely alloyed with the silver, forming a molten button, and the recrementitious matters are in full fusion. (The same precautions are to be observed as in the fusion of ores containing volatile ingredients.) By this procedure, the earths and

\* [In “Phillips’ Introduction to Mineralogy” is the following on Earthy Silver-Glance (Black Sulphuret of Silver):—“It is dark lead-grey, inclining to black, and without lustre, or only feebly glimmering; it occurs massive and pulverulent, sometimes investing other ores of silver, and filling up cavities in them; fracture uneven; is more or less sectile; and gives a shining metallic streak. Before the blowpipe it is converted into a slaggy mass, containing globules of impure silver. It occurs in the veins of primitive mountains with other ores.”]—*Trans.*



difficultly-reducible metallic oxides are vitrified, the easily-reducible ones, *e. g.*, the oxide of lead, of roasted lead ores, &c., reduced and combined with the proof lead, the chloride of silver decomposed, and the chloride of lead formed volatilized as a white vapour, part of which coats the charcoal; the silver fuses, and alloys with the lead, forming *workable lead*.

When the operation is terminated, the *workable lead* is allowed to refrigerate, then detached from the scoriæ, and beaten into a cube upon the anvil. The oxidation, cupellation, and the weighing or measuring, are performed in the ordinary way.

Should the substance contain more volatile ingredients than chlorine, the *workable lead* will appear of a black or dark grey colour; when this occurs, it ought to be re-fused with the scoriæ for some minutes in the oxidating flame, in order to expel any sulphur or arsenic that may be present. Ores containing very small portions only of real silver, present no black aspect, as the minute quantities of sulphur and arsenic are evolved on the first application of the heat.

(c) EXAMINATION OF FURNACE PRODUCTS, CONSISTING  
OF METALLIC OXIDES WHICH ARE EASILY  
REDUCIBLE UPON CHARCOAL.

The most prominent of these are the *litharges* and the *abstrichs*.\*

Their content of silver is generally inconsiderable, and often cannot be ascertained in an assay of 1 decigramme. As, however, these substances consist of no foreign metal

\* [If the *workable lead* resulting from plombiferous ores, be cupellated for silver, several sorts of *litharge* are obtained. The first appearing immediately after the fusion of the substance, is the *abzug*, which adheres to plombiferous minerals. The second are termed *abstrichs*, or *black litharges*, and present themselves when the molten mass is exposed to the blast; their aspect changes from black and metallic, to yellow and yellowish-grey, and they contain all the easily-oxidizable metals; the metal generally predominating in them is antimony. The *red litharges* formed after the *abstrichs*, until the termination of the process, consist of nearly pure oxide of lead, contaminated only by traces of silver and oxide of copper.]—*Trans.*



besides lead, whose reduction is easily effected, their quantity of silver is readily determined.

Five decigrammes of the body are weighed, pulverized, and dressed with 50 milligrammes of soda, and 50 milligrammes of borax, enveloped in a cornet of soda-paper, placed in a charcoal cavity, treated with the reducing flame, until all the oxide is reduced, and the scoriæ, free from lead, are perfectly liquid.

The globule obtained from the *litharge* is generally exempt from volatile metals, and contains but a small proportion of copper; whereas, that obtained from the *abstrich* is usually contaminated with antimony, arsenic, copper, zinc, &c. These ingredients, however, disappear, if the scoriæ are treated for a long time with the reducing flame. The oxidation and cupellation are effected in the ordinary way. The weight of the button is determined by the scale.

(d) EXAMINATION OF MINERALS, WHICH ARE NOT  
DECOMPOSED BY BORAX OR LEAD UPON  
CHARCOAL.

Up to this time no mineral can be arranged under this head, except Molybdena-glance. It contains 0.176 per cent. of silver.

For analyzing this mineral, I found the following flux the most satisfactory:—

Take 1 decigramme of the specimen, and dress it with—

Soda, 150 milligrammes;

Borax, 150 milligrammes;

Lead, 5 decigrammes.

In a cornet of soda-paper inserted in a cavity in the charcoal, place the assay, and subject it to a strong reducing flame. The soda decomposes the mineral, sulphuret of sodium is formed, and the liberated molybdenum partly combines with the lead, and partly volatilizes, coating the charcoal with a white sublimate. When the liquid scoriæ flows quietly, it is a sign that the decomposition is terminated. The argentiferous globule must now be exposed to the air, and treated with the reducing flame, until all the molybdenum (which



gives a white and brittle alloy with the lead,) is expelled. The addition of the borax is to prevent the recrements from dilating upon the support. The *workable lead* obtained, is then oxidated and cupellated.

### ¶ B *Metallic Compounds.*

#### (a) EXAMINATION OF THOSE HAVING SILVER AS A PRINCIPAL CONSTITUENT.

Among these rank native silver, *blicksilber*, *brandsilber*, *amalgamsilber*, cupellated silver, and standard silver. The operation for these consists only in fusing with pure lead, and cupellating. The quantity of the substance taken, may vary from 80 to 100 milligrammes, as it is very difficult to obtain a piece of a certain weight. On account of such compounds being obtained with great difficulty in small particles,\* as foreign matters on the surface of the sample might prevent exactness in the results, they should be scraped off previously to weighing, and treated on charcoal. If native silver, *blicksilber*, or *brandsilber*, is to be assayed, 1 decigramme of lead and 50 milligrammes of borax, are requisite for cupriferous amalgams and standard silver,—however, the proportion of lead varies from 2 to 5 decigrammes, according to the quantity of copper. The mixture is fused in the reducing flame, until the noble metal becomes alloyed with the lead, and the borax appears perfectly free from plumbiferous particles. The assay is then allowed to refrigerate, and the button removed and separated from the flux. Though the addition of borax is not absolutely required, still it prevents any violent action ensuing, which, if occurring, would falsify the results.

\* As the examination is not effected upon exactly 100 milligrammes, a particular calculation is necessary for deducing the per-centage of the assay from the weight of the treated substance; *e. g.*, 85.5 milligrammes were treated with 200 milligrammes of lead, from which 83.6 milligrammes of pure silver were obtained, the cupellating loss of which is 0.27 milligrammes, therefore the following proportion is indicated:—

$$85.5 : 83.6 + 0.27 :: 100 : x.$$

$$x = \frac{(83.6 + 0.27) 100}{85.5} = 98.09 \text{ per cent.} \text{—Author.}$$



Such alloys as require but 1 to 2 decigrammes of proof lead, are assayed upon the cupel. Cupriferous alloys, however, treated with 3 to 5 decigrammes of lead, must first be submitted to oxidation, to dispel the greater part of the copper, and then cupellated.

The fine silver globule is, after termination of the process, removed from the cupel, struck slightly with the hammer upon the anvil, and then weighed upon the balance.

(b) EXAMINATION FOR SILVER IN METALLIC COMPOUNDS  
CONSISTING CHIEFLY OF GOLD.

Under this head is placed Native Gold, and alloys consisting of gold, silver, and copper.

In these the per-centage of silver is readily determined at the same time with that of gold. Hereafter I shall specify the procedure under the Gold Assay.

(c) EXAMINATION FOR SILVER IN ALLOYS WHEREIN  
COPPER AND NICKEL PREDOMINATE.

These compounds are:—*black copper* and *gaarkupfer* affined on a large scale; copper coins containing silver; brass, bell-metal, german silver, &c.

For procuring quantities sufficient for weighing, the alloy must be ground with a strong file, and when *gaarkupfer*, *black copper*, coined copper, or german silver are assayed, the following proportions must be employed:—

Substance,	1 decigramme ;
Lead,	15 decigrammes ;
Borax,	1 decigramme.

The dressed sample is then wrapped in a soda-paper cornet, and inserted in a cavity of charcoal and treated with the reducing flame directed upon the assay, until it has been some time in fusion, and all metallic particles have disappeared from the surface. In this operation, cobalt, iron, and zinc oxidize; the first two dissolve in the borax, whereas the zinc volatilizes.

The *workable lead* obtained, which contains copper or nickel, or both metals, may, without being cooled, be brought



upon the steel anvil. This spares the detaching of the scoriæ. It is now to be cupellated in the common way, but if the globule produced contains copper, a dilation ensues, which will prevent a determination of its weight upon the scale; this is, however, remedied, by adding 50 milligrammes of lead, and re-cupellating.

When brass or bell-metal is to be assayed, the following proportions must be taken :—

Alloy, 1 decigramme;

Lead, 10 decigrammes;

Borax, 1 decigramme.

The assay is fused like the former, in the reducing flame, until the borax is exempt from metallic particles. The flame is then directed only upon the flux, to allow the atmospheric air access to the lead, to oxidate the tin, and the non-volatilized portions of zinc. The oxide of tin combines with the borax; the oxide of zinc is entirely dispelled. If, then, the surface of the assay appears bright, it must be poured upon the anvil. The subsequent operations are conducted according to the method prescribed for cupriferous substances.

(d) EXAMINATION OF METALLIC COMPOUNDS, THE  
PREDOMINATING METALS BEING LEAD OR  
BISMUTH.

To this class appertain *workable lead*, prepared on a large scale, revived lead, argentiferous bismuth, &c.

Of these alloys, the *workable lead* is the richest in silver. A portion is to be laminated, cut into shreds with the scissors, 2 decigrammes weighed out, placed upon a cupel, fused in a moderate oxidating flame, and cupellated immediately. If little or no copper is present, a bright and orbicular globule will be obtained, but when much copper is contained in the alloy, dilation ensues, without any external brightening; should this occur, 1 decigramme of lead ought to be added, and the operation terminated upon another part of the cupel. The weight of the button obtained, divided by two, gives the per-centage of silver for 1 decigramme.



As the *workable lead* is a furnace product requiring a further treatment, in which a loss of silver also takes place, it is evident that the cupellating loss in this operation need not be taken into consideration. If, however, the per-centage of silver is to be determined exactly, 5 decigrammes of such lead ought to be weighed, fused, and refined in two periods, and if, owing to too large a quantity of copper, the silver globule is not yet sufficiently pure, it is to be re-cupellated with one decigramme of proof lead. To the weight of the fine silver globule thus obtained, the amount of the cupellating loss, answering to the oxidized quantity of lead, is added, by which, and division of the sum by 5, the real per-centage of silver for 1 decigramme of the examined *workable lead* will be found.

If revived lead or bismuth is to be examined for silver, the former is cut into shreds like the *workable lead*, but the latter, on account of its brittleness, must be pulverized under the hammer. As no high per-centage of silver can be expected in these compounds, an assay of 5 decigrammes ought generally to be employed. The weighed quantity of the alloy must either be immediately introduced, like *workable lead*, into a good cupel and fused, or in case the assay consists of a number of small particles, they should be first fused together on charcoal, and the globule removed from the support in two portions.

The separation of the argentiferous bismuth bead, which is to be subsequently cupellated from the oxide of bismuth formed during the oxidation, should be effected with great care, in order to prevent particles of the brittle material remaining. The globule ought never to be raised from the accumulated oxide, but the latter always detached by means of the pincers.

The pure globules obtained in the cupellation are to be measured in the scale, and the content of silver in 1 decigramme calculated from the weight found.

(e) EXAMINATION OF METALLIC COMPOUNDS IN WHICH  
ANTIMONY OR ZINC PREVAIL.

The subsequent observations apply particularly to argentiferous antimony or zinc.



If the antimony contains some per cents. of silver, it can be treated on charcoal in the oxidating flame; the antimony volatilizes, and the silver remains as a dull bead. If, however, this does not happen, the silver may probably have been carried away by the current of air, and consequently the following operation must be performed.

Weigh 1 decigramme of the alloy and fuse it upon charcoal in the reducing flame with 2 decigrammes of proof lead and a small quantity of borax, and subject the metallic button to the oxidating flame, to expel the antimony or zinc. Oxidation of the lead does not commence until nearly the whole of these metals are volatilized. When the fumes cease, the blowing must be discontinued, the *workable lead* allowed to refrigerate, then separated from the scoriæ, and cupellated. This treatment is preferable for antimony, even when much silver is present, because, without an addition of lead, the last traces of antimony are expelled but imperfectly from the silver, and often a minimum of this metal might remain; besides, if other metals, *e. g.*, copper or iron, are present, these are segregated by the lead in cupellation.

(f) EXAMINATION OF METALLIC COMPOUNDS IN WHICH  
TIN IS THE PRINCIPAL INGREDIENT.

*E. g.*, Argentiferous tin.

As tin cannot be separated from silver, either by cupellation or volatilization, upon charcoal, it must be treated in the following manner:—

Weigh, of the substance to be examined, 1 decigramme, and dress it with—

Lead, 5 decigrammes,  
Soda, 50 milligrammes,  
Borax, 50 milligrammes.

Envelope the assay in a cornet of soda-paper, place the whole in a prepared charcoal cavity, and heat in a strong reducing flame, until the metals form an orbicular alloy, and the borax and soda (the latter is employed to prevent an oxidation of the tin,) are vitrified. When this is accomplished, the metallic globule alone is to be touched with the blue flame,



(care being taken to prevent a violent oxidation of the tin), so that the glass may imbibe all the oxide formed. If reduced particles of tin appear upon the sides of the scoræ, discontinue the blowing, and allow the assay to cool. The refrigerated globule, to which 1 decigramme of borax is to be added, must now be treated upon another piece of charcoal, first with the reducing flame, and if perfect fusion is effected, in the oxidating flame, until it presents a bright surface. The *workable lead*, rendered by this operation free from tin, is submitted to oxidation and cupellation, and the per-centage of silver ascertained upon the balance, or if too small, upon the scale.

(g) EXAMINATION OF METALLIC COMPOUNDS, WHOSE  
PRINCIPAL INGREDIENT IS MERCURY.

To this class belong, native and artificial amalgams of silver, and argentiferous mercury.

One decigramme of the substance is weighed, placed in a small glass tube, with a bulb at one end, as in *Fig. 44, page 179*. The tube is held inclined, and heated gradually over a spirit-lamp. A very moderate heat volatilizes the greater part of the mercury, which deposits gradually in a metallic state, upon the upper part of the tube. The bulb must now be heated to redness, and kept in this state until no more mercury is eliminated, when the tube is allowed to cool. After refrigeration, by slightly agitating the vessel, the globules of mercury readily cohere, and the main globule formed, can be poured out, by gradually inverting the tube.

If the substance submitted to this operation was an amalgam, the silver remains in the bulb as a porous bead, and can readily be extracted. This globule must be fused in the reducing flame with borax, and 1 decigramme, or, if it contains copper, 2 to 3 decigrammes of proof lead, and the formed *workable lead* treated in the usual way. The weight of the silver globule obtained is then ascertained upon the balance, and if a native amalgam has been employed, the cupellating loss deducted.



However, if the distilled alloy be argentiferous mercury, the residuum in the tube is very inconsiderable, and cannot be detached from the bulb without great difficulty. In this case, the greater part of the tube must be cut off with a file, and what remains in the bulb dressed with 1 decigramme of lead and 50 milligrammes of borax, and the whole placed in a charcoal cavity and submitted to a strong reducing flame, until the metallic compound exudes from the glass. The argentiferous lead is, after cooling, easily separated from the glass and charcoal, and must be refined upon a well-burned cupel of levigated bone-ashes, and the resulting silver globule measured upon the scale.

The cupellation is necessary in both cases, (1,) because the silver cannot be fused in the tube, and consequently all the mercury is not eliminated; (2,) because the amalgam obtained from amalgamated ores often contains various metals, which remain in the distillation and can only be separated from the silver by cupellation. If, however, gold be extant in the assay, it cannot be segregated in this way, but must be treated in the manner which I shall give under the examination for that metal.

(h) EXAMINATION OF METALLIC COMPOUNDS WHOSE  
PRINCIPAL INGREDIENT IS IRON OR STEEL.

As iron or steel cannot be united immediately with lead in the blowpipe-flame, their union must be effected in an indirect manner. It is generally known that sulphuret of iron combines with lead, if these bodies are treated with borax in the reducing flame; and ultimately, by heating in the oxidating flame, sulphur volatilizes, and the iron oxidizes and is absorbed by the borax, either in the state of a pro- or a peroxide. Therefore, if iron or steel is united with sulphur, the silver contained in the mineral may be separated as easily as if argentiferous sulphuret of iron, &c., were assayed.

When hardened steel is to be examined for silver, it must first be submitted to a red heat, allowed to refrigerate, and, when cold, its surface cleansed, and about the necessary quantity for an examination taken off it with a file. One



decigramme is then to be weighed and dressed with—

Sulphur, 50 milligrammes,

Lead, 8 decigrammes,

Borax, 1 decigramme.

The whole must then be enveloped in a soda-paper cornet, placed in a cylindrical charcoal cavity, and heated in the reducing flame until the assay forms a molten bead. By this treatment the sulphur combines with the lead and iron.

As one decigramme of borax is not sufficient for imbibing the whole oxide of iron formed in the oxidation following the fusion, another decigramme of the flux is to be added, the whole re-fused, and submitted to a very strong oxidating flame until the impure lead begins to exude from the glass. The assay must now be held so that the lead is in contact with the flame,—the sulphur volatilizes, the iron oxidizes, and the formed oxide combines with the borax. After elimination of the sulphur, and segregation of the iron, the blowing is to be interrupted, and the lead, presenting a bright surface, and containing all the silver of the iron, allowed to refrigerate. If, when cold, it be of a whitish colour, it must be treated like an ordinary *workable lead*, and the weight of the silver globule ascertained; if, however, it be brittle, and of a blackish aspect, it should, before cupellation, &c., be submitted to a re-oxidation.

## 2. THE GOLD ASSAY.

Gold can be separated in the dry way, like silver from substances in combination.

As it is not susceptible of oxidation, even when treated with borax or lead in the outer flame, and sustains no loss in the cupellation, its per-centage in ores, minerals, furnace products, &c., may be ascertained with the greatest exactness. The examination for gold, however, is more difficult than for silver, as, in most instances, gold, submitted to blowpipe analyses, contains a quantity of the latter metal, which in native gold varies from 2 to 35 per cent.



Concerning the quantity of gold in several silver ores, I have had occasion to examine all the ores dressed in the Freyberg works, and the results obtained are the following, viz.—That all sulphurets of iron holding from 0·0142 per cent. to 0·456 per cent. of silver, contain gold. This metal forms an ingredient also of the furnace-products which are produced in the smelting of auriferous silver ores.

The various auriferous substances, are brought, according to their nature and composition, under several heads, viz.:—

¶ Gold Ores, Auriferous Silver Ores, and furnace products containing Gold and Silver.

¶ Metallic Compounds, consisting of—

- (a) Gold and Silver.
- (b) Gold and Silver, alloyed with other metals.
- (c) Gold and Mercury.

¶ A *Examination of Gold Ores, Auriferous Silver Ores, and furnace products containing Gold and Silver.*

Under this class are ranged :—

1. Native Tellurium, containing, according to Klaproth, 0·25 per cent. gold, but no silver.

2. All ores and minerals containing silver, and also gold; *e. g.*, Graphic Tellurium (Graphic Gold), the composition, according to Klaproth, is 60 tellurium, 30 gold, and 10 silver;\* Yellow Tellurium (Weiss-silvanerz), containing, according to Klaproth, 44·75 tellurium, 26·75 gold, 19·50 lead, 8·50 silver, and 0·50 sulphur; Black Tellurium, analysis by Klaproth, 32·2 tellurium, 54·0 lead, 9·0 gold, 0·5 silver, 1·3 copper, and 3·0 sulphur; Noble Molybdena Glance, sent me by Professor Breithaupt, I found to contain 4·9 gold, and about 0·3 silver.

3. All argentiferous sulphurets of Iron and Copper.

4. *Roh-* and *Blei-stein*.

5. The auriferous dross of gold- and silver-smiths.

\* [According to Berzelius, it contains 52·0 tellurium, 24·0 gold, 11·3 silver, and 1·5 lead.]—*Trans.*



Of gold ores, containing little or no silver, a quantity sufficient for one examination is prepared, and treated in the same manner as an assay of silver. After cupellation, the colour of the globule must be observed, for if only 2 per cent. of silver were present, the gold would acquire a brass colour. If the gold be pure, which is known from the colour, its weight may be determined either upon the balance or the scale. When its colour is very light, it must be submitted to a new treatment, which I shall hereafter describe.

Of substances containing more silver than gold, a portion of 10 to 15 decigrammes must be reduced to powder, dressed, and treated as in an examination for silver. From the quantity of silver obtained, the number of assays necessary for obtaining a sufficient quantity of gold, to make a quantitative determination, is calculated. Should the substance contain not more than 0.114 per cent. of silver, a considerable quantity of it ought to be pulverized, and at least 2.5 grammes taken; on the contrary, if the per-centage be larger, *e. g.*, 0.29 per cent., 10 to 15 decigrammes are sufficient for an examination. The dressing of the assay with lead and borax is effected in exactly the same manner as an argentiferous one, but when copper is present, the proportion of lead must be augmented. The fusion and cupellation can also be achieved in a similar way to the assaying of a silver alloy. I shall refer to the further treatment.

Sulphurets or minerals containing large quantities of sulphur, and little silver, are analyzed in the following way: 24 to 36 decigrammes of the pulverized substance are divided into parcels of 3 decigrammes each, placed in a clay basin, (see *Fig. 12, page 12*), the inside of which is covered with a coating of redde, and roasted without addition of charcoal, like a copper assay.

When nearly all the sulphurous vapour is eliminated, the assay is to be re-powdered, and heated until no appreciable quantity of sulphur remains. This being accomplished, the residuum must be placed in a porcelain basin over a lamp, and treated with a sufficient quantity of hydrochloric acid to dissolve out the formed oxide of iron or copper. The metallic



oxides and the silver being dissolved, the gold, with some extraneous matters, will remain. The solution is then to be evaporated to dryness, and treated with boiling water. The soluble chlorides dissolve, and are to be separated from the chloride of silver, &c., by filtration and edulcoration. The filter holding the insoluble ingredients is to be dried, and after desiccation opened and dressed cautiously with 5 decigrammes of lead and 1 decigramme of borax. The dressed assay must now be placed in a charcoal cavity, submitted to a feeble oxidating flame in order to carbonize and consume the filter, and then treated with a strong reducing flame. In this operation the chloride of silver is decomposed, its base alloying with the lead and gold, the earthy constituents being dissolved in the borax. The metallic alloy is oxidized and cupellated in the ordinary way, and the auriferous silver globule obtained, treated according to the described method for the separation of gold from silver.

It sometimes happens that sulphurets containing less than 4 loths,\* 0.114 per cent. of silver, are to be analyzed. In this case, 36 decigrammes are not sufficient, therefore, according to the per-centage of the noble metal, the following proportions must be taken, viz.—Of a substance containing:—

3 loths = 0.0939 per cent. of silver, 48 decigrammes.

2 „ = 0.0616 „ „ 72 „

1 „ = 0.0370 „ „ 144 „

$\frac{1}{2}$  „ = 0.0142 „ „ 288 „

As the weighing of a quantity of nearly 290 decigrammes in portions of 3 decigrammes each, would be very troublesome, it is sufficient to weigh upon an ordinary balance, according to the apothecaries' weight, of an ore containing—

3 loths of silver in the cwt., 80 grains;

2 „ „ „ 120 „

1 „ „ „ 240 „ and

$\frac{1}{2}$  „ „ „ 480 „

and to pulverize it well.

For the same reason, as it would exceedingly inconvenient

\* 1 loth = 14.58808 grammes = 239.68 English grains.



to roast a substance in parcels of three decigrammes each, the following expert method may be undertaken:—

Upon an iron foil, bent into a basin shape, place the whole assay, besmeared several times with clay-water, and well dried; and submit it to a strong red heat, continually stirring with a platinum spatula until no more sulphurous acid vapours are evolved, then triturate the residuum in a mortar, and subject the powder to a second roasting.

The assay, after these operations, must be introduced into a porcelain basin, placed over a spirit-lamp, treated with muriatic acid, evaporated to dryness, and then mixed with a quantity of water to dissolve out the soluble chlorides, the menstruum filtered, what remains on the filteredulcorated, desiccated, and if it exceeds 1 decigramme, weighed in parcels of 1 decigramme each.

Each decigramme of this residue is to be dressed with

Lead, 5 decigrammes,

Borax, 1 decigramme,

fused like an argentiferous assay, and cupellated as already stated. The treatment of the auriferous globule I shall describe in the subsequent paragraphs.

#### ¶ B *Metallic Compounds.*

##### (a) EXAMINATION OF SUCH AS CONTAIN SILVER AND GOLD, ONLY.

To these belong, native gold, alloys of gold and silver, and the argentiferous gold, or auriferous silver, obtained from the assayings of auriferous minerals and ores.

As, up to the present time, no process is known for separating gold from silver in the dry way, the moist way, termed in this instance “Quartation,” or “Refining of Gold and Silver,” must be employed.

For effecting the separation, the proportion of the gold to the silver should not exceed 1 to 3, as, if more gold be present, the solution will be performed either imperfectly or not at all.\*

\* [When the gold is more than the above proportion, it so protects the silver, that even when hot fuming nitric acid is poured over the alloy, a very slight action only ensues.]—*Trans.*



It is therefore indispensable to make a preparatory assay for determining the composition of the alloy, and if too small a portion of silver be present, add a quantity sufficient for producing the given standard.

Gold containing even no more than 2 per cent. of silver, is, as has been previously stated, of a brass colour; if 30 per cent. of silver are present, it appears lightly brass-coloured, but when the quantity of silver amounts to 60 per cent., a yellow colour is no longer perceptible. Therefore, from the intensity of the tinge in an auriferous alloy, conclusions as to its composition, and the necessary quantity of silver, can be drawn.

When native gold of a brass colour is to be assayed, it is generally supposed that the per-centage of silver associated with it is not high; in this case, 20 to 30 milligrammes of the alloy are weighed, fused with ( $3 \times 20 = 60$ ), to ( $3 \times 30 = 90$ ) milligrammes of pure silver, (reduced from the chloride of silver,) and a small quantity of borax, in the reducing flame. Of a light-coloured auriferous body, 50 milligrammes are weighed for examination, and alloyed in the same manner with  $50 \times 2 = 100$  milligrammes; or, if the per-centage of silver does not amount to 30 per cent., with 110 to 120 milligrammes of pure silver. In alloys presenting a bright white aspect, and composed of about 40 gold and 60 silver, the per-centage of the latter metal cannot be previously known; therefore 1 decigramme of the alloy should be fused on charcoal, with 60 to 80 milligrammes of pure silver, and a small addition of borax. In compounds of gold and silver obtained from the assaying of minerals or gold ores, gold is generally prevalent, necessitating their fusion with double or triple their weight of pure silver. On the other hand, the composition of alloys produced from roasted ores or sulphurets, are nearly always in the proportion of 25 gold to 75 of silver; therefore, such compounds may be analyzed without an extra addition of the latter metal.

After weighing, according to the quality of the alloy, 20 to 100 milligrammes, and fusing upon charcoal with the necessary quantity of silver, the produced globule must be placed upon an anvil, laminated, re-heated upon charcoal,



introduced into a small porcelain vessel (*see Fig. 40, page 28,*) supported over a lamp, treated with pure nitric acid, and covered with the watch-glass (*a*) to prevent an elimination of nitrous fumes. When the solution of silver is complete, the whole must be filtered and affused three or four times with hot distilled water, to remove the last traces of the nitrate of silver. The metallic gold remaining on the filter must be then dried, heated to redness in a platinum crucible, and weighed; but should the quantity be too minute for the balance, it must be alloyed with lead, cupellated, and its weight determined upon the scale. One decigramme of lead will be sufficient for the dressing, and a small quantity of borax. The affining of the auriferous globule is often very difficult, owing to the proportion of lead present, but a little practice will greatly facilitate the assayist in his experiments. For the purpose of rendering the calculations of per-centage of gold in alloys more easy, I annex the following example:— If an alloy of 30 milligrammes has been assayed, and the product of gold obtained, 25·5 milligrammes, we have,—

$$30 : 25\cdot5 = 100 : x = 85 \text{ per cent. of gold.}$$

If in native gold, or in an artificial alloy, the per-centage of silver is also to be ascertained, the assay must be cupellated (before fusion with fine silver) with 1 to 2 decigrammes of proof lead, to segregate any other metals, such as iron or copper: after affining, weighed; and the per-centage of silver calculated from the difference, provided the quantity of gold has been ascertained.

(*b*) EXAMINATION OF METALLIC COMPOUNDS,  
CONTAINING OTHER METALS BESIDES  
GOLD AND SILVER.

The most numerous of these are gold, alloyed with copper and silver.

Of this alloy, 30 to 50 milligrammes are to be weighed, fused according to the per-centage of copper, with 3 to 8 decigrammes of proof lead, and an addition of borax, upon charcoal, in a good reducing flame; then affined like a cupriferous *workable lead*. The copper oxidizes with the lead,



the gold and silver remaining pure. If, however, after cupellation, the gold globule does not appear sufficiently pure, owing to a trace of copper, the bead ought to be immediately re-fused with 1 decigramme of lead, and again affined upon a clear portion of the cupel.

The other observations agree with those described in the preceding pages.

(c) EXAMINATION OF METALLIC COMPOUNDS,  
CONSISTING OF GOLD AND MERCURY.

Although these compounds are not found in nature, they are produced in auriferous amalgamations. To effect their analyses, 50 milligrammes ought to be distilled in a glass tube, (like amalgams of silver), and the residue cupellated with 1 decigramme of lead, and the refined globule weighed. If its colour be very white, it is a proof of the presence of silver; and, therefore, it must be submitted to "Quartation," which has been previously described, and the quantity of gold and silver determined. Of poor amalgams of gold and silver, several samples of 1 decigramme should be introduced into a glass retort, and distilled, and when the mercury is eliminated, new quantities of the amalgam ought to be added, and distilled, until the residue of argentiferous gold is sufficient for weighing. The operation being terminated, the gold globe is to be weighed,—and, if necessary, fused with 2 to 3 parts of pure silver; treated with nitric acid; and the weight of the pure gold determined, from which the proportion of silver may be calculated.

The weight of the gold and silver is to be divided by the number of decigrammes of mercury submitted to distillation, whereby the per-centage of the two metals in 1 decigramme of the assay under examination, is obtained.

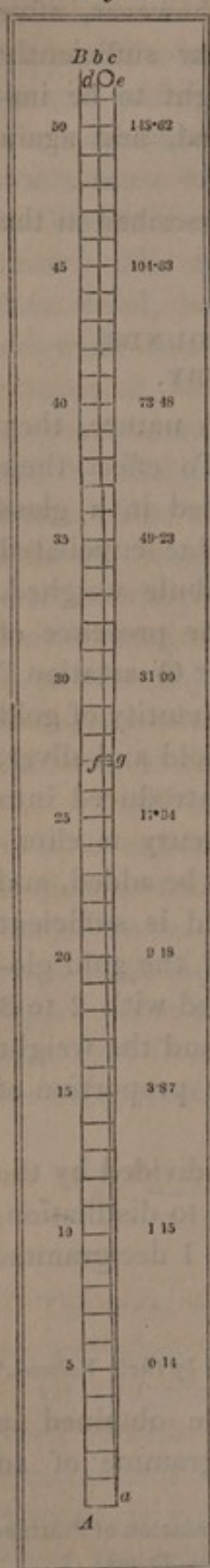
1. *Determination of the Weight of Gold or Silver Globules by their Volume.\**

It often happens that the affined globule obtained in quantitative blowpipe analyses, from 1 decigramme of an

\* [The description of the Scales is from the French translation of Plattner by my friend Dr. Sobrero, of Turin,—as it is more lucid.]—*Trans.*



Fig. 47.



argentiferous or auriferous substance, is so small, that its weight cannot be determined exactly, even upon the most delicate balance; on the contrary, quantities sufficient to compensate for this poverty, could not be operated upon, without depriving the blowpipe examinations of their most important feature, viz.—their simplicity and promptitude of execution. In order to avoid this difficulty, Harkort endeavoured to calculate the per-centage of any assayed substance by measuring the diameter of the metallic globule obtained. Experience has proved the correctness of this method, notwithstanding the differences arising from inequalities in the form of the globules. To give an idea how this design was effected, I shall describe the instrument employed by Harkort, and afterwards modified by Plattner.

*Harkort's Scale.*—Fig. 47 represents a well-polished ivory plate, upon which the line  $AB$  is drawn, parallel with one of its sides; then at a short distance from  $AB$  are two other lines,  $ab$  and  $ac$ , convergent at  $a$ , and forming an acute angle. The line  $AB$  is divided into equal sections by small parallel lines, which cross the convergent lines  $ab$ ,  $ac$ , forming the bases of so many triangles, similar to the large one,  $bac$ ; all these triangles possess a common angle, the sides opposite to which are parallel to one another, consequently a proportion between these homologous sides can be established: *e. g.*, between the large isocles triangle,  $bac$ , and the small one,  $fac$ , there exists the proportion:—

$$ab : af :: bc :: fg, \text{ and } :: ac : ag.$$



According to the above, the space comprised between the two lines  $ab$  and  $ac$  may serve for the determination of the volume of a sphere, and consequently of its weight.

If we suppose that a spherical metallic button be placed upon the scale between the lines  $ab$ ,  $ac$ , and moved towards  $a$ , until the convergent lines from it become tangents, its diameter will be ascertained and expressed by the line uniting both tangent points. If, for instance, the button is found to correspond with the space between the two lines  $de$ , its diameter is thus represented; and another globule, introduced into the scale, and touching the lines at  $fg$ , would have the diameter of this part.

The position of the two metallic globules being ascertained, and their distances from the point  $a$  established, by the divisions upon the line  $AB$  with which they correspond, it is easily conceived how, from the relative proportions of their diameters, their weights may be ascertained. It will be as well here to remark, that the weight of homogeneous spheres is in direct proportion to that of their volumes, and the volumes are as the cubes of their diameters. Therefore, if  $L$  and  $l$  be the distances between the small globes and the point  $a$ ;  $V$  and  $v$  the two volumes,  $W$  and  $w$  the two weights, we have—

$$V : v :: (de)^3 : (fg)^3;$$

and because

$$de : fg :: ad : af :: L : l$$

we have

$$V : v :: L^3 : l^3$$

and consequently, the proportion of the weights to the volumes—

$$W : w :: L^3 : l^3$$

Though this proportion, indicating only the relation existing between  $W$  and  $w$ , gives no value to these quantities, it is evident that if the value of  $W$  be fixed, that of  $w$  is immediately established, according to the following equation:—

$$w = \frac{W \times l^3}{L^3} \text{ or } = \frac{W}{L^3} \times l^3$$

In order to give a known value to  $W$ , the weighing of the



button *d e* is sufficient. Harkort, therefore, selected an ore of a common per-centage, and having obtained equal results from the different globules, after repeatedly cupellating, he weighed the most regular, and found that its weight corresponded to a content of 154 loths in a cwt.,\* or 4.354 per cent. He placed it upon the scale, and finding that it agreed exactly with the number 52, of the line *A B*, he inscribed there the weight indicated by the ball corresponding with this point, thus establishing a standard of comparison for smaller globules†. The first point of the proposition being established in this manner, it is evident that by introducing the value of *W* into the formula,  $w = \frac{W}{L^3} \times l^3$ , the weight of any ball *w* may be determined. It is also clear that the constant factor  $\frac{W}{L^3}$  remains the same in all the different values of *w*; and therefore, the only care to be taken, is, to determine the exact position of the globule upon the scale,—consequently, the distance *l* between it and the point *a*: for effecting this, the button is held between a pair of forceps, placed between the two lines on the scale, and then moved until its tangent point is ascertained. In order to accomplish this exactly, a good magnifying glass is requisite,—also a perpendicular direction from the eye to the scale.

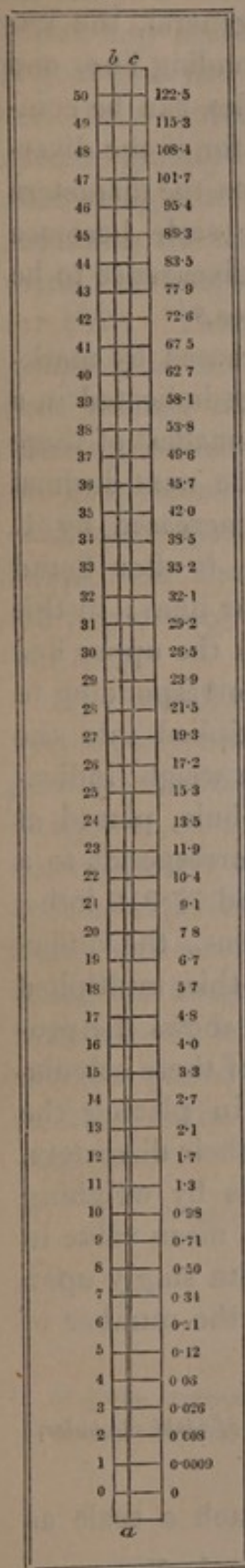
Although in such an operation it is supposed that the globules are always orbicular, and of the same specific gravity, this is not the case, because during the fusion the globules are compressed by virtue of their gravity, and conserving their flat form after refrigeration; also the degree of density is dependent upon the time allowed for cooling. However, in the limits between which the scale may be employed, the influence of temperature, as well as the form of the globule, are of no avail, as Harkort has ascertained from numerous carefully-conducted experiments.

\* [The Saxon cwt. = 110 pounds, each of which is divided into 32 loths. As Plattner graduated his scale according to this division, it is retained, care being taken to give also the expression in per cents., in order to avoid any mistakes.]—*Trans.*

† [This is the reason why the gradation of the scale (*Fig. 47*) begins with the number 52.]—*Trans.*



Fig. 48.



*Plattner's Scale*—Its use in quantitative silver assaying. The examinations for silver described in the preceding pages, being effected from 1 decigramme, (a standard representing the cwt. in small operations,) Plattner constructed a scale in order to use it for metallic globules obtained from an assay of this weight. He triturated, therefore, intimately, a quantity of *ruby silver*, with an equal quantity of a poor calcareous silver spar, and cupelled the mixture. He repeated this operation several times, and constantly found 122.5 loths, or 5.48 per cent. silver. He then submitted the assay to the blowpipe, and obtained the same result.

The annexed wood-engraving (*Fig. 48*) is his scale, divided into 50 parts, the first of which answers to the globule obtained from 1 decigramme of an ore containing 5.48 per cent.\* The constant factor  $\frac{W}{L^3}$  is therefore equal to 0.00098 for the division in loths; and  $\frac{5.48}{50^3} = 0.00004384$ , if the content be expressed decimally.

It is evident that a scale so constructed might be greatly extended, in order to serve for measuring metallic globules of a far greater volume than what is indicated in the foregoing remarks. This, however, is inconvenient; as the weight of spheres varies directly as the cubes of their diameters, the differences of weight for each division of the scale increase considerably,—therefore, a button having a greater gravity than can be ascertained upon any scale, may readily be weighed upon the balance.

\* [The prefixed Scales are only intended to enable the reader to comprehend the description more easily.]



The divergence of the two lines  $a b$  and  $a c$  requires a remark. The smaller the divergence is, in general, the less will be the variation in weight for each descending line, and the more accurately the diameters of globules can be compared; but this is again limited in practice, for if the divergence be so minute that the difference between the diameters of two globules cannot be ascertained, the scale becomes useless. I have found the most convenient divergence to be 2-7ths of a line, in a length of 50 English lines.\*

The value of the scale is considerably increased by marking at the side of each division the per-centage indicated in a metallic globule corresponding to it. For the smallest of these three decimals are sufficient; if, however, the next decimal number exceed 5, the preceding may be increased by 1. A silver globule, when placed upon the scale, is often found not to coincide with any of the perpendicular lines: in this case the distance of the touching point from the upper line is ascertained; then the difference of weight corresponding to the divisions enclosing the globule, is multiplied into the known fraction, and the product added to the weight indicated by the under line. *E. g.*, if a silver globule, placed at 2-3rds of the distance between 43 and 44, corresponds to a richness of silver comprised between 83.48 and 77.916 loths, or, in decimals, 2.37160 and 2.21545 per cent., the difference of the centesimal value being 0.15615,—this, multiplied by  $\frac{2}{3}$ , gives 0.10410, which, added to 2.21545, shews the product to be 2.31955 per cent. The exactness of these calculations depends chiefly on the skill exercised in placing the smallest globules upon the scale, to ascertain their diameters. The operator may however control his results by weighing a number of globules obtained from an ore of a mean value in one lot upon the balance (after measuring them singly upon the scale), and dividing the whole weight by the number of globules taken.

2. *Application of the Scale for determining the weights of Gold Globules, obtained in quantitative assayings.*

It is very easy to comprehend, that upon such a scale as

\* [The English line is equal to 3.17494 millimetres.]—*Trans.*



the one just described, small gold globules obtained in cupellation, may also have their diameters and weights determined. If gold globules were as much compressed in refining as silver, their contents might be ascertained from the relative specific gravity of the two metals, but the cohesion of molten gold being much greater than that of silver, prevents it from assuming the flat form that generally accompanies the latter metal. It was therefore necessary to calculate a new factor for (W). This, Plattner effected, by dissolving 946 milligrammes of fine gold in nitro-muriatic acid (aqua regia), and precipitating the metal with protosulphate of iron,\* then filtering,edulcorating, desiccating, and igniting the residue. He next mixed the metallic gold with 14.554 grammes of calcareous spar, and submitted portions of this to cupellation, and also to the blowpipe assaying. The mixture (15.5 grammes) contained 946 milligrammes of gold, or 6.103 per cent.

The metallic buttons obtained in the cupellation agreed very closely with the above, weighing, each, 6.09,—and the globules resulting from the analysis with the blowpipe weighed altogether, 30.5, indicating a mean gravity of 6.08 for each, corresponding to 214.5 loths in a cwt.

The experiments agreeing, Plattner placed one of the globules between the lines *a b* and *a c* upon the silver scale, and found its tangent points to be in the middle, between the numbers 46 and 47. Consequently, the fact that a gold globule placed upon this part weighed 6.07 milligrammes, = 214.5 loths per cent., establishes the subjoined general equation:—

$$w = \frac{W}{L^3} l^3$$

$$W = 6.07, \text{ or } 214.5 \text{ loths.}$$

$$L = 46.5 \quad \text{,,} \quad \text{,,}$$

\* [The proto-sulphate is converted into per-sulphate, and per-chloride of iron.]—*Trans.*



<i>Divisions of the Scale.</i>	SILVER.		GOLD.	
	<i>Loths per cwt.</i>	<i>Per Cents.</i>	<i>Loths per cwt.</i>	<i>Per Cents.</i>
1	0.00098	0.000028	0.00213	0.00006
2	0.00784	0.000223	0.01706	0.00048
3	0.02646	0.000752	0.05760	0.00164
4	0.06272	0.00178	0.13653	0.00388
5	0.12250	0.00348	0.26667	0.00757
6	0.21168	0.00601	0.46081	0.01309
7	0.33614	0.00955	0.73174	0.02079
8	0.50176	0.01425	1.09229	0.03103
9	0.71442	0.02029	1.55523	0.04418
10	0.98000	0.02784	2.13338	0.06061
11	1.30438	0.03705	2.83952	0.08066
12	1.69344	0.04811	3.68648	0.10473
13	2.15306	0.06116	4.68703	0.13296
14	2.68912	0.07639	5.85399	0.16630
15	3.30750	0.09396	7.20015	0.20455
16	4.01408	0.11404	8.73832	0.24824
17	4.81474	0.13678	10.48129	0.29776
18	5.71536	0.16237	12.44187	0.35346
19	6.72182	0.19096	14.63285	0.41570
20	7.84000	0.22273	17.06704	0.48485
21	9.07578	0.25783	19.75936	0.56134
22	10.43504	0.29644	22.71623	0.64534
23	11.92366	0.33874	25.95683	0.73741
24	13.54752	0.38487	29.49184	0.83784
25	15.31250	0.43501	33.33406	0.94699
26	17.22448	0.48933	37.49628	1.06523
27	19.28934	0.54799	41.99131	
28	21.51296	0.61116	46.83195	
29	23.90122	0.67903	52.03100	
30	26.46000	0.75170	57.60126	
31	29.19518	0.82941	63.55552	
32	32.11264	0.91229	69.90659	
33	35.21826	1.00052	76.66727	
34	38.51792	1.09426	83.85036	
35	42.01750	1.19368	91.46866	
36	45.72288	1.29894	99.53497	
37	49.63994	1.41022	108.06209	
38	53.77456	1.52769	117.06282	
39	58.13262	1.65149	126.54996	
40	62.72000	1.78182	136.53632	
41	67.54258	1.91882	147.03468	
42	72.60624	2.06268	158.05785	
43	77.91686	2.21595	169.61864	
44	83.48032	2.37160	181.72984	
45	89.30250	2.53700	194.40425	
46	95.38928	2.70992	207.65467	
47	101.74654	2.89053	221.49391	
48	108.38016	3.07898	235.93476	
49	115.29602	3.27545	250.99002	
50	122.50000	3.48011	266.67250	



## 3. THE COPPER ASSAY.

The composition of natural and artificial substances containing copper, is always to be considered before the commencement of the operation, as upon this the difficulty of the examination depends. For the same reason, cupriferous substances are to be divided into the classes of,—

¶ Ores, Minerals, and Furnace Products :—

- (a) Containing volatile ingredients ;
- (b) Containing the Copper in an oxidized state, with or without acids and water, vitrified or washed in another manner with earthy constituents.

¶ Metallic Compounds, the Copper of which is either a principal or accidental constituent :—

- (a) Plumbiferous Copper, and Cupriferous Lead ;
- (b) Alloys of Copper with iron, nickel, cobalt, zinc, and bismuth, (one or several of these metals,) often also with lead, antimony, and arsenic, as accidental constituents ;
- (c) Stibiferous Copper ;
- (d) Stanniferous Copper.

For the ores, minerals, and furnace-products belonging to the first class, roasting is necessary, previously to the fusion of the copper, in order to eliminate sulphur and arsenic ; but it is not so with those of the second class. The impossibility of producing pure copper immediately from ores, minerals, and furnace-products, containing several volatile metals, many of which cannot be expelled by roasting, is well known from the fusion of copper ores on a large scale, as well as from the common test in the dry way. As it may be necessary to give particulars concerning this point, I shall proceed at once to the manipulation for obtaining the copper, as correctly as possible, by the aid of the blowpipe.



¶A *Ores, Minerals, Products of Furnaces and Manufactures.*

(a) EXAMINATION OF SUCH AS CONTAIN VOLATILE INGREDIENTS (SULPHUR AND ARSENIC).

To this class belong all copper ores prepared on a large scale; of minerals,—Kupfer Glanz, Purple Copper, Tennantite, Grey Copper, common sulphurets of copper and tin, Bourmonite, Melanglanz, &c.; and of furnace products, *kupferstein*, *kupferleg*, *cupriferous rohstein*, *bleistein*, *tutty*, &c.

Of these substances, the necessary ground ore is prepared, and 1 decigramme weighed for examination. As they must be roasted previously to the fusion, the sample is mixed in the agate mortar, either with three times its volume of dry pulverized charcoal, or with 20 to 25 milligrammes of graphite, (which in most cases, particularly for substances containing much arsenic, is more advantageous than charcoal.) It is then placed upon a clay basin, and besmeared with red ochre (peroxide of iron).

A charcoal prism of sufficient size is introduced into the charcoal holder, and tightened; a cavity is then made in it, and from its contiguous side so much cut out with a knife as is prescribed by the orifice *b*, *Fig. 26*, *page 21*, in order to allow the flame access to the cavity. The platinum foil and wire is placed in it, the clay basin, taken with the pincers by the rim, is sunk in a horizontal position, until it reposes upon the platinum wire. For obtaining a horizontal position of the basin, it is necessary to hold the pincers so that their arms can pass through the orifice. After these preparations, the blowpipe is provided with a point of medium size, then an oxidating flame, not too strong, is directed through the orifice upon the open space below the basin.

In order to produce a red heat on the surrounding space, as well as on the basin, the charcoal holder should be at the distance of an inch and a half from the wick of the lamp, for if it is too near the flame, sufficient heat is not produced; whereas, at a greater distance, by the introduction of a large



supply of heated air into the cavity, the combustion of the charcoal is too much accelerated, impairing generally the operation. The basin is kept for some minutes in a state of redness, though care must be taken to blow moderately, in order to prevent a partial or perfect fusion of the sample. In this operation, the presence or absence of volatile ingredients may be detected from the smell of the eliminated gases. Further; the charcoal, added to the ore, is wholly destroyed, which can be ascertained by examination with the iron spatula, heated over the free flame of the lamp, in order to prevent the adhesion of small particles of the substance. The basin is removed from the holder, its contents placed in the agate mortar, and reduced to powder. Generally the ore changes its colour in the first roasting, forming a porous mass, which may be easily detached from the basin, and seldom requires the use of the spatula.

In this roasting, which is terminated in about ten minutes, the greater part of the volatile ingredients is removed, (sulphur, arsenic, and antimony,) the addition of charcoal preventing the formation of sulphates and arseniates. For the complete elimination of the volatile substances preventing the reduction of the copper, a second roasting is necessary. The residuum of the first roasting is again mixed with three times its volume of pulverized charcoal, the basin, prepared as before, placed on the holder; if the charcoal, mixed with the substance, is in full incandescence, a somewhat stronger heat is applied, and as soon as half the charcoal is consumed, the assay is examined for volatile ingredients. If no smell indicating sulphur or arsenic is perceived, the remainder of the charcoal is allowed to burn under continued insufflation, after which the roasting is considered to be completely finished. On the other hand, if the volatile constituents are not wholly eliminated, a third roasting is necessary. It very seldom occurs (only in substances containing much arsenical nickel) that a fourth roasting is required; two are generally sufficient. In order to learn when the roasting is finished, it is better to weigh the basin several times successively, as after the assaying its weight remains constant; this, however, is difficult, and



occupies too much time; therefore the smell exhaling from the roasted substance usually serves for ascertaining this period.

Regarding the richness of substances, roasted with pulverized charcoal; conclusions may be derived with sufficient certainty, from the colour of the sample. The blacker the colour, the richer it is in copper; if reddish and whiteish, it is poor. If, instead of charcoal, graphite is employed, the assay is kept at a red heat, until the gases disengaged, become inodorous. The graphite being destroyed slower than charcoal, and consequently remaining longer in immediate contact with the substance, reduces continuously the volatile ingredients; preventing, at the same time, the formation of sulphates or arseniates. As soon as the smell of the escaping gas ceases, the basin is taken from the charcoal, and the metallic substance, mixed with the remainder of the graphite, is triturated again in the agate mortar. This operation is indispensable, as, generally, during the roasting, the upper is sooner consumed than the lower portion of the graphite, in which unaltered metallic particles might remain, these are again brought into contact with new portions of graphite. The sample is re-spread on the clay basin, and submitted to a new roasting in a higher temperature. At the commencement of this second operation, the smell of volatile matter is often perceived for some moments, indicating the volatilization of the remaining traces of arsenic and sulphur. The blowing is continued until nearly all the graphite is destroyed; then the basin is removed from the charcoal.

The absence of smell from an assay roasted with graphite, is a sufficiently-certain sign of the termination of the process. The constancy of the weight cannot be taken as a characteristic, as the consumption of the whole graphite is not necessary. The possibility of adding a surplus of graphite to an assay, is the chief advantage of its employment in roastings. It is necessary to employ it in a pure state. If a cupriferous mineral prepared on the large scale, contains heavy-spar or gypsum, the sulphuric acid cannot be driven off by roasting;



though the heavy-spar is reduced, in a red heat, to sulphuret of barium, and the gypsum to sulphuret of calcium, both substances re-oxidize after the combustion of the charcoal. For the reduction of the copper, contained in 1 decigramme of the substance, roasted in the manner here described, I adopted the following dressing, viz.:—

Soda, 100 milligrammes;

Borax, 50 milligrammes.

The soda reduces the oxide of copper, and other easily-reducible metallic oxides; and the borax dissolves the oxides of iron, manganese, cobalt, and the earthy constituents.

The roasted substance, mixed with the soda and borax in the agate mortar, are enveloped in a soda-paper cornet, and pressed into a cavity, prepared on the diametrical section of a good piece of charcoal, near one of its corners.

These preparations being effected, a pure and strong reducing flame is deflected directly upon the sample, until the paper, the substance roasted, and the graphite, are destroyed, and the metallic copper is seen floating, either on the scorïæ, (with its peculiar greenish-blue colour,) or separately near them, in the form of a metallic globule.

In rich substances, the reduced copper speedily appears, but slower, and sometimes not at all, if the ore is poor. Sometimes, after a long-continued strong insufflation, the copper unites to one globule. It however occurs oftener, that minute metallic particles remain in the scorïæ. I therefore think it preferable to interrupt the process of reduction, so soon as the melted copper appears in the perfectly glassy scorïæ; to withdraw the scorïæ cautiously from the charcoal (if not coloured red, which indicates vitrified sub-oxide of copper); reduce it to fragments between folded paper, upon the anvil; detach the visible globules; levigate and lixivate the mass in a porcelain basin, with a view to remove dross and charcoal; and desiccate the basin with the copper, over the lamp flame. If the scorïæ are not completely separated, they remain united with the copper, and adhere to the basin, from which they must be detached by the spatula. In this case, it is necessary to re-levigate with water, and to desiccate a second



time, after its affusion, when the copper may be easily detached. If the copper reduced in this way, and examined by a microscope, displays its natural colour, and is easily detached upon the anvil, its quantity may immediately be determined; should it, however, appear of a grey colour, fragile, or too ductile, these are signs of its contamination with other reduced metals. In this case, it ought to be fused on charcoal in the reducing flame, with 50 milligrammes of lead, and a small addition of soda and borax, until the copper unites with the lead, in the form of a bright globule, and the borax combines with the soda, as a limpid bead.

After refrigeration, the cupriferous lead is detached from the recrements, and affined according to the subsequent prescription. If, in the reduction of copper from a roasted substance, easily-fusible metallic globules appear, which can be readily united to a greater one, it may be concluded that:—

1. The roasting has not been entirely performed; or that heavy spar or gypsum is present, the sulphur combining with the copper as sulphuret, surrounding the pure copper, and forming with it a single button:

2. The substance contains other metals, making an easily-fusible alloy with copper.

As, by levigation and lixiviation, in the first case, a considerable loss will be sustained, owing to the inferior specific gravity of the sulphuret of copper and its divisibility, another process for separating the sulphuret, together with the pure copper, is to be pursued. Therefore, when these globules appear, the insufflation is interrupted, to ascertain their colour. A black tinge indicates the presence of sulphur, whereas if greyish or whiteish, a mixture with other metals may be inferred. Then the reduction is continued until all the copper is reduced into a ball, which may be effected by bringing the main globule successively in contact with all parts of the scorix. The reduction being completed, the ball is watched until it congeals, then quickly removed from the fluid scorix by the forceps, and allowed to cool. Should the earthy sulphate in the treated substance possess but half the weight of the whole, the metallic copper forms only a button,



enveloped in a thick crust of sulphuret. But if a small quantity only of earthy sulphates be present, the *kupferstein* forms a thin coating. The result is the same if the roasting be imperfect, or if we find sulphur in an acid state. The globule, removed from the scoriæ, is fused on charcoal in the reducing flame, with twice its weight of lead, and a little borax, until all the sulphur of the cupriferous lead is volatilized, and a bright metallic surface appears. After refrigeration, the metallic bead is detached from the colourless glass, and affined in the manner described hereafter. In this operation, if other metals are extant, these are separated at the same time.

In the second case, if the substance contains, besides copper, other metals oxidized in the roasting, it is also necessary to continue the fusion until the reducible metals form a single globule, and the recrementitious matters are fused. After the termination of the process, the scoriæ, with the metallic globule, are allowed to cool, and cautiously broken upon the anvil to detach all extraneous matters: as the alloy, in the reduction, rises above these scoriæ, and can easily be united to one ball, it is evident that no pulverization and lixiviation are necessary. Should antimony, tin, or bismuth be present, the globule is brittle; ductile, however, with lead. With a view to avoid pulverization and lixiviation for compounds difficultly combining in one globule, it is only necessary to add 30 to 50 milligrammes, (according to the per-centage of copper,) of granulated proof lead, by which means an easily-fusible metallic globule is produced, which is to be affined in the manner subsequently described; the affining, however, occupies less time than the lixiviation.

(b) EXAMINATION OF SUBSTANCES CONTAINING THE COPPER  
IN AN OXIDIZED STATE,

*Either Pure or combined with Acids and Water, or Vitrified with Earthy  
Constituents, &c.*

*Minerals.*—Red Copper Ore, Cupriferous Manganese, Black Copper, Muriate of Copper, Azure Copper Ore, Mala-



chite, Arseniate of Copper, Phosphate of Copper, Sulphate of Copper, &c. *Furnace products*.—All varieties of copper scorïæ; products of manufactures, particularly the pigments prepared from copper and its oxides, and the cupriferos vitriols.

The substances are not roasted, and, with the exception of the vitriols, are treated as follows, viz.—

Substance,	1 decigramme,
Soda,	1 decigramme,
Borax,	50 milligrammes;

if the copper exceeds 30 per cent., from 10 to 20 milligrammes of oxide of antimony, or 50 milligrammes of proof lead, are added.

The sample is mixed in the agate mortar, placed in the soda-paper cornet, and introduced into a cylindrical cavity prepared on charcoal. The reduction of the copper is effected in the same manner as the assays resulting from previously-roasted substances; the soda acting as the flux, combining with chlorine as well as with phosphoric acid; whereas, the borax dissolves the earthy particles, and the more difficultly-reducible metallic oxides, and prevents the absorption of the soda by the charcoal, when chlorine and phosphoric acid are absent.

By the addition of antimony and pure lead, considerable losses, resulting from the spattering of the fine copper, (often exceeding 3 per cent.,) will be avoided: though it is certain, that the evitiation of this inconvenience produces another one, namely, the remaining of a small proportion of foreign metals; this, however, is unimportant, as the final purification is one of the most simple and easy operations.

For the examination of substances containing upwards of 30 per cent. of copper, or where the presence of lead is surmised, the oxide of antimony is not to be employed. In the first place, it is to be feared that the copper, forming a very small globule, might be driven off mechanically, should the antimony be volatilized. In the second place, an affinage with boracic acid would be unavoidable. The sulphates of copper cannot be treated in the manner already described,



for, though the sulphuric acid is decomposed, and its sulphur partially combines with the sodium in the commencement of the operation, yet the former, owing to its great affinity for copper, separates again from the radical of the soda in a continued reduction, forming sulphuret of copper, which can with difficulty be rendered pure, and seldom without loss. It is therefore necessary to dissolve 1 decigramme of vitriol in water over the flame of a lamp; to precipitate the oxide from the boiled solution by caustic potash; to filter, edulcorate, desiccate the filter in a porcelain basin; to burn the dry filter over a mortar; to mix the ashes and metallic oxides with 50 milligrammes of soda and 30 milligrammes of borax; and to fuse the sample, enveloped in a soda-paper cornet, on charcoal in the reducing flame. By this operation, the copper is immediately obtained pure, if the original substance contained no foreign metals. If, in this case, the levigation of the scorïæ should be avoided, 20 to 30 milligrammes of proof lead are to be added before the reduction, to form an easily-fusible plumbiferous alloy, which can readily be affined by means of boracic acid.

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#### ¶ B *Metallic Compounds.*

The copper can be separated with the blowpipe from its compounds, with other metals, by oxidation only, which, acting upon those metals, partly volatilizes, and partly converts them into scorïæ; whereas, the copper remains in a metallic state. Though this operation could be effected by a process analogous to the affinage of copper in smelting-works,—it is, however, difficult, and always liable to a loss of the metal. The following process will answer best in docimastic designs. It is founded upon the property that boracic acid possesses of dissolving, in the reducing, as well as in the oxidating flame, several metallic oxides, particularly that of lead,—and of maintaining this combination, if it is not too much saturated, even in the strongest reducing flame; so that, whatever the proportions of the copper



to the lead may be, the separation can always be effected without the slightest loss to the former, and if a small portion oxidizes, it is reduced immediately. If the copper contains other easily-oxidizable metals, besides the lead, these are all converted to oxides. It is, therefore, evident that alloys containing copper, lead, and such metals, may be affined with the aid of boracic acid. Alloys of copper with antimony and tin, require another treatment.

(a) EXAMINATION OF ALLOYS OF COPPER AND LEAD.

The plumbiferous copper, obtained by reduction of plumbiferous copper ores.—The cupriferous lead, produced by reduction of cupriferous lead ores.—The cupriferous *workable lead*, produced on a large scale; *Frischstücke, Saigerdörnern, Darrlinge*, &c.

Alloys of copper and lead, obtained generally in one globule, from the reduction of 1 decigramme of a substance, are separated as follows, viz.:—

In a cavity, prepared on the diagonal section of a piece of charcoal, near one of its corners, a quantity of vitrified boracic acid, equal in weight to the plumbiferous copper, is fused to a bead, the metallic globule placed near it, and both covered with a good reducing flame. As soon as the boracic acid is fused, and the metallic globule presents a shining surface, the point of the blowpipe, whose opening ought not to be too large, is advanced in the flame, in order to change, gradually, the reducing flame to a blue one, which is so directed upon the fused boracic acid, that the alloy remains untouched. It must also be well observed, that the metal always remains in contact on one side with the glass, and on the other with the charcoal, as, even on the slightest inclination of the latter to the wrong side, the globule escapes under the glass, thus causing an interruption of the process. In this case, it is necessary to incline the charcoal to the other side, under a feeble insufflation, to allow access of air to the globule.

While the glass is treated with the blue flame, the lead bound in the alloy absorbs oxygen from the atmosphere; the



resulting oxide is carried to the boracic acid by the motion of its surface, and instantly dissolved.

This process is continued until nearly all the lead is oxidized, and the metallic globule seems to assume a greenish-blue colour; then a broader flame is directed upon the glass, to render the oxidation of the remaining lead slower, and to prevent a spattering of the copper. This inconvenience it is almost impossible to avoid, if the operation is performed in a groove of insufficient depth; therefore, the cavity ought always to have at least 3-16ths of an inch in depth, and 3-8ths of an inch in width. As soon as the greenish-blue colour, peculiar to fused copper, and indicating the desired degree of purity, is obtained, the process is interrupted, the solid globule removed from the still-fluid scoriæ, and its properties examined after cooling. The best indications of its perfect purity, are, the common red colour of copper, perfect malleability, and arborescent appearance upon the surface of the broken globule, when viewed through the microscope. This last sign is only perceptible in globules resulting from an alloy of high per-centage. If the scoriæ remaining upon the charcoal, are of a yellow colour, and limpid, the examination has been performed without a chemical loss of copper, and the bulb may be weighed. If the scoriæ appear reddish, or perfectly red, a loss of copper is indicated, which can be recovered in the following manner:—

If, as already mentioned, the boracic acid is not oversaturated with oxide of lead, the oxide, and sub-oxide of copper may be easily reduced, and separated in a metallic state by a good reducing flame. The glass treated after the detachment of the affined copper globule, with the reducing flame, becomes limpid, and of a yellowish colour, after refrigeration; the reduced copper being scattered through it in small globules. These globules are obtained either by fusing again the great copper globule, in a good reducing flame, allowing it to float in the scoriæ, covered by the flame, until all the small globules are united with it, and detaching it from the scoriæ as before; or by breaking the scoriæ enveloped in paper upon the anvil, and removing them by levi-



gation and lixiviation, in a porcelain basin. In the first instance, the copper can be weighed immediately; whereas, the globules remaining after removal of the scoriæ, ought to be dried in a basin over the lamp flame, and their weight ascertained, together with the large one.

From a glass containing a quantity of oxide of lead, besides the oxides and sub-oxides of copper, a single globule of lead and copper is obtained by reduction. For removing the lead from this alloy, it is only necessary to fuse the ball for some moments with a small addition of boracic acid in another cavity on charcoal. With the exercise of due caution, however, these labours are very seldom required, as, generally, the copper is obtained pure and without loss.

If the substance examined for copper in this way contains also a quantity of silver, (determined by an examination for this metal,) it must be deducted from that of the obtained copper; if, however, the per-centage of silver has not been determined previously, it ought to be treated as an assay of silver, with 15 times its weight of proof lead, and submitted to oxidation and cupellation, by which means the quantity of the former metal is determined.

If, instead of plumbiferous copper, as in the preceding case, cupriferous lead (as it is sometimes obtained by the treatment of cupriferous lead ores) is examined, the alloy cannot immediately be affined, owing to the time necessary for oxidation; in this case, the operation ought to be effected in two periods,—

- (a) The concentration, and
- (β) The affinage of the copper.

Although the concentration and affinage of a cupriferous alloy are performed with boracic acid in the manner already described, a quantity of the latter sufficient for the solution of all the oxide of lead cannot be added, because the remaining copper globule will seldom be obtained pure, owing to a tendency to imbed itself among the scoriæ. Therefore, such assays must always be concentrated previously to the affinage. The cupriferous lead is weighed exactly, fused to a globule with a small addition of soda and borax on charcoal



in the reducing flame; it is then treated with its weight of vitrified boracic acid, as though it were to be affined, until reduced globules of lead appear on the surface of the glass. The greater portion of the lead being oxidized in this way, and separated from the copper, the insufflation is suspended, and after cooling the metal in which the copper is now concentrated, it is detached from the scoriæ. If the glass appears like enamel, which generally is the case, the operation is performed without loss of copper. By the concentration, a plumbiferous globule is obtained, which may be treated as already described.

(b) EXAMINATION OF ALLOYS CONSISTING OF COPPER,  
IRON, NICKEL, COBALT, ZINC, AND BISMUTH,

*And in which the Copper is combined with one or several of these metals, and frequently with lead, antimony, and arsenic.*

To this class belong:—

(a) The impure black copper obtained from the treatment of cupriferous lead ores on a great scale; and the *Saigerdörnern*.

( $\beta$ ) The black copper obtained on a large scale from sulphuret of copper, copper slate, and other copper ores containing no lead.

( $\gamma$ ) The german silver, or argentan, and such compounds of copper with nickel, as contain little or no lead.

(a) *Affinage of the impure Black Copper prepared on a large scale from cupriferous lead ores.*

This species of black copper contains, besides lead and copper,—iron, nickel, antimony, zinc, arsenic, and various other metallic constituents. It is very brittle, and can be reduced only by percussion, to easily-segregating laminae.

One decigramme of the substance is weighed, fused on charcoal to a single ball, if not already consisting of one piece, and treated with one decigramme of boracic acid, as already described, until the globule is covered with a coating of oxides, and has lost its fusibility. In this process, lead, iron, antimony, zinc, arsenic, and other easily-oxidizable metals, and



a portion of the nickel, become oxidized. The formed oxides partly combine with the boracic acid, and partly volatilize. A portion of the nickel remains with the copper, in the form of a thin layer of oxide, greatly obviating the affinity. Though, by a prolonged oxidation, this coating, as well as the rest of the nickel, are dissolved by the scoriæ, this cannot be effected without a considerable loss of copper. It is, therefore, better to add a quantity of pure lead, equal to the weight of the alloy, and to submit it to a new oxidation, perfectly equal to the first one. By this the nickel is oxidized, together with the lead, without loss of copper. Should the latter, notwithstanding these precautions, be dissolved in the scoriæ, these ought to be submitted to a reduction, and after levigation and lixiviation, weighed, together with the main globule. The oxidized nickel is only reduced in a continued strong inner flame. As this sort of copper often contains half per cent. of silver, the per-centage of this metal ought to be determined, and deducted from the weight of the copper.

( $\beta$ ) *Affinage of the Black Copper obtained by the treatment on a large scale of sulphuret of copper, copper-slate, and other non-plumbiferous ores in general.*

The copper in these alloys is generally in combination with either iron or zinc, or both. Brass also belongs to this class, although its per-centage of zinc is large.

The metallic compounds of this order can be affined in the same manner as the plumbiferous copper:—1 decigramme of the substance is reduced to fragments, fused on charcoal with 1 decigramme of proof lead, according to the per-centage of zinc and iron, and a small quantity of soda and borax; after refrigeration and detaching of the scoriæ, the globule is treated with boracic acid, like plumbiferous copper; the iron and a portion of the zinc oxidize, another portion of the latter volatilizes, the copper remaining pure. If a small portion of the copper is also oxidized, it can be recovered by reduction and levigation. The per-centage of silver in these alloys is seldom so great as to be taken into consideration.



(γ) *Determination of the Per-centage of Copper in German Silver, and other Nickeliferous Compounds containing little or no Lead.*

Nickel, in combination with easily-fusible metals, oxidizes with difficulty, its oxide dissolving in boracic acid. If, however, the glass saturated by it is treated with the reducing flame, its greater portion will again be separated in the metallic state. Therefore, in the separation of nickel from copper by means of boracic acid and pure lead, the greatest care should be taken to prevent an oxidation of the latter metal, it being impossible to render it free of nickel in a reduction.

As a loss of copper will be sustained if an alloy contains upwards of 20 per cent. of nickel, it is necessary to examine such substances in samples of about 50 milligrammes; or 45 to 50 milligrammes of the substance are weighed, and fused with 2 decigrammes of proof lead, and some borax on charcoal in the reducing flame; if the alloy contains zinc, it is volatilized in this operation. After cooling, the borax glass (which, if iron or cobalt were present, would have the colouring from those metals) is detached from the metallic globule, and the latter treated with 1 decigramme of vitrified boracic acid, until the lead, with the greater portion of the nickel, is oxidized and imbibed by the scoriæ; the remaining globule requires a higher temperature for continuing in fusion. This operation must be performed by means of the blue flame directed upon the scoriæ, in order to allow the oxidation and scorification of the nickel. The metallic ball is to be kept in contact on one side with the scoriæ, on the other with the charcoal, for if the point of the blue flame only acts upon the sample, the copper will oxidize,—the flame ought to be so directed that it spreads over it.

So soon as the scoriæ are saturated with lead and nickel, (indicated by the reduction of a portion of the lead, and the difficulty with which the ball is kept in fusion,) the button is allowed to refrigerate, and is then removed from the scoriæ by the pincers. This ball, containing but a minute portion of nickel, is treated with its volume of proof lead and boracic acid on charcoal, as already described. From an alloy, poor in nickel, the copper is obtained pure by this operation; on



the other hand, if the portion of nickel be considerable, the globule obtained is still nickeliferous. In this case, it is fused again with its volume of proof lead, and 1 decigramme of boracic acid; and this operation ought to be repeated with twice the quantity of lead, and 1 decigramme of boracic acid, if, after the second oxidation, the copper has not lost its white colour.

The scorïæ formed in these operations must be of a red colour (from the oxide of nickel), and transparent, when compressed with the pincers. Green-coloured transparent scorïæ indicate dissolved oxide of copper; a brownish tinge, with opacity of the scorïæ, indicates the presence of dissolved sub-oxide of copper: these colourings, however, only appear when the affined copper is kept fusing too long. Scorïæ of a brownish-red colour ought to be covered with the reducing flame after removing the copper globule, to reduce the copper; and then obtain it by levigation and lixiviation. The reduction should be continued until the red colouring of the scorïæ has disappeared; on the contrary, if the scorïæ are coloured blue by the oxide of copper, it is difficult to deoxidize it, even in the best reducing flame. The copper obtained from this species of scorïæ is generally nickeliferous, and seldom exceeds 0.1 milligramme.

If, after these operations, the copper has attained its natural colour, its weight is ascertained, in order to calculate the value of the treated alloy. The following examples shew that, notwithstanding all precautions, a small loss of copper is sustained in the described treatment; this takes place in the affinage as well as in the first and second treatment with boracic acid, though it cannot be perceived, owing to the dark colour of the scorïæ:—

(1.) I composed an alloy of 50 copper, 25 nickel, and 25 zinc; by an examination effected upon 50 milligrammes of this compound, I obtained 24.5 milligrammes of pure copper. In this instance, therefore, I had a loss of 0.5 milligramme, = 1 per cent.

(2.) A mixture of 40 milligrammes of granulated nickel, with 10 milligrammes of rasped copper, yielded, in the



examination, only 8.6 milligrammes of copper,—loss, 1.4 milligrammes = 2.8 per cent.

Although I performed many experiments to discover a shorter and better way for examination, all my endeavours have afforded as yet no more satisfactory results than the treatment with lead and boracic acid.

(c) EXAMINATION OF ALLOYS OF COPPER AND ANTIMONY.

Among this order are found, particularly, the stibiferous alloys resulting from the examination of Grey Copper and copper ores containing no lead, and those obtained from rich copper ores, by the addition of oxide of antimony.

The separation of these metals is easily, and without loss, effected upon charcoal in the oxidating flame. The sample is placed in a cavity prepared on charcoal, and fused by a good oxidating flame at the greatest possible distance, taking care to direct the flame aside from time to time, in order to allow freer access of atmospheric air. The antimony volatilizes, the copper remaining pure. If, owing to the high per-centage of antimony, the oxidation is continued for a long time, the depth of the cavity increases, so that the flame touches the alloy with difficulty; when this occurs, the process ought to be interrupted, and then continued on a new piece of charcoal.

The purity of the copper is recognized by its ductility, by its greenish-blue colour in the fused, and its red copper colour in the solid state. If the metallic globule does not possess these properties, it must be submitted to a new oxidation.

This sort of copper is generally argentiferous; it ought therefore to be examined for silver. The per-centage of this metal is then deducted from the weight of the crude globule.

(d) EXAMINATION OF ALLOYS OF COPPER AND TIN.

The compound of copper and tin, obtained by examinations of 1 decigramme of pure sulphuret of tin, bell, and gun-metal, belongs to this class of alloys.



Boracic acid cannot be employed advantageously to separate tin from copper, not only as it is fused with difficulty, if not in combination with oxide of lead, but also, because it scarcely acts as a solvent upon the tin. Better results are obtained by the use of a flux which combines fusibility with the property of dissolving the oxide of tin. Its composition is as follows, viz.—

Soda,	100 parts,
Boracic acid,	50 parts,
Silica,	30 parts.

If stanniferous copper is to be examined, about 60 milligrammes of this composition are fused to a ball on charcoal, and placed by the side of the alloy. Of the compound obtained by reduction from 1 decigramme of sulphuret of tin, the whole quantity is taken, while, of the bell and gun metal, the quantity ought not to be larger than 45 to 50 milligrammes.

The glass bead, together with the alloy, is then fused in the reducing flame, until a rotatory motion ensues; from this the reducing is changed to the oxidating flame, which is so directed upon the glass, that it is protected from the access of air. The metallic globule then begins to oxidize on its surface, and if the oxide of tin, as well as of iron, is present, the latter is dissolved by the glass.

During the oxidation of tin, the sample ought to be kept in such a position, that the alloy may always be in contact with the charcoal on one, and with the fused glass on the other side, to prevent an oxidation of the copper. As this glass readily dissolves tin, the process ought to be continued until it is perfectly saturated with the metal. Saturation is recognized by the formation of cavities in the enamelled glass, near which small particles of reduced tin appear. The solid metal is then removed from the fused scoræ, and heated with 60 milligrammes of the above-mentioned glass, upon another piece of charcoal, without detaching the small adhering portions of scoræ, until the colour of fused copper appears; at this period, the glass is covered with a pretty strong reducing flame, until it affords the properties of pure copper. It is



then removed from the scorïæ, to observe its physical properties, colour, and ductility. If it presents the characteristic signs of pure copper, it may be weighed; if not, it ought to be treated again, with 20 to 30 milligrammes of the flux.

In an examination of this nature, care must be taken not to oxidize a portion of the copper, together with the tin; if this occurs, the sub-oxide of copper dissolved in the glass will communicate to it a brownish-red tinge. Such scorïæ must be treated for some minutes with the reducing flame; the copper is thus de-oxidized, combining with the main globule. A reduction of oxide of tin does not so soon take place, if the glass is not over-saturated. In the separation of the last portion of tin from the copper, a quantity of the latter always oxidizes; which, however, if proper care be taken, does not exceed the average of 0.3 milligrammes upon 25 milligrammes.

#### 4. THE LEAD ASSAY.

This metal is found in four different states of combination in minerals, ores, furnace, and artificial products,—

- (a) As a metallic alloy;
- (b) As a sulphuret;
- (c) Oxidized, with mineral acids;
- (d) As a pure oxide, or only in combination with organic acids.

These four states of combination must be well considered in quantitative examinations with the blowpipe, as it is sometimes necessary either to change certain substances in antecedent labours, or to separate wholly some constituents, before it is possible to render the lead pure. In substances containing the lead in the state of sulphuret, I ascertained its per-centage in two different ways, viz.—Substances containing other volatile ingredients besides sulphur, as arsenic, antimony, &c., are, as much as possible, purified from these bodies by roasting. The roasted sample is then mixed with soda and borax, placed between pulverized charcoal in two clay basins,



one of which, serving as the smelting vessel, is covered with a paste of charcoal, next submitted to the reducing flame, in order to deoxidize the lead, and to scorify the other substances present in the assay. Here the reduced lead is scattered through the scoriae in balls of different sizes. The second treatment, employed only lately, is more simple, shorter, and likewise leads to exact results. Substances containing sulphurets and arseniurets, without antimony, are fused, without previous roasting, in a crucible, together with soda, borax, tartaric acid, and metallic iron; here the lead unites to one ball, the earthy and non-reducible oxides undergoing scorification.

I would have omitted the first treatment, for its inconvenience,—but as it is useful in cases where the presence of copper has to be determined, I have described both.

With regard to the quantitative examination for lead, the minerals, ores, furnace and manufactured products, may be divided into,—

- (a) Such as contain the lead in the state of a sulphuret;
- (b) Such as contain it oxidized, and in combination with mineral acids.
- (c) Oxides of lead, either pure or combined with organic acids.
- (d) Metallic compounds.

(a) EXAMINATION FOR LEAD, IN MINERALS, ORES, AND  
PRODUCTS OF SMELTING WORKS, CONTAINING THIS  
METAL COMBINED WITH SULPHUR.

*First Method.*

The substances belonging to this class, which can be examined quantitatively for lead by this method, are, among minerals,—Lead-glance or Galena, Jamesonite, Bournonite, Zinkenite, &c.; among ores dressed on the great scale—Galena, and all lead ores containing sulphurets and arseniurets of other metals; and among products of smelting works particularly,—*bleistein*, *plombiferous kupferstein*, *plombiferous flue rakings*, &c.



A decigramme of these substances, reduced to a finely-divided powder, should be weighed; and then freed from volatile ingredients, by roasting on a pipe-clay capsule. As, however, the roasting of lead ores is similar to that of copper with charcoal powder, I will say nothing about it in this part of the work, except where a difference exists,—in order to avoid, as much as possible, unnecessary repetitions.

As soon as the roasting is ended, *i. e.*, when all the charcoal is burned off at a low red heat, and no more vapours of volatile ingredients are observed, the roasting capsule should be removed from the charcoal, the ore triturated in a mortar, and again mixed with twice its weight of charcoal powder. This mixture is to be spread on the pipe-clay capsule, and roasted a second time. As soon as the fire reaches the mixture, any escape of volatile ingredients will be readily ascertained by the smell. If this be the case, the charcoal should be allowed to burn off at a moderate red heat, the ore then triturated in a mortar, and roasted a third time with charcoal. It seldom happens that it is necessary to make three roastings; it cannot, however, be avoided, if the lead to be examined contains sulphurets and arseniurets, which are with difficulty decomposed. If no odour is observed during the ignition of the charcoal, in the second roasting, or only a slight odour of sulphurous acid, the assay may be considered to be fully roasted, as soon as the charcoal has been slowly consumed.

The roasting of pure lead glances, or rather such as contain neither sulphuret of arsenic or blende, proceeds much quicker than that of ores containing such ingredients. The cohesion of the particles of the ore is, however, less to be feared with the latter than with the former. Perfectly pure glances cannot be roasted *per se* with charcoal, without *sintering*, and therefore something which hinders this from taking place, must be added. I have found that this end is best effected, when 75 milligrammes of pure lead glance, containing about 80 per cent. of lead, are mixed with 25 milligrammes of pure pulverized pyrites, and this mixture then subjected to a second roasting



with charcoal. As 100 milligrammes of galena, mixed in the above proportion with iron pyrites, would be too much, not only for the operation of roasting, but also for the subsequent reduction of the lead, this proportion may be changed according to the state of purity of the galena, so that the quantity of both substances together does not exceed 100 milligrammes. It is always very convenient, in calculating the per-centage, that the lead resulting from the galena should be less than 100 milligrammes.

The characters of a perfectly-roasted lead assay are as follows:—

1. The ignited assay, after the termination of the roasting, should give off no odour of sulphurous acid or arsenic.
2. It must exhibit a dull earthy appearance, without any shining particles of undecomposed sulphuret of lead.
3. It would also be desirable that the assay be reduced to a loose friable state.

It is necessary to endeavour in some measure to recognise, from the appearance of the ores before roasting, as well as their colour after undergoing this operation, not only the predominating admixed substances of the lead ores, but also to arrive at a conclusion as to the per-centage of lead, as both have an important influence on the *dressing* required for the reduction of this metal. This is easily done, as pure galena, when roasted, has a greyish or yellowish white colour, the blendiferous galena, a greyish brown, and the pyritical, or ferruginous, a more or less red.

The dressing of the roasted lead ores for the reduction of their oxide of lead, is very simple, depending principally on the other metallic oxides to be vitrified, and on the earthy constituents. The flux should consist of—

Soda, 100 milligrammes, and  
Borax glass, 25 to 50 milligrammes.

The soda, and the charcoal which surrounds the dressing during the fusion, serve as reducing agents for the oxide of lead. If other metallic oxides are contained in the assay, such as are reducible in this way give reguli, and those which are not, are converted into protoxides.



The quantity of borax to be employed, is regulated by the quality of the ore. If the galena to be operated upon be in a *pure* state, it may be treated, after the roasting, with soda alone; or, if the slag formed during the fusion of the assay, is one which is not absorbable by the charcoal, the mineral may then be treated with about 25 milligrammes of borax. The quantity of borax increases in proportion with the poverty of the ore, or with the richness of the extraneous ingredients, as this flux is best adapted for dissolving most of the earthy constituents and irreducible metallic oxides. Thus, for example, in a mixture of galena, pyrites, arsenical pyrites, and blende, containing probably only from 3 to 5 per cent. of lead, the latter cannot be perfectly separated during the fusion, when too small a proportion of borax has been employed, as, during the reduction of the lead, the slag formed is not sufficiently fluid to allow the finely-divided metallic lead to separate and collect in larger globules; a quantity of borax of at least 50 milligrammes should therefore be used.

The weighed soda and borax glass should be triturated with the roasted ores in an agate mortar, the powder shaken in the mixing capsule, and then introduced into a soda-paper cornet, taking care to remove from the former, with a camel-hair pencil, the adhering particles, in order that nothing may be lost. The cornet must be so closed, that the folded empty part (which, in a packed dressed assay of silver or copper, is pressed up somewhat from both sides,) is in this case folded down on the full part, in order that it may have the form, as near as possible, of a hemisphere.

The assay, thus prepared, is then laid on a pipe-clay capsule, lined with charcoal, (*see page 15,*) which should be already prepared, or formed immediately after the roasting, in order that it may dry during the weighing of the dressing. The assay should then be covered with just so much fine charcoal powder, that when a similar pipe-clay capsule is placed over it as a cover, the whole space between both capsules may be filled up.

If the charcoal has not been very much burned off by the roasting, it may still be employed for the fusion; but if, on



the other hand, it has been so much burned that the necessary degree of heat cannot be attained, the part used should be removed, the capsule again placed in the charcoal-holder, being arranged in the manner already given for the roasting of Copper Assays, and the platinum wire and foil be attached to it.

The two capsules are then to be held in the forceps, and so placed on the charcoal, while the upper capsule is lightly pressed with the two forefingers of one hand, that not only the rim of the under capsule rests on the edges of the cavity, but, at the same time, comes horizontally on the platinum wire, as is shewn in *c*, *Fig. 27*, *page 22*. If the capsule does not lie horizontally, the side which is lowest must be lifted up into the required position with the forceps, the upper capsule at the same time being kept in its position by the pressure of the finger. When the capsules are arranged in their proper position, the orifice in the charcoal-holder must be closed, and the whole covered with a primitive piece of charcoal, having on its inner side a cavity (*o*) 3-8ths of an inch deep, formed with the largest of the charcoal borers, and a cylindrical opening (*p*) 3-10ths of an inch wide, which fits on to the charcoal-holder, and which is kept in its place by the projecting sides of the latter.

When every thing is thus arranged, a strong oxidating flame should be directed through the circular opening (*a*) of the charcoal-holder, which is to be held at a distance of from  $1\frac{1}{2}$  to 2 inches from the blowpipe orifice. A stream of strongly heated air will by this means be forced into the empty part of the charcoal, which soon raises the inner sides, and also both capsules, to a red heat. The temperature increases so rapidly, that a flame soon issues from the orifice (*q*) in the primitive charcoal cover. When a blowpipe-nozzle with too small an orifice is not employed, it may be considered certain that the most difficultly-fusible lead assay will be melted by an uninterrupted blast of five minutes,—unless the charcoal be too hard, and thus prevent the necessary temperature from being readily obtained.

At the end of the proper time, the blast should be inter-



rupted, the piece of charcoal used as a cover lifted off, and the platinum wire, with both capsules, removed from the charcoal with the *workable-lead* tongs, (the wire should be caught firmly at *n*,) and placed on the anvil to cool. As soon as the assay has so far cooled that the capsules can be touched with the finger, the upper capsule is to be taken off, and the charcoal powder, used as a protection and also as a reducing agent, removed, and the fused assay examined. The full success of the operation may be judged of from the presence of the following characteristics:—

1. No sublimate of oxide of lead should appear near the orifice on the upper side of the charcoal, covering the assay. If this be the case, the heat has been too strong, and a portion of the lead has been volatilized.

2. The fused assay should be a perfect globular button, with a smooth surface.

3. The button must separate, on the slightest touch, from the almost uninjured charcoal powder with which the capsule is lined.

If the assay agrees with these characters, the button should be rolled in paper, placed on the anvil, struck lightly with the hammer, and again examined.

4. Although the slags of poor lead ores have a glassy fracture, such a fracture cannot be expected in the case of rich ores, because soda, with a slight addition of borax, does not yield vitreous slags with the small earthy or metallic constituents, particularly when silica is not present.

If the reduction has properly succeeded, the lead (which very seldom occurs as a single globule, but generally as a number of large and small grains, disseminated through the slag,) should be separated from the latter in the following way:—The assay is to be again rolled in paper, and broken to a powder on the anvil. The paper is then to be carefully unfolded, the powdered assay shaken into a porcelain capsule, the larger grains of lead, free from slag, picked out, and the remainder of the lead separated from the slag by levigation, in the manner described for Copper Assays. If the large lead grains previously selected, are not perfectly



free from slag, they must be broken still smaller on the anvil, purified with water, and dried with the other lead grains, in a capsule.

In this way the whole of the lead is perfectly separated; but it still remains to be ascertained whether the lead, thus obtained, is pure from extraneous metals and other substances. This may be determined in the following manner:—

1. If the roasting has been carefully conducted, the lead must be free from sulphur and arsenic; if not, a *bleistein* or *speiss* would be formed, which compounds would be reduced to powder during the trituration of the slag, and may be distinguished from the lead after the levigation, with a magnifying glass, but cannot be mechanically separated from the latter without loss. When this occurs, the assay cannot be looked upon as successful, and the operation should therefore be repeated.

2. If the ore under examination was a pure galena, the resulting lead can contain no other metal than silver, as galenas in general (particularly those occurring on the sides of veins or lodes,) are argentiferous.

3. If, on the other hand, it was an impure galena, with which copper pyrites and sulphuret of antimony may occur in the assay powder, or a product of smelting operations composed of such ingredients, a portion of copper (owing to the easy reducibility of oxide of copper,) and antimony, which does not volatilize during the roasting, but remains behind as antimonious acid, may be also present, and must be examined for. The presence of the latter metal, is, however, readily discovered in the resulting lead, by its being not only harder, but more brittle, and less malleable than pure lead.

As, however, galena generally contains so little, (in case no rich silver ores are mixed with the dressed ores,) at most not more than from  $\frac{1}{2}$  to 1 per cent., and as copper and antimony rarely occur (with the exception of a few minerals) in dressed lead ores, a further separation of these metals from the resulting lead is only necessary, where it would be desirable to estimate, at the same time, the value of silver and copper.



When it is required to estimate accurately the value of the silver, copper, and antimony, contained in such plumbiferous ores, the resulting lead should be weighed on the balance, and examined for the previously-mentioned metals in the following manner:—

1. For silver, it is only necessary to cupellate on levigated bone-ashes, in the manner described at pages 274, 275; the lead having been previously fused on charcoal in the reducing flame, with borax glass.

2. Copper is found, when the lead is fused on charcoal, and treated with boracic acid, as given under that metal.

3. I know of no accurate method of separating antimony by the dry way. Whether such an alloy contains antimony, may, however, be readily discovered, when treated on charcoal in the reducing flame, by which the antimony is volatilized, and the charcoal coated with a white oxide.

The quantitative determination of the antimony present, which seldom amounts to more than 1 per cent., owing to the greater part being driven off during the roasting of the ore, can only be accomplished by the moist way; but, as this would be too tedious, I need not mention anything further about it.

After the examination of such alloys for silver or copper, the weight of the metals separated in the operation, must be subtracted from the original weight of the compound, and the remainder considered as the true value of the lead contained in the assays under investigation.

#### *Second Method.*

The substances which can be analyzed quantitatively for lead by this method, are, (among minerals and ores dressed on the great scale,) Galena, and all those lead ores which are mixed with sulphurets and arseniurets; and among products of smelting-works, *bleistein*, particularly, and plumbiferous *furnace-rakings*, and also impure *roh* and *blei* slags.

The necessary assay-powder should first be prepared from those substances, of which a quantity equal to 1 decigramme ought to be weighed. The quantity thus weighed, should be



shaken into a pipe-clay crucible, made as described at page 13, and well burned; a piece of iron wire, about 50 milligrammes in weight, and of the thickness of a moderately strong knitting-needle, must then be laid on it. In order to prevent the cupel from falling, it may be placed in the small cupel-mould. Then weigh, of—

Soda, 150 milligrammes,

Borax-glass, 30 milligrammes,

Tartaric acid, 20 milligrammes,

triturate these ingredients in an agate mortar, and put the mixture into the crucible containing the substance. The whole should then be covered with 50 milligrammes of dry chloride of sodium.

The iron serves to separate the sulphur and arsenic; the soda and borax-glass for the production of the necessary slags, and solution of the other sulphurets which may happen to be mixed with the sulphuret of lead, and also of the earthy constituents; the tartaric acid, for preventing the intumescence of the dressing in the crucible, as also by its carbon to keep the slags (which, after the decomposition of sulphuret of lead, generally contain sulphuret of sodium,) from acting as a solvent on the metallic lead; and, lastly, the chloride of sodium for a cover, in order that the lead globules, separately set free, may the more readily combine, for which purpose it is admirably suited, owing to its great fluidity in the state of fusion, and its property of not combining with slags.

The ingredients employed for "the dressing," can also be mixed with the substance to be examined; but I have, however, found that a larger number of globules of lead come to the surface of the slag in this case, and that it requires a longer time to combine them to one globule, than when the substance is placed with the iron *per se* in the bottom of the crucible.

The crucible, with its dressing, is then placed on a piece of charcoal, held on the charcoal holder, arranged in the same manner as for the roasting of a copper or lead ore, the platinum foil, however, not being used in this case, so that it rests quite free on the wire in the cavity; *i. e.*, does not touch



the charcoal, the rim of the crucible being on a level with the surface of the latter. The orifice of the charcoal-holder is then closed, and the crucible covered with a piece of charcoal arranged exactly in the same manner as in the fusion of a lead assay between two pipe-clay capsules.

When every thing has been thus arranged, a strong oxidating flame is deflected on the round orifice in the charcoal-holder, at a distance of about two inches from the lamp, so that an intensely-heated air passes into the hollow in the charcoal, by which the inner side and the crucible are raised to ignition, and the dressing fused. If the charcoal is not too hard, the heat diffuses itself rapidly, and the assay is perfectly reduced in a blast of four, or at most five minutes' duration. As soon as the blast is discontinued, the upper charcoal or cover should be lifted up, and the charcoal holder tapped with the broad side of the forceps, in order that the small globules of lead, that may happen to be here and there disseminated through the slag, (which however rarely occurs,) may sink to the bottom, and combine with the main globule. The crucible is then to be lifted from the charcoal, and placed for a few seconds in the small cupel mould to cool. As soon as it is cold, it must be broken with the hammer, on the anvil, and the lead and accompanying iron separated from the slag. The lead button is then caught with the forceps, placed on the anvil, so that the adhering iron may be upwards, and the latter,—which is sometimes (when the ore under examination is very pyritical) covered with sulphate of iron,—separated from the lead, by a few slight percussions. The lead button, freed from iron, may be purified from any adhering alkaline slag, by flattening on the anvil, between moistened filtering paper; and then weighed.

If it be suspected that the substance under examination contains silver or copper, the former may be separated by cupellation on bone ashes, and the latter by treatment with boracic acid; and the weight of the metals thus separated subtracted from the original weight of the assay.

The value of the silver thus obtained, when the substance



is a pure galena, is equal to the value deduced from a special assay for silver, but it is too small when the substance is an argentiferous pyrites, or contains any other argentiferous sulphuret. The value of copper is generally too small; the cause of this is easily understood, and therefore requires no further explanation.

If the substance under examination, (as, for example, iron pyrites, arsenical pyrites, blende, or *roh-* and *blei-* slags, with finely-disseminated particles of *rohstein*, &c.,) contain only from 1 to 10 per cent. of lead, it is always difficult to separate the lead button from the iron, so that its weight can be ascertained with accuracy. In such cases, a fine, accurately-weighed silver button, of about 50 to 80 milligrammes, should be added to the dressing; and instead of 20 milligrammes of tartaric acid, 50 milligrammes should be added to the slag, which may probably still contain oxide of lead. The reduced lead combines with the silver, and may be separated from the iron, and its value ascertained, by again weighing the silver button, and subtracting the weight of the original silver button,—the remainder being the value of the lead.

When such substances are rich both in silver and copper, the assay succeeds by the first method.

If an ore prepared on the great scale, consisting principally of galena, should contain a plumbiferous ore, in which the lead is contained in an oxidized state, 100 milligrammes of tartaric acid should be added instead of 20, in order that a sufficient quantity of carbon may be present to reduce the oxide of lead.

(b) ESTIMATION OF THE LEAD IN MINERALS, ORES, AND  
ARTIFICIAL PRODUCTS, CONTAINING THIS METAL  
IN THE STATE OF OXIDE, COMBINED WITH  
MINERAL ACIDS.

To this division belong the *green*, *brown*, *yellow*, and *red* varieties of earthy carbonate of lead, sulphate of lead, and artificial chromate of lead, or the commercial chrome-yellow.

All these compounds are characterized by being either not at all or only partly decomposed by roasting, as the stability



of many of the acids is too great, and their affinity for the oxide of lead too strong.

If lead salts, free from other salts, particularly metallic sulphates, and also earthy ingredients, are to be assayed for lead, they may be considered as nearly pure roasted galena. An assay powder being first prepared, a decigramme is to be dressed with—

Soda, 100 milligrammes ;

Borax Glass, 25 milligrammes ;

and subjected to treatment similar to what has been already given for Lead Assays which require to be roasted previous to reduction.

If, on the other hand, the acidiferous lead ores contain other metallic salts, particularly sulphates, or if sulphuretted and arsenical metallic combinations be mixed with them, a perfect roasting with charcoal is necessary.

A decigramme of the substance so roasted, should be dressed with—

Soda, 100 milligrammes ;

Borax Glass, 40 to 50 milligrammes.

The further treatment of such an assay, remains, however, the same.

If acidiferous lead ores are mixed with other metallic salts, whose bases are reduced with the lead, the latter must be examined for silver, copper, and antimony, in the manner above described.

In the reduction of oxide of lead from lead salts, the soda, with the surrounding charcoal, serves as a reducing agent for the oxide of lead and the acids. Thus, for example, arsenic acid is reduced to metallic arsenic, which volatilizes ; sulphuric acid, to sulphur, which combines with the sodium of a portion of the soda, forming sulphuret of sodium ; chromic acid, to oxide of chromium, which permeates the slag. Borax, however, serves only, as with pure galena, to prevent the absorption of the soda by the charcoal support ; as it fuses with soda, and probably with reduced portions of the ores, to a bead, while the metallic lead, in various sized globules, collects on its surface.



In the reduction of the oxide of lead, contained in a roasted assay composed of lead salts, contaminated with other substances, the soda plays the same part as in the preceding case, in addition to its solvent action, which it probably exerts on traces of silica; but an excess of borax, however, principally serves as a solvent for the non-reducible metallic oxides.

(c) METHOD OF ESTIMATING THE VALUE OF LEAD, IN  
PRODUCTS OF SMELTING WORKS,

*And other artificial substances containing Lead in a pure oxidized state,  
or only combined with Vegetable Acids.*

To this class belong *litharge*, *abstrich*, *cupel grounds*, pure *roh* and *blei* slags, all kinds of plumbiferous glass, white lead, sugar of lead, &c. &c.

The bodies of this class do not require to be roasted; but in dressing them for the reduction of their oxide of lead, it should be ascertained whether the body under examination is a simple oxide of lead, or a combination of oxide with other substances.

As the preceding substances may be at once subjected to the reduction process, they should be dressed,—the pure oxide of lead, or such oxides of lead as are merely combined with organic acids, with—

Soda, 100 milligrammes;

Borax Glass, 25 milligrammes;

and those in which difficultly-fusible ingredients predominate, as for example, *cupel grounds*, *roh* and *blei* slags, and with a quantity of borax amounting to about 50 per cent.

If such products contain, besides lead, other easily-reducible metals, the resulting lead button, after being weighed, must be examined for such metals, in the manner already given.

(d) METHOD OF ESTIMATING THE VALUE OF LEAD, IN  
MINERALS CONTAINING THIS METAL  
ALLOYED WITH OTHERS.

Black Tellurium, Yellow Tellurium, Seleniuret of Lead,



Seleniuret of Lead and Copper, and Seleniuret of Lead and Mercury, come under this class.

Of these minerals, I have only had an opportunity of examining seleniuret of lead quantitatively for its lead, before the blowpipe, and can therefore only give the method by which the value of lead in this mineral was found.

The assay succeeds most readily, and most accurately, when 100 milligrammes of the finely powdered mineral are introduced into a pipe-clay crucible, with a small piece of iron of 30 to 50 milligrammes in weight, covered with a mixture of 150 milligrammes of soda, 30 milligrammes of borax glass, and 20 milligrammes of tartaric acid; on which mixture, about 50 milligrammes of dried chloride of sodium should be shaken, and the fusion then proceeded with in the manner described at pages 334, 335. If the seleniuret of lead contains an admixture of sulphuret of lead, it will be decomposed; the selenium, however, combines with the radical of the soda, forming seleniuret of sodium, from which a small portion of the selenium volatilizes, and the lead, combined in one globule, deposits itself on the iron, at the bottom of the crucible. When the fusion is finished, and the fused assay has cooled, the crucible is broken, and the reduced lead freed from the iron and slag. The malleability of the lead button should be tested, and its weight ascertained.

When required, the value of the silver present may be ascertained by cupellating the button on bone-ashes, and weighing the resulting silver button, when sufficiently large; if too small for the balance, it should be measured on the scale.

## 5. DETERMINATION OF TIN.

The usual method of determining tin by the dry way, which has been for so long a time employed, and is so still in most assay-laboratories, is far from being accurate. There are a great many different methods of determining quantitatively the value of tin in an ore by the dry way; but if it be a ferruginous tin ore, by one method pure tin will be obtained, but generally in too small a quantity,—while by another, it



appears in excess, but never pure.\* The cause of this is, probably, that with a sufficient quantity of carbon, the reduced tin acts itself during the fusion as a reducing agent on the peroxide of iron, by which a portion of the latter is not only reduced to protoxide, but even to the metallic state, which latter combines with the reduced tin, affording a hard tin button, which, when weighed, gives too high a result.

After having convinced myself, by a number of tin assays made according to different methods, of the above-mentioned source of error, I concluded, from my experiments on the determination of the tin in ores by the blowpipe, that these discrepancies could not be avoided in all the control-assays made according to the usual methods, and for this reason I have introduced a new one.

For this purpose, I prepared pure peroxide of tin; mixed various quantities of it with a number of pulverized minerals, (as Iron Pyrites, Sulphuret of Arsenic, Blende, &c., which generally occur along with tin ores); calculated the value of tin which each should afford, and submitted them to the action of the blowpipe, with the view to determine this value by a number of experiments. The assays made with these mixtures fully convinced me that the same difficulties are to be encountered, with the blowpipe, as with the usual tin assays by the dry way made on a large scale. I often obtained from 1 to 2, and once even 4 per cent., too much,—that is, of ferriferous tin. A number of other experiments, in which I employed all possible means to slag perfectly as protoxide in the dry way by fusion, the peroxide which would be formed during the roasting, were also in vain; in no case was the resulting tin free from iron. I was at length compelled to separate the iron in an oxidized state from the oxide of tin by a simple yet accurate means in the moist way, namely, by hydrochloric acid, and then to submit the oxide of tin left behind with the earthy ingredients to reduction.

By the employment of this method, I always obtained a

\* I must here observe, that in practice, where it is certain that too much, and therefore impure tin, is obtained, the value is taken at a few per cents. less than that obtained by experiment.—*Author.*



pure malleable tin, whose weight agreed well with that found by calculation. Mixtures of the pure tinstones with other ores containing either copper or iron, yield exactly the same result.

It may be objected to the employment of hydrochloric acid for the separation of oxide of tin from oxide of iron, that it does not belong to blowpipe analyses; but if the difficulties which are to be encountered in the dry way with the blowpipe, in order to purify peroxide of tin from the oxides of iron and copper, be compared with the easy and accurate method by the moist way, there is no doubt the latter will be chosen as the best which can be employed in the quantitative examination of ferruginous and cupriforous minerals and dressed ores for tin.

In respect to the quantitative examination for tin;—minerals, ores, and artificial products, in which this metal forms an essential ingredient, may be classed as follows:—

- (a) Such as contain the tin combined with sulphur;
- (b) Those containing the tin in an oxidized state; and,
- (c) Those in which metallic tin is alloyed with other metals.

(a) DETERMINATION OF TIN IN MINERALS, ORES, AND ARTIFICIAL PRODUCTS, IN WHICH THE TIN IS COMBINED WITH SULPHUR.

Besides Tin Pyrites, tin schlichs,\* dressed on the great scale, may be placed in this division; although they contain tin in the state of oxide, they very often, notwithstanding the roasting, exhibit traces of metallic sulphurets and arsenical compounds.

Of the artificial products which belong to this class, I will only mention Mosaic Gold (sulphur, combined with excess of tin).

To determine the tin in any of the substances belonging to this class, an assay powder should be prepared according to the method given at page 263,—100 milligrammes of which is to be weighed and roasted, in order to free it from volatile ingredients. But as the roasting of tin assays is exactly

\* [Explained in note, page 161.]—*Trans.*



similar to those of copper with charcoal dust, any further notice of it here is unnecessary.

If the volatile bodies contained in a substance to be roasted, be confined to sulphur, or to sulphur with a trace of arsenic, or only a few per cents. of arsenic, which last is sometimes the case with tin schlichs prepared on the great scale, the roasting is very soon finished; but if a large quantity of arsenic is present, the roasting with charcoal is longer, and must be continued until not the slightest odour of arsenic can be observed, when a fresh portion of charcoal is added, and the mass kept in a state of ignition for some time. Tin schlichs, dressed on the great scale, if already roasted, require to be again roasted only once; the other substances, on the other hand, in which the tin is combined with sulphur, or mixed with metallic sulphurets or arsenical compounds, require to be roasted with charcoal two or three times.

By this process of roasting, when carefully performed, sulphur, arsenic, the greater part of the antimony, and a small portion of the zinc, will be volatilized from a stanniferous substance, mixed with, probably, Iron Pyrites, Arsenikkies, Copper Pyrites, Speissglanz, Blende, Wolfram, &c.,—while the other metals, tin (when it is not present in an oxidized state), copper, iron, manganese, and the remainder of the zinc, will be oxidized. Those metals (exclusive of arsenic,) which are capable of acidification, and which are difficultly or not at all volatilized during the roasting,—as, for example, a small portion of the antimony, molybdenum, tungsten, titanium, &c.,—remain behind as acids.

The characters which distinguish a well-roasted tin ore, are exactly similar to those of a well-roasted lead one. Thus, the roasted ore should yield no odour in contact with charcoal in a state of ignition; and no shining particles of metallic sulphurets or arsenical compounds should appear, when the substance, after the charcoal is perfectly burned away, is rubbed in a mortar; it should also be perfectly friable on the roasting capsule.

If a well-roasted tin ore, consisting, for example, of the oxides of tin, iron, manganese, and copper, be immediately



subjected to reduction, a brittle, grey, and too heavy tin button is obtained, even with the best fluxes and reducing agents, owing to the oxide of copper being at the same time reduced, affording a brittle metallic compound; and, secondly, as above mentioned, to a portion of the oxide of iron being reduced to the metallic state, and which likewise combines with the tin.

As the disadvantages just mentioned, cannot, however, be guarded against in the dry way, it is better to remove the oxides of iron, manganese, and copper, from the roasted tin ore, with hydrochloric acid. This end may be attained in the following manner:—

The well-roasted ore is to be introduced into a small porcelain capsule or digester (see *Fig. 40*, and *page 28*,) spread out thinly on its bottom, and hydrochloric acid poured on it, the quantity depending on the substances to be dissolved; the capsule is then placed on the triangle, at about  $2\frac{1}{2}$  inches over the flame of the spirit-lamp, the wicks of which should be pressed so far into the wick-holder, that only a very small flame is afforded, just sufficient to warm the capsule strongly. But in order to prevent the fumes from escaping into the room, the capsule should be covered with a watch-glass, placed with its convex side downwards.

The acid should then be observed, whether it is coloured from dissolved metallic oxides, and whether small bubbles rise here and there from the substance, which show that it has commenced to boil. The digestion should be conducted in this manner, uninterruptedly, for four or five minutes, taking care that the acid does not boil too strongly, as in that case particles of matter may be lost. The vapours which arise during the solution, are generally condensed on the convex side of the watch-glass, and fall back again into the solution.

After the expiration of at most five minutes, in which time all the oxides of iron, manganese, copper, and zinc, and also the antimonious acid, will be dissolved out, the triangle and capsule should be removed from the lamp, and the whole allowed to cool.

The watch-glass should then be taken off, and the adhering drops of acid removed from it with bibulous paper.



The clear yellow, or green supernatant solution, resting on the undissolved powder, which consists of only oxide of tin, or tinstone (sometimes, however, contaminated with earthy matter, or with wolframic and titanitic acids), is then to be drawn off with a pipette, (see *Fig. 38, page 26*), and replaced with from three to four times as much water. The water should always be poured down the side of the vessel, so that the substance on the bottom of the vessel be not disturbed, and the light particles suspended in the fluid, as otherwise the deposition of these particles again would require some time.

In order to remove the whole of the coloured solution, the vessel should be again heated over the lamp, and nearly the whole of the water drawn off with the pipette, by slightly inclining the capsule to one side.

If very minute particles of the powder, which are generally earthy ones, should float about on the surface of the water, which sink with difficulty to the bottom of the capsule, the fine orifice of the pipette, in removing the water, should be kept about a quarter of an inch under the surface of the water, in order that nothing may be lost.

The rest of the water is separated from the substance, by placing the capsule on the triangle over the spirit-lamp, until the powder is perfectly dry.

The whole operation, (by which, with necessary caution, no loss of tin need be apprehended,) does not, including the time employed in solution, require at most more than a quarter of an hour.

That the protoxides of iron and manganese, chemically combined with the tin-stone, which scarcely amount together to from two to three per cent., even in an exceedingly dark-coloured tin-stone, cannot be removed in this way, is easily seen; but as this protoxide of manganese, and the greater part of the protoxide of iron, are dissolved by the slag in the reduction, only an unappreciable trace of iron in the metallic state combines with the reduced tin.

The next operation is the reduction of the oxide of tin, resulting from the roasting, or in an unchanged state of tin-stone, freed from admixed metallic oxides by hydrochloric



acid. This is effected by the aid of the proper fluxes and reducing agents, in a space surrounded by charcoal, just in a similar manner to the reduction of oxide of lead, in roasted lead ores. The dressing for this purpose is very simple, consisting of—

Soda,           100 milligrammes;  
Borax Glass, 30 milligrammes.

This dressing is to be mixed with the ore in an agate mortar; the mixture is then to be introduced into a soda-paper cornet, like roasted lead ores, dressed with soda and borax, (see page 329), and fused between two pipe-clay capsules. Tin assays, however, require to be fused for eight minutes. When the assay has cooled, a bead is found in the bottom of the capsule, consisting of slag, in which the reduced tin is enclosed, sometimes, as one—but, oftener, disseminated through the mass in various-sized globules. These globules, like those of lead and copper, are freed from slag, by levigation with water, and then dried.

The purity of the resulting tin may be judged of from its action on the magnet, its colour, and its malleability.

If the roasting, and the separation of the oxides of iron and copper, and the antimonious acid, be carefully conducted, a pure tin of the proper weight may always be obtained by employing the necessary degree of heat in the reduction. If the ore has not been properly treated with hydrochloric acid, a brittle tin is obtained after the reduction, when copper and antimony are present; and when a little iron is present, the tin obtained, although malleable, acts on the magnet when in a finely-divided state, under water, the weight of which is naturally too high. If the ore under examination contains tungstic or titanic acids, they cannot be separated by means of hydrochloric acid, as already mentioned; but as they combine with soda in the reduction of the peroxide of tin, they produce no injurious effect on the result.

If the metal obtained has the properties of pure tin, it can be weighed; if not, a fresh assay must be more carefully performed.

As tin-stone occurs in *interlaced masses*,\* *flat or dilated*



*veins* and gangues, or vein-stones, with Granite, Gneiss, Micaceous Schist, *litharge*, Talk-Clay, Calcareous Spar, and many other minerals and rocks, as also with Iron and Arsenical Pyrites, Speiss-Glanz, Blende, Wolfram, Molybdic Ochre, Iron Ochre, Magnetic Iron Ore, and, in a very large number of these minerals, only in so finely-disseminated a state that the presence of tin in them can scarcely be suspected, and cannot be ascertained with accuracy, even by a trial with the blowpipe, owing to the minute quantity present; but by careful levigation and decantation of such an ore, reduced to a fine powder and weighed, the earthy particles may be removed and a schlich obtained in which the tin ore is concentrated, and from which, when perfectly dried and weighed, at least two quantitative tin assays should be made according to the manner described; the resulting tin is then weighed, tested as to its purity, and the value for the undressed ore calculated.

Thus, for example, if 5000 milligrammes of such a fine pulverized ore yield by levigation with water, and subsequent decantation, a quantity weighing 700 milligrammes when dried, from which, when thoroughly mixed and triturated in an agate mortar, two tin assays are prepared; now, if both these assays give 1.5 per cent. of tin, there would be contained in the 700 milligrammes of schlich,—

$$100 : 700 :: 1.5 : x;$$

or,  $1 : 7 :: 1.5 : x = 7 \times 1.5 = 10.5$  milligrammes of tin. If the levigation has been carefully performed, these 10.5 milligrammes represent very nearly the whole value of the tin contained in the above 5000 milligrammes of raw ore, which gives the quantity in 100 at—

$$5000 : 100 :: 10.5 : x;$$

$$\text{or, } 50 : 1 :: 10.5 : x = \frac{10.5}{50} = 0.21 \text{ per cent. of tin.}$$

(b) DETERMINATION OF TIN IN MINERALS, AND PRODUCTS CONTAINING THIS METAL IN AN OXIDIZED STATE.

To this class belong, of minerals, pure Tinstone; and, of artificial products, *tin-ashes*,\* enamel, &c.

\* [The dross left on the floor of the reverberatory furnace in the refining of tin.]—*Trans.*



Such substances do not require roasting, nor, if oxides of iron, copper, or antimony, be not accidentally present, the treatment with hydrochloric acid, previous to the reduction of the oxide of tin. It is merely necessary to weigh 100 milligrammes of the perfectly-dried and finely-pulverized tinstone, or of such artificial products as are not combined with silicic acid; to dress them with—

Soda, 100 milligrammes,

Borax-glass, 25 milligrammes;

and subject them to reduction in the same manner as the substances belonging to the preceding class.

To determine the value of tin in enamel, where the oxide is in combination with silicic acid, 100 milligrammes of the substance should be dressed with—

Soda, 150 milligrammes,

Borax-glass, 30 milligrammes,

by which the silicic acid combines with the soda, and the oxide of tin is reduced. But, as oxide of lead is often present in enamel, and is also very easily reduced, the resulting tin will not be pure, owing to its combining with a portion of lead. Such a compound cannot be separated into its elements in the dry way, but with nitric acid the lead can be dissolved out, leaving the tin as oxide undissolved. It is then only necessary to edulcorate this oxide, dry it, ignite it strongly in a platinum spoon, and calculate the value of metal from the weight of the ignited oxide,—100 parts of oxide of tin containing 78.62 parts of metallic tin.

#### (c) DETERMINATION OF TIN IN STANNIFEROUS ALLOYS.

To this class belong bell and gun metal, and all combinations of tin with lead, bismuth, zinc, and antimony. But as the quantitative determination of tin in the latter compounds in the dry way, with the blowpipe, is very uncertain, while in the moist way, on the other hand, it may be easily and accurately determined, the application of the blowpipe is therefore confined to bell and gun metal, which are compounds of tin with copper.

The method of separating tin from copper, has been



already fully described, in speaking of the Copper Assay at page 324. In this method, the copper is the principal object of investigation, no reference being made to the tin. But if it is required to find the value of tin at the same time, the glass which contains the whole of the tin as oxide, should not be lost, because the metal may be again reduced from it.

When desirable to determine the tin before the blowpipe, in a combination of tin and copper, the tin must be oxidized by the method previously described, and in this state separated, together with the flux, consisting of soda, borax, and silica. This glass should then be pulverized, mixed with about 50 milligrammes of soda, this mixture introduced into a soda-paper cornet, and fused, surrounded with charcoal, between two pipe-clay capsules, like an ordinary tin assay. But the tin is found, after the fusion, contaminated with a trace of copper, owing to an exceedingly small quantity of the latter combining with the glass in this operation.

The resulting tin is now to be weighed, and the centesimal value found by calculation, as only 50 milligrammes of such metallic compounds are generally employed in an assay.

THE END.



## APPENDIX.



## APPENDIX



## APPENDIX.

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### I.

#### A SIMPLE METHOD OF FINDING THE WEIGHT OF ANY VERY MINUTE METALLIC GLOBULES, WITHOUT A BALANCE.

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*Contributed to Dr. J. S. Muspratt's Translation of Plattner's Work on the  
Blowpipe, by PROFESSOR OLIVER BYRNE, Mathematician, author  
of "The Doctrine of Proportion," &c. &c.*

LET ABCD, and ABFG, (*Fig. 49, page 354,*) be two pieces of very finely polished plate-glass, joined together at AB, forming with each other a very acute angle.

The necessary mounting is omitted, for the purpose of exhibiting the parts more distinctly. AC and BD are scales of equal parts, of a greater or a less degree of fineness, according to the exactness required; they may be drawn on paper, and pasted to the glass. XY is a cylindrical piece of wire of any convenient length, or of any sort of metal, placed between the plates of glass, near the top, parallel to AB: the planes are tangent planes to the cylindrical wire. This wire must have a fixed position while experimenting.

The diameter of this cylinder can be calculated with very great accuracy, by weighing a pound, or half-a-pound, of the wire from which it is cut; which weight, divided by the length of XY, will give its weight, and, from knowing the specific gravity of the metal, the diameter of the wire can be thus accurately determined; this diameter will be the diameter of the globe  $n$ , the greatest possible that can be made out of the cylinder XY. The solidity of this globe can be readily ascertained, and would stand in the same position, subtending the same acute angle, if the cylinder were removed.

Supposing the inch and the ounce to be the units of measure and weight respectively, L the length of a piece of wire weighing  $q$  ounces, from which  $XY = l$  is supposed to be taken, then  $\frac{ql}{L}$  = the weight of XY.

Then say as,  $S : \frac{ql}{L} :: c : \frac{cql}{SL}$  = the solidity of XY in cubic inches,



S being = the specific gravity of the metal, or the number of ounces in 1728  
 (= c) cubic inches;  $\therefore \frac{c q}{S L} = \text{area of the circular cross section of XY.}$

$$\pi = 3.14159, \text{ \&c.}$$

$\sqrt{\frac{4 q c}{\pi L S}} = \text{diameter of this section, or of the globe } n, \text{ the solidity of}$

which =  $\frac{\pi}{6} \sqrt{\frac{64 q^3 c^3}{\pi^3 L^3 S^3}} = \frac{4}{3} \sqrt{\frac{q^3 c^3}{\pi L^3 S^3}} = \sqrt{\frac{16 q^3 c^3}{9 \pi L^3 S^3}}$ , an expression well adapted to logarithmic computation, which is independent of the length of XY.

Example:—Suppose a piece of copper-wire, 376.5 inches long, to weigh 5 ounces, what is its diameter, and what is the solidity of the greatest globe which can be taken out of its cross-section?

Let  $L = 376.5$ ,  $S = 9008$ ,  $c = 1728$ ,  $q = 5$ , and  $\pi = 3.14159$ , &c.

Then Log. 4. = 0.6020600

Log.  $q$ . = 0.6989700

Log.  $c$ . = 3.2375437

Sub-log.  $\pi$ . = 9.5028501

Sub-log.  $L$ . = 7.4242350

Sub-log.  $S$ . = 6.0453716

Reject 30. .. 27.5110304

2) 3.5110304

Diameter of the wire = .0569528 = 2.7555152

3

4.2665456

Log.  $\frac{\pi}{6}$  ..... 1.7189986

Log. .00009672622 = 5.9855442

So that the diameter and solidity of a globe placed in the position of any wire XY, can be determined to any degree of accuracy required. If the part XY be taken from such a wire as the one given in the example, the diameter would be .0569528 inches, and the solidity of  $n$ , .00009672622 cubic inches. The solidity of any other globule,  $a$ ,  $b$ ,  $c$ , or  $d$ , &c., suspended between the planes of glass, can be readily found, from knowing the distance of its centre from the line AB, where the planes meet; and the distance of the axis of the cylinder from the same line. These distances may be measured in a slant line by the scales AC, BD, and will answer as well as the perpendicular distances from the axis and centre. If great accuracy be required, two distances are necessary,—the one above, and the other below each globule: for instance, let us take the globule  $d$ , the distance of its lower limb from AB is 61, according to the diagram given in Fig. 49, and the distance of the upper limb is 69; therefore its centre may be said to be  $\frac{61 + 69}{2} = 65$  parts from the line AB. It is convenient, but not necessary



to have the wire  $XY$  so placed, that the distance of its axis  $AB$  may correspond with 10, 100, 1000, &c., on the scales  $AC$ ,  $BD$ , &c.

Suppose the wire  $n$  to be so placed, that  $s't'$  reads on the scale  $AC$  and  $BD$ , referring to the first figure at 1002, and  $w'v'$  to read 998; then  $\frac{1002 + 998}{2} = 1000$ , the best position for the wire which keeps the planes apart. Let  $m'n'$  be at 645, and  $p'q'$  at 643, then the globule may be said to stand at  $\frac{645 + 643}{2} = 644$ .

Indeed, if the globules were read off all above, or all below, it would answer the same purpose—observing to keep the lines  $s't'$ ,  $m'n'$ , or  $w'v'$ ,  $p'q'$ , &c., parallel.

Then  $1000^3 : 644^3 :: S : S^4$ ;— $S$  and  $S^4$  being the solid contents of  $n$  and  $d$  respectively.

$$\therefore S^4 = \frac{644^3 S}{1000000000}.$$

$\therefore 1000^3 : 644^3 :: W : W^4$ ;  $W$  and  $W^4$  being the weights of  $n$  and  $d$  respectively.

$$\text{Hence } W^4 = \frac{644^3 W}{1000000000}.$$

The weights and solidities, or volumes of the small globules  $a, b, c, d, e, f, g, h, i$ , &c., supposing them to stand at 947, 895, 763, 644, 602, &c., respectively, will be in like manner expressed by—

$$\begin{array}{lll} a & . & . & . & W^1 = \frac{947^3 W}{1000^3}; S^1 = \frac{947^3 S}{1000^3} \\ b & . & . & . & W^2 = \frac{895^3 W}{1000^3}; S^2 = \frac{895^3 S}{1000^3} \\ c & . & . & . & W^3 = \frac{763^3 W}{1000^3}; S^3 = \frac{763^3 S}{1000^3} \\ d & . & . & . & W^4 = \frac{644^3 W}{1000^3}; S^4 = \frac{644^3 S}{1000^3} \\ e & . & . & . & W^5 = \frac{602^3 W}{1000^3}; S^5 = \frac{602^3 S}{1000^3} \\ \&c. & & & \&c. & & \&c. \end{array}$$

$$\therefore W^1 + W^2 + W^3 + W^4 + \&c., = W \left\{ \frac{947^3 + 895^3 + 763^3 + 644^3 + \&c.}{1000000000} \right\};$$

$$\text{And } S^1 + S^2 + S^3 + S^4 + \&c., = S \left\{ \frac{947^3 + 895^3 + 763^3 + 644^3 + \&c.}{1000000000} \right\}.$$

The co-efficient,  $\left\{ \frac{947^3 + 895^3 + 763^3 + 644^3 + \&c.}{1000000000} \right\}$ , or similar ones, can

be determined with great ease by the ordinary table of the cubes of numbers; in fact, they may be determined by addition. If this co-efficient =  $f$ ,  $W^1 + W^2 + W^3 + W^4 + \&c., = fW$ .

$$\therefore W = \frac{W^1 + W^2 + W^3 + W^4 + \&c.}{f}.$$

It is evident that if the weights  $W^1, W^2, W^3$ , &c., of a great number of globules,  $a, b, c, d$ , &c., of any sort of metal, be taken together, with



a very fine balance, and the co-efficient  $f$  determined by a table of cubes, the weight  $W$  of the globule  $n$  can be determined with the greatest possible accuracy, no matter what the specific gravity of the globules may be.

Thus having determined the Weight and Volume of  $n$ , (and this may be done to any degree of accuracy,) the weight and volume of any other globule, or number of globules, may be at once determined, from—

$$W^1 + W^2 + W^3 + W^4 + \&c. = W \left\{ \frac{947^3 + 895^3 + 763^3 + \&c.}{1000000000} \right\} = W f.$$

$$S^1 + S^2 + S^3 + S^4 + \&c. = S f; \text{ or}$$

$$\text{Log. } (W^1 + W^2 + W^3 + \&c.) = \text{log. } W + \text{log. } f.$$

$$\text{Log. } (S^1 + S^2 + S^3 + \&c.) = \text{log. } S + \text{log. } f.$$

It may be remarked that a detached scale on box or ivory, will answer the same purpose as the detached scales  $AC$ ,  $BD$ .

Fig. 49.

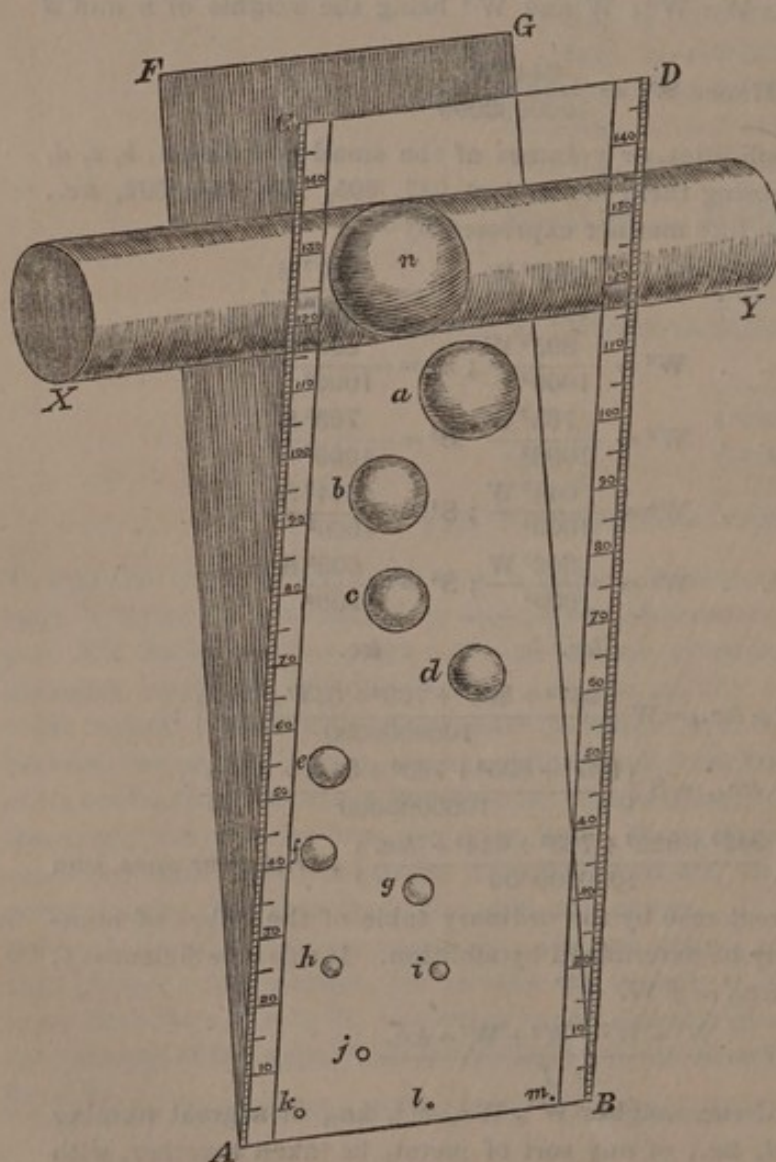
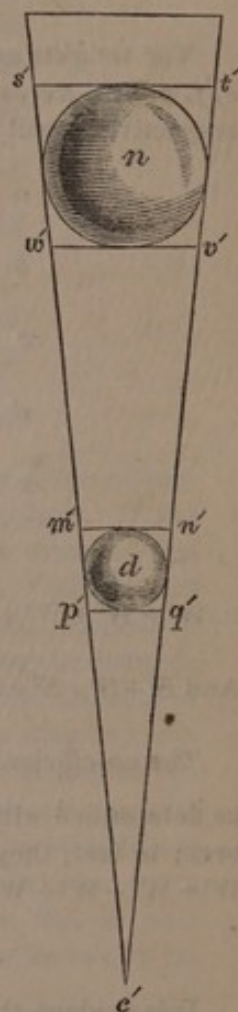


Fig. 50.





## (II.)

## TABLE OF ATOMIC WEIGHTS,

WITH THEIR LOGARITHMS FOR CALCULATIONS IN ANALYSIS.\*

Name.	Symbol.	Atomic Weight.	Logarithm.
Aluminum . . . .	Al	171.17	2.233427
Ammonia . . . .	N H <sup>3</sup>	213.13	2.32864
Antimony . . . .	Sb	1612.90	3.20761
oxide of . . . .	Sb O <sup>3</sup>	1912.90	3.28169
sulphuret of . .	Sb S <sup>3</sup>	2216.40	3.34565
Arsenic . . . . .	As	940.10	2.973174
sulphuret of . .	As S <sup>3</sup>	1543.6	3.18854
sulphuret of . .	As S <sup>5</sup>	1945.9	3.28912
Arsenic acid . . . .	As O <sup>5</sup>	1440.1	3.15839
Arsenious acid . . .	As O <sup>3</sup>	1240.1	3.09346
Barium . . . . .	Ba	856.88	2.93292
chloride of . . .	Ba Cl	1299.5	3.11378
Barytes . . . . .	Ba O	956.88	2.98086
carbonate of . .	Ba O, C O <sup>2</sup>	1231.9	3.09058
phosphate of . .	2 Ba O, P O <sup>5</sup>	2806.1	3.44811
sulphate of . . .	Ba O, S O <sup>3</sup>	1458.1	3.16379
Bismuth . . . . .	Bi	886.92	2.947884
Boron . . . . .	B	136.25	2.134336
Bromine . . . . .	Br	978.31	2.990476
Cadmium . . . . .	Cd	696.77	2.843089
Calcium . . . . .	Ca	256.02	2.408273
chloride of . . .	Ca Cl	698.67	2.84427
Carbon . . . . .	C	75.00	1.87506
Carbonic acid . . . .	C O <sup>2</sup>	275.00	2.43933
Carbonic oxide . . . .	C O	175.00	2.24304
Cerium . . . . .	Ce	574.70	2.759441
Chlorine . . . . .	Cl	442.65	2.646060
Chromium . . . . .	Cr	351.82	2.546320
Cobalt . . . . .	Co	368.99	2.567015

\* [These tables are taken from Parnell's Chemistry, as they may very often be of service to the analyst.]—*Trans.*



Name.	Symbol.	Atomic Weight.	Logarithm.
Columbium (tantalum) .	Ta	2307.43	3.363128
Copper . . . .	Cu	395.70	2.59737
protoxide of . .	Cu O	495.70	2.69522
suboxide of . .	Cu <sup>2</sup> O	891.39	2.95007
Fluorine . . . .	F	233.8	2.368845
Glucinum . . . .	G	331.26	2.520168
Gold . . . . .	Au	2486.03	3.395506
Hydrogen . . . .	H	12.48	1.09621
Iodine . . . . .	I	1579.50	3.198520
Iridium . . . . .	Ir	1233.50	3.091138
Iron . . . . .	Fe	339.21	2.53047
peroxide of . .	Fe <sup>2</sup> O <sup>3</sup>	978.43	2.99053
protoxide of . .	Fe O	439.21	2.64267
Lead . . . . .	Pb	1294.5	3.11210
carbonate of . .	Pb O, C O <sup>2</sup>	1669.5	3.22259
chloride of . .	Pb Cl	1737.1	3.23983
phosphate of . .	2 Pb O, P O <sup>5</sup>	3681.3	3.56600
protoxide of . .	Pb O	1394.5	3.14442
sulphate of . .	Pb O, S O <sup>3</sup>	1895.7	3.27777
Lime . . . . .	Ca O	356.02	2.55147
carbonate of . .	Ca O, C O <sup>2</sup>	631.03	2.80004
sulphate of . .	Ca O, S O <sup>3</sup>	857.19	2.93308
Lithium . . . . .	L	80.33	1.904878
Magnesia . . . .	Mg O	258.35	2.41220
sulphate of . .	Mg O, S O <sup>3</sup>	759.52	2.88054
Magnesium . . . .	Mg	158.35	2.199618
Manganese . . . .	Mn	345.9	2.53895
protoxide of . .	Mn O	445.9	2.64924
deutoxide of . .	Mn <sup>2</sup> O <sup>3</sup>	991.8	2.99642
Mercury . . . . .	Hg	1265.8	3.10236
subchloride of . .	$\frac{1}{2}$ (Hg <sup>2</sup> Cl)	1487.1	3.17234
oxide of . . . .	Hg O	1365.8	3.13539
suboxide of . .	Hg <sup>2</sup> O	2631.6	3.42022
Molybdenum . . . .	Mo	598.52	2.777078
Nickel . . . . .	Ni	369.68	2.567826



Name.	Symbol.	Atomic Weight.	Logarithm.
Nitrogen . . . .	N	175.6	2.24452
Osmium . . . .	Os	1244.49	3.094989
Oxygen . . . .	O	100.00	2.000000
Palladium . . . .	Pd	665.9	2.823409
Phosphoric acid . .	P O <sup>5</sup>	892.31	2.95052
Phosphorous acid . .	P O <sup>3</sup>	692.21	2.84030
Phosphorus . . . .	P	392.31	2.59363
Platinum . . . .	Pt	1233.3	3.09107
ammonio-chloride of	{ (Pt Cl <sup>2</sup> + N H <sup>4</sup> Cl)	2786.5	3.445059
potassio-chloride of.	{ (Pt Cl <sup>2</sup> + K Cl)	3051.2	3.48447
Potash . . . .	K O	589.92	2.77079
carbonate of . .	K O, C O <sup>2</sup>	864.93	2.93698
sulphate of . .	K O, S O <sup>3</sup>	1091.1	3.03786
Potassium . . . .	K	489.92	2.69013
chloride of. . .	K Cl	932.57	2.96968
Rhodium . . . .	R	651.39	2.813841
Selenium . . . .	Se	494.58	2.694236
Silicon . . . .	Si	277.31	2.442965
Silver . . . .	Ag	1351.6	3.13085
chloride of . .	Ag Cl	1794.3	3.25389
oxide of . . . .	Ag O	1451.6	3.16185
ditto . . . .	2	2903.2	3.46288
ditto . . . .	3	4354.8	3.63897
ditto . . . .	4	5806.4	3.76391
ditto . . . .	5	7258.0	3.86082
Soda. . . .	Na O	390.9	2.59207
carbonate of . .	Na O, C O <sup>2</sup>	665.91	2.82341
sulphate of . .	Na O, S O <sup>3</sup>	892.07	2.95040
Sodium . . . .	Na	290.9	2.46374
chloride of . .	Na Cl	733.55	2.86543
Strontian . . . .	Sr O	647.29	2.81110
carbonate of . .	Sr O, C O <sup>2</sup>	922.3	2.96487
sulphate of. . .	Sr O, S O <sup>3</sup>	1148.5	3.060131



Name.	Symbol.	Atomic Weight.	Logarithm.
Strontium . . . .	Sr	547.29	2.738217
chloride of . . . .	Sr Cl	989.94	2.99561
Sulphur . . . .	S	201.17	2.30356
Sulphuric acid . . . .	S O <sup>3</sup>	501.17	2.69998
Sulphurous acid . . . .	S O <sup>2</sup>	401.17	2.60333
Tellurium . . . .	Te	801.76	2.904044
Thorium . . . .	Th	744.9	2.872098
Tin . . . .	Sn	735.29	2.86645
peroxide of . . . .	Sn O <sup>2</sup>	935.29	2.97095
protoxide of . . . .	Sn O	835.29	2.92184
Titanium . . . .	Ti	303.66	2.482387
Tungsten . . . .	W	1183.00	3.072985
Uranium . . . .	U	2711.36 *	3.433187
Vanadium . . . .	V	856.89	2.932924
Water . . . .	H O	112.48	2.05107
Ditto . . . .	2	224.96	2.35210
Ditto . . . .	3	337.44	2.52820
Ditto . . . .	4	449.92	2.65314
Ditto . . . .	5	562.40	2.75005
Ditto . . . .	6	674.88	2.82923
Ditto . . . .	7	787.36	2.89618
Ditto . . . .	8	899.84	2.95417
Ditto . . . .	9	1012.3	3.00531
Ditto . . . .	10	1124.8	3.05107
Ditto . . . .	11	1237.3	3.09248
Ditto . . . .	12	1349.8	3.13027
Ditto . . . .	13	1462.2	3.16501
Yttrium . . . .	Y	402.51	2.604776
Zinc . . . .	Zn	403.23	2.60555
oxide of . . . .	Zn O	503.23	2.70177
. . . .	2	1006.5	3.00281
. . . .	3	1509.7	3.17889
. . . .	4	2012.9	3.30382
Zirconium . . . .	Zr	420.20	2.623456

\* [Rammelsberg, after a series of carefully-conducted experiments, has found the atomic weight of Uranium to be only 750.00].—*Trans.*



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### ERRATA.

- Page 58, line 3 from bottom, ( note ) *for* "is readily decomposed," *read* "decomposes the glass."
- „ 100, „ 17 from top, . *for* "Rantenspar," *read* "Rautenspar."
- „ 136, „ 9 from bottom, *for* "S O<sub>3</sub>," *read* "Si O<sub>3</sub>."
- „ 152, „ 11 from top, . *for* "Co S" *read* "Cu S"; and *for* "3 Co S" *read* "3 Cu S".
- „ 168, „ 10 from bottom, *for* "Fuchroite," *read* "Euchroite."
- „ 173, „ 12 from bottom, *for* "copper, nickel, or arsenic," *read* "the copper, nickel, and arsenic."
- „ 233, „ 13 from top, . *for* "chloride of lime," *read* "chloride of calcium."



