

**A course of analytical chemistry (qualitative and quantitative) : to which is prefixed a brief treatise upon modern chemical nomenclature and notation / by William W. Pink and George E. Webster.**

### **Contributors**

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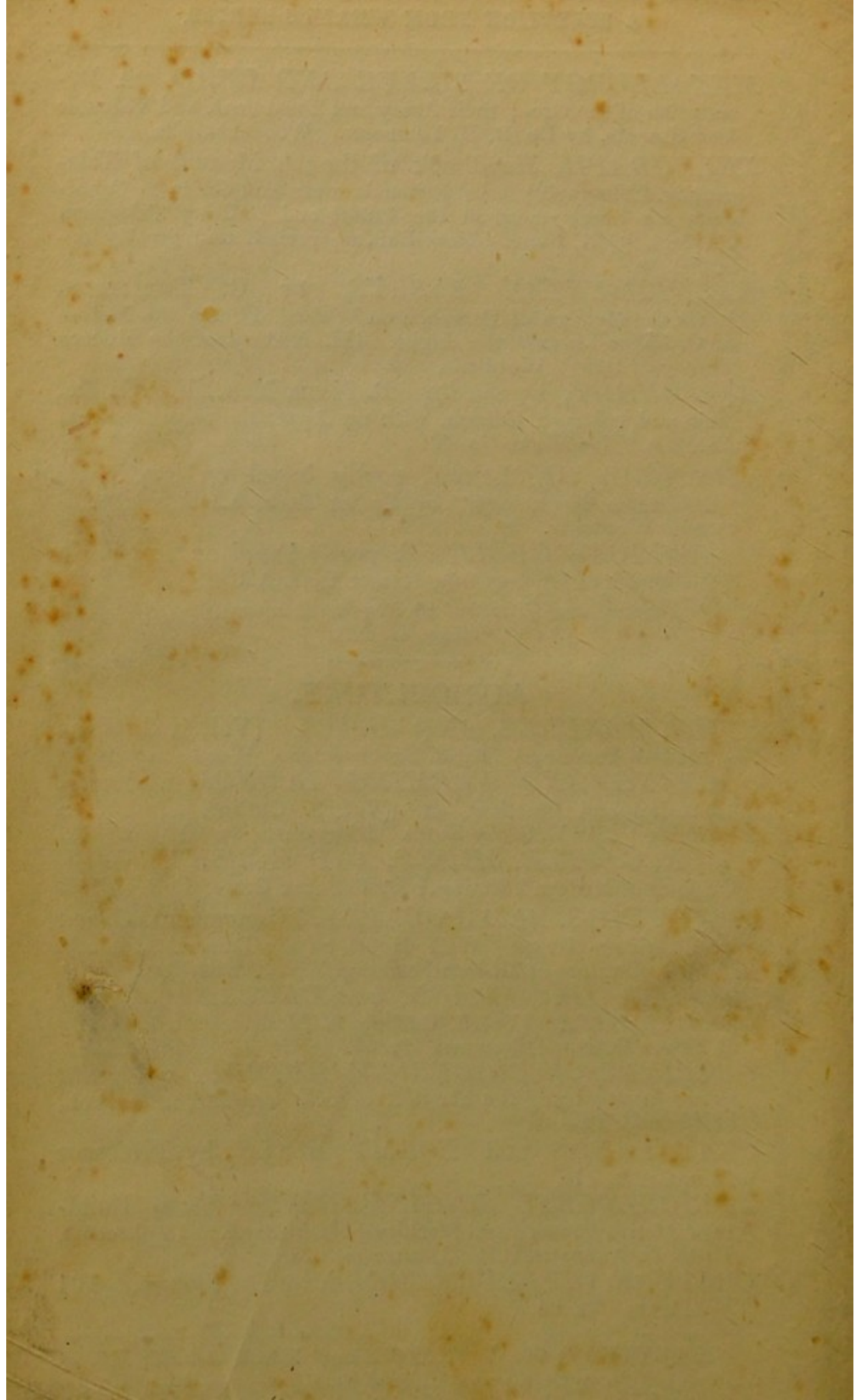
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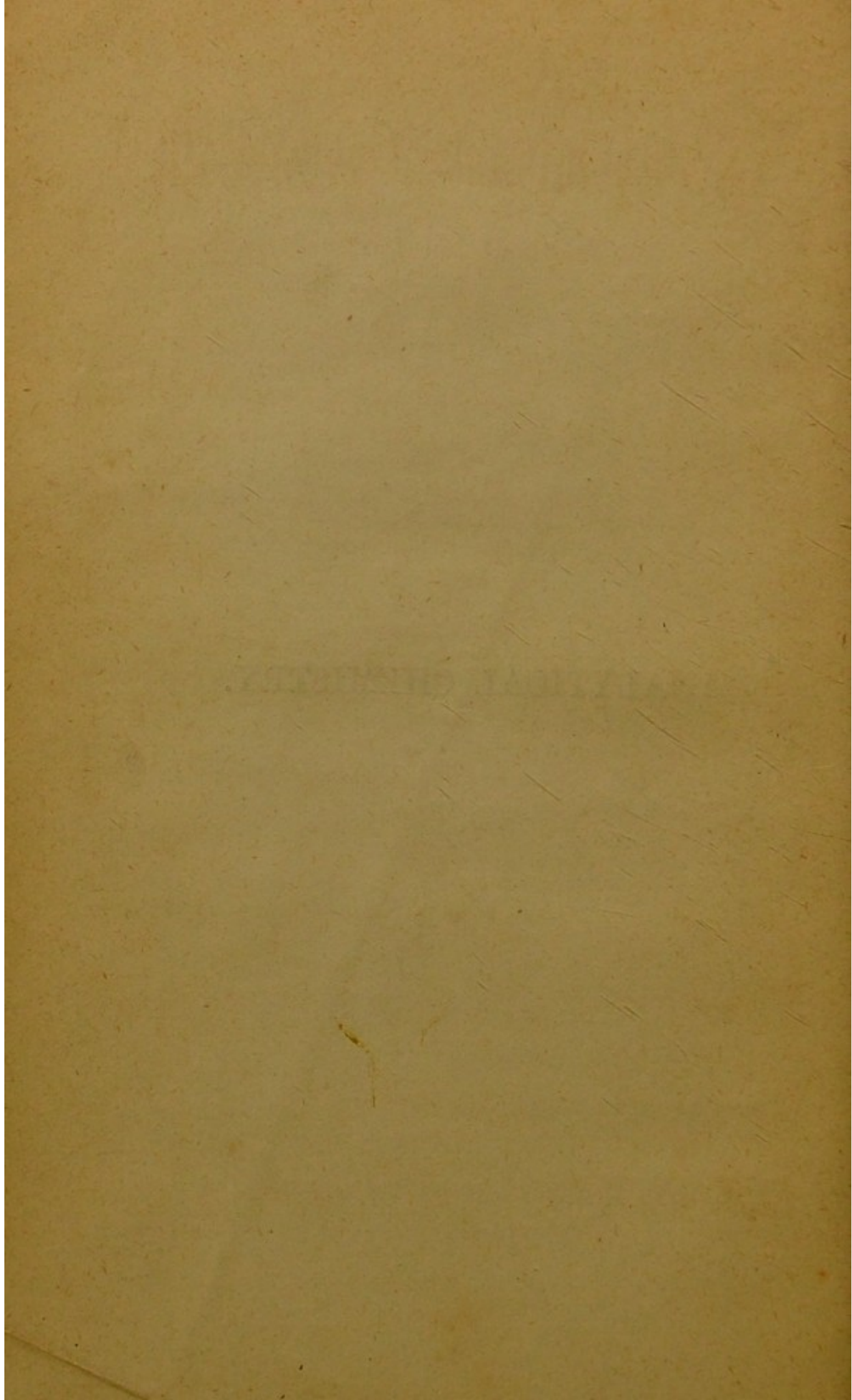
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ANALYTICAL CHEMISTRY.



*A COURSE OF*  
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(Qualitative and Quantitative)

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*A BRIEF TREATISE UPON MODERN CHEMICAL  
NOMENCLATURE AND NOTATION.*

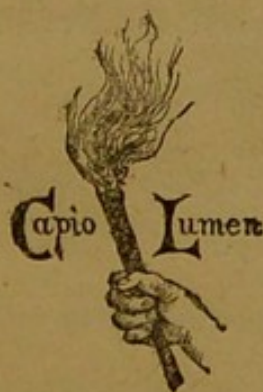
BY WILLIAM W. PINK

PRACTICAL CHEMIST AND METALLURGICAL ANALYST.

AND

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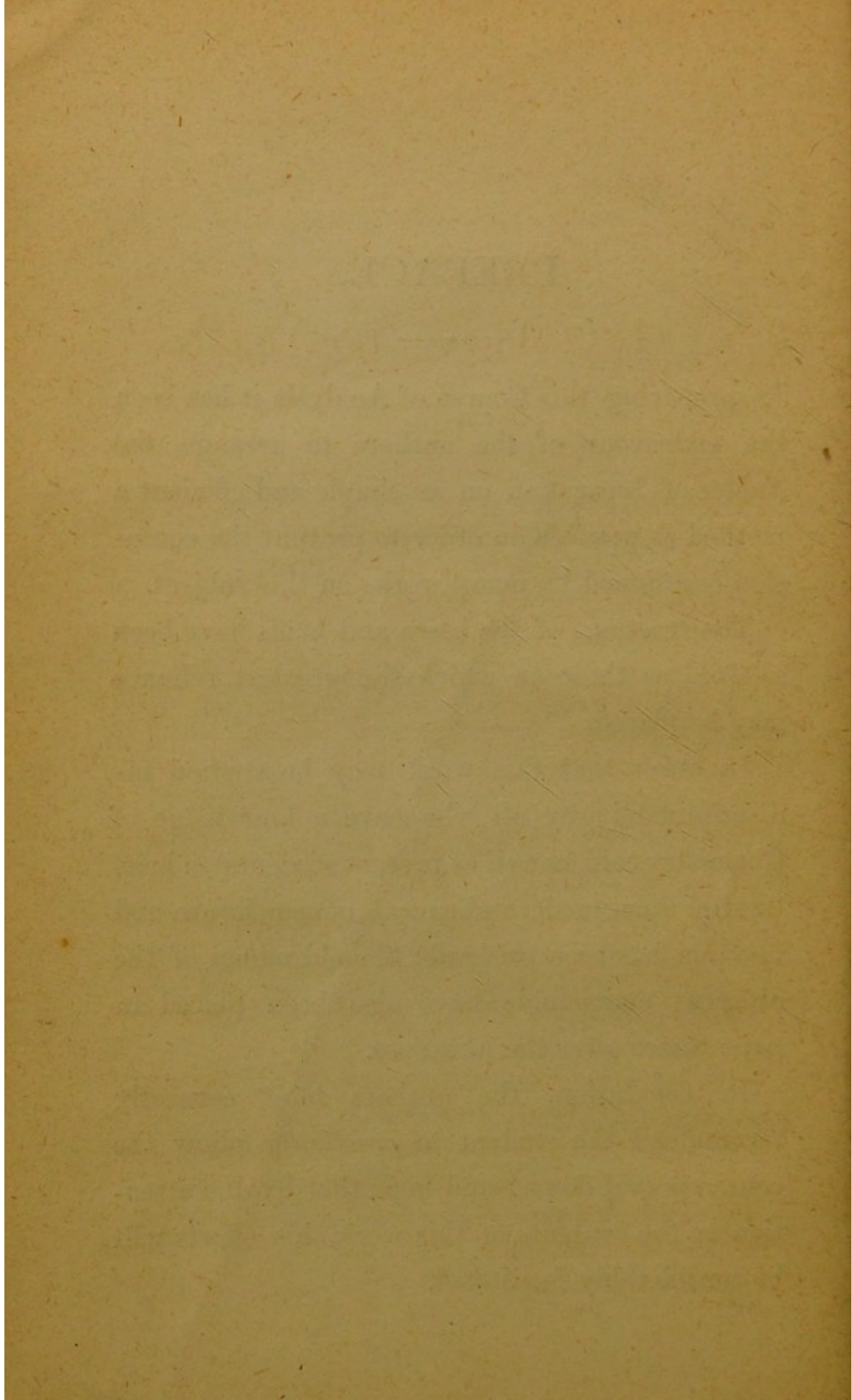
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IN preparing this Course of Analysis it has been the endeavour of the authors to arrange the Tables of Separation on as simple and efficient a method as possible, in order to prevent the confusion occasioned by many works on this subject.

The reactions of the bases and acids have been selected as those on which the greatest reliance may be placed.

In order that this work may be studied indiscriminately by all who have a knowledge of Chemistry (old as well as present students), a brief treatise upon modern chemical nomenclature and notation has been prefixed; the old names of the different compounds have also been placed in parentheses after the new ones.

In conclusion, the authors most earnestly recommend the student to *practically* follow the course as laid down; and hope that by due attention to the contents of this work, his efforts will be considerably facilitated.



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## ERRATA.

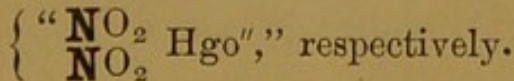
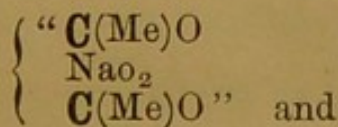
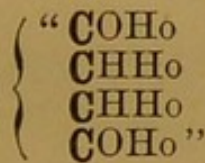
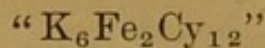
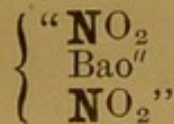
Page 23, first example, for "1 crith," read "16 criths."

Page 32, par. 2, beginning of line 2, for "lie," read "be."

Page 36, lines 1 and 3, for names in brackets, read "(chloride of lime)" and "(chloride of soda)"; for "K" in formula of tartaric acid, read "H," and omit "(cream of tartar)"; last line, for "tumeric," read "turmeric."

Page 38, line 18, for "It," read " $\text{SO}_2\text{Ho}_2$ ," and omit "for."

Pages 41, 50, 51, 109, and 113, instead of formulæ as printed,  
read

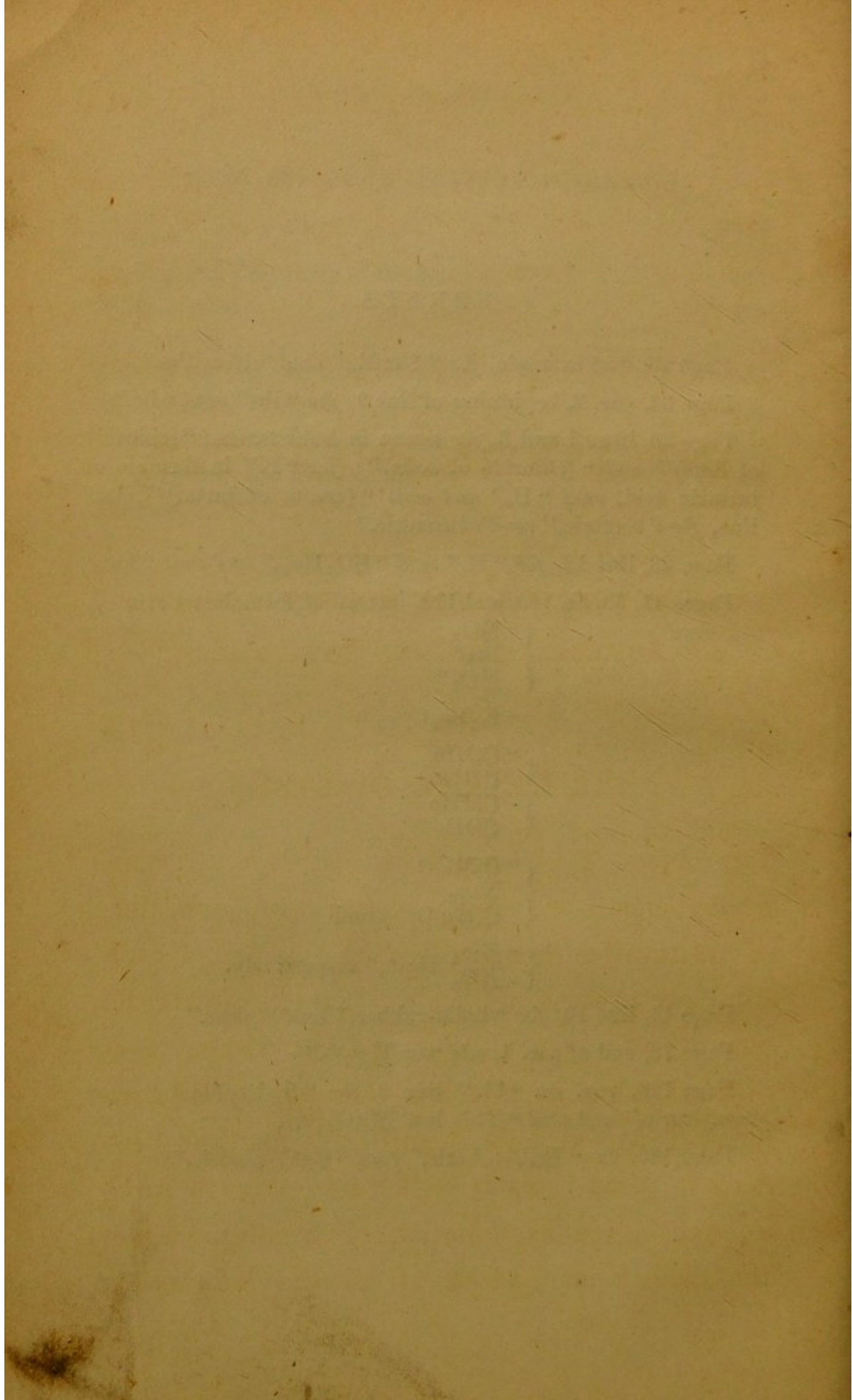


Page 46, line 10, for "india-rubber," read "glass."

Page 78, end of par. 1, add "or  $\text{Hg}_2\text{O}$ ."

Page 120, par. on "Cl.," line 3, for "hydrochloric," read "sulphuric," and omit "This last  $\text{MnO}_2$ , &c."

Page 136, for " $\text{BaCl}_2$ , baric," read " $\text{CaCl}_2$ , calcic."



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PRELIMINARY REMARKS.

THE object of qualitative analysis is to ascertain what elements are present in any compound, that of quantitative to determine the quantity of each substance present, and, as we cannot estimate the quantity of any particular element without first distinguishing its presence, therefore, qualitative must always precede quantitative analysis; indeed in some cases it is not necessary to make a quantitative examination of a substance, as the quality may afford us all the information

required for fixing the proportions in which they are combined together. Qualitative and quantitative analyses are thus very closely allied, and the one should never be studied without going through the other.

---

## MODERN CHEMICAL NOMENCLATURE AND NOTATION.

The student must speedily endeavour to obtain a complete knowledge of the composition of bodies (if he has not already done so), and make himself conversant with the formulæ, or notation, by which the number of equivalents which enter into the composition of substances are expressed. In order to facilitate this acquisition, he should study the following tables, especially for the first part of this work, the Atomic Weights; and for the second, the Specific Gravities.

Names of Simple Bodies.	Symbols.	Atomic Weights Hydrogen = 1.	Specific Gravities as Solids or Liquids. Water=unity.
Hydrogen .....	H	1.	—
Chlorine .....	Cl	35.5	—
Bromine .....	Br	80	2.96 to 2.97
Iodine .....	I	127	5
Fluorine .....	F	19	—
Oxygen .....	O	16	—
Sulphur .....	S	32	2.07 to 2.08
Selenium .....	Se	79	4.28
Carbon .....	C	12	3.5
Silicon .....	Si	28.5	2.49
Boron .....	B	11	2.69
Nitrogen .....	N	14	—
Phosphorus .....	P	31	1.77

TABLE OF METALLIC ELEMENTS.

Names of Simple Bodies.	Symbols.	Atomic Weights Hydrogen = 1.	Specific Gravities as Solids or Liquids. Water=unity.
Potassium (Kalium) ....	K	39	0.865
Sodium (Natrium) .....	Na	23	0.972
Lithium .....	Li	7	0.59
Barium .....	Ba	137	4.5
Strontium .....	Sr	87.5	2.5
Calcium .....	Ca	40	1.6
Magnesium .....	Mg	24	1.7
Aluminium .....	Al	27.5	2.6
Iron (Ferrum) .....	Fe	56	7.79
Chromium .....	Cr	52.5	7
Manganese .....	Mn	55	6.86
Zinc .....	Zn	65	7
Nickel .....	Ni	58.8	8.8
Cobalt .....	Co	58.8	8.54
Tin (Stannum) .....	Sn	118	7.29
Antimony (Stibium) ....	Sb	122	6.7
Arsenic .....	As	75	5.88
Bismuth .....	Bi	208	9.82
Cadmium .....	Cd	112	8.7
Copper (Cuprum) .....	Cu	63.5	8.96
Platinum .....	Pt	197.3	21.5
Lead (Plumbum) .....	Pb	207	11.45
Mercury (Hydrargyrum)	Hg	200	13.59
Silver (Argentum) .....	Ag	108	10.47
Gold (Aurum) .....	Au	196.7	19.5

Some of the elements and compounds have popular names, such as quicksilver for mercury, brimstone for sulphur, muriatic acid for hydrochloric, &c.; these have, in most cases, been given after their proper or scientific name.

**SPECIFIC GRAVITY OR WEIGHT** is the number which expresses the ratio which the weight of a cubic inch of the body has to the weight of a cubic inch of distilled water, at a temperature  $15.55^{\circ}$  C., or  $60^{\circ}$  (Fah.). For gases.—Hydrogen is taken for unity, and their specific gravities referred to that standard.

**ATOMS AND ATOMIC WEIGHTS.** All bodies are supposed to be built up of an infinite number of small particles called *Atoms*; and further that these *Atoms*, with one or two exceptions, are all the same size, but of *different* weights, which difference is expressed by the atomic weights of the elements. The exceptions are arsenic and phosphorus, whose atoms are supposed to be half the usual size; and mercury, zinc, and cadmium, whose atoms are twice the size. An atom is defined by Dr. Dalton (whose name the theory bears), to be the least part of an elementary body which can enter into, or be expelled from, a compound; but it is sometimes applied to compound,

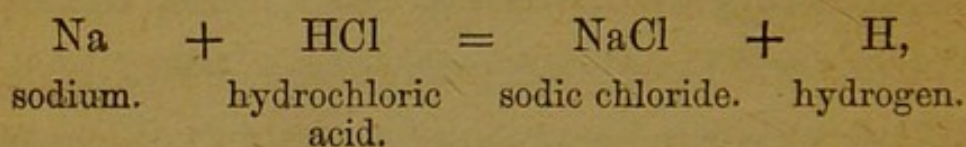
as well as simple radicals, such as ammonia, hydroxyl, &c.

The *Atomic Weights* are only relative weights referred to *hydrogen as unity*. If, therefore, it is stated that the weight of oxygen is 16, it is meant that the weight of an atom of oxygen is 16 times that of hydrogen. For fixed solids which do not vapourize, the atomic weights are referred to the element lithium, the atomic weight being determined by the amount of heat which any body contains when it is at the same temperature as lithium, both being the same weight, lithium being considered as seven. For example: It requires 55 parts of manganese, 65 of zinc, or 63·5 of copper, when heated to 150° C. or 302° F., to raise a pound or litre of water through as many degrees of temperature as 7 parts of lithium would do, so that 55, 65, and 63·5 are the atomic weights of manganese, zinc, and copper respectively.

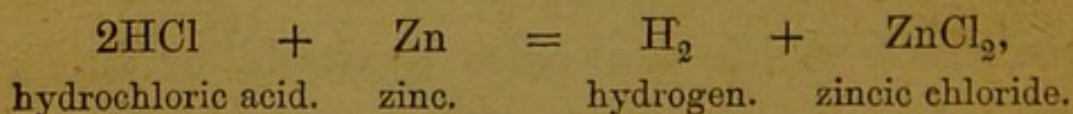
**MOLECULES AND BONDS.** The term molecule is given to the smallest portion of an element or compound which can exist by itself, or in an uncombined state. All molecules, by the law of Ampère, are the same size, and "all gases and vapours contain the same number of molecules within the same volume," though some of the

elementary molecules consist of pairs of atoms, and the molecular weight is then twice the atomic weight. Also, the molecules of compounds occupy the same volume as one molecule of hydrogen, no matter how many volumes the constituents may measure.

In chemical changes the atoms of elementary bodies have very different values. For example: if hydrochloric acid (HCl) and sodium be allowed to act on each other we shall have the sodium replacing the hydrogen (which will be given off) atom for atom, thus—



But if we substitute zinc for the sodium, we shall have two atoms of hydrogen displaced for each atom of zinc, which combines with the chlorine, or it seems that the power of combination of zinc were twice that of hydrogen.



Similarly an atom of nitrogen or arsenic can be substituted for five, tin for four, iron or cobalt for six of any monad element such as hydrogen.

This difference of attraction is called the bond affinity, that is, it is assumed that the different atoms possess power, lines of force, or *points of attraction*, called by Dr. Frankland, *bonds*; thus—chlorine and sodium are single bonded, tin has four bonds, nitrogen and arsenic, five, iron and cobalt, six, &c.

The following list expresses the bonds of most of the common elements, which have been classified according to their highest known atomicities. For example: Although an artiad can never become a perissad, nor a perissad an artiad, yet the pentads frequently take the form of triads or monads, and the hexads, tetrads or diads.

#### ATOMICITY OF THE MOST COMMON ELEMENTS.

##### *Perissads (Atomicity odd).*

<u>MONADS (1).</u>	<u>TRIADS (3).</u>	<u>PENTADS (5).</u>
Hydrogen.	Boron.	Nitrogen.
Chlorine.	Gold.	Arsenic.
Bromine.		Phosphorus.
Iodine.		Antimony.
Fluorine.		Bismuth.
Silver.		
Sodium.		
Lithium.		
Potassium.		

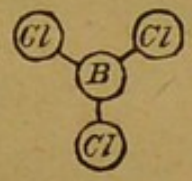
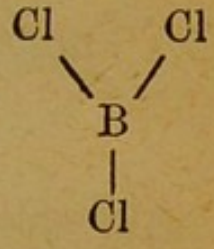

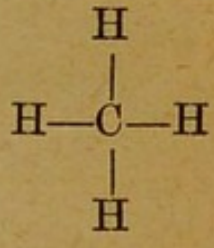
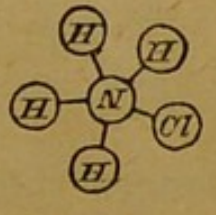
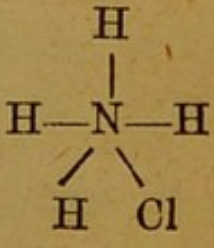
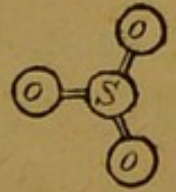
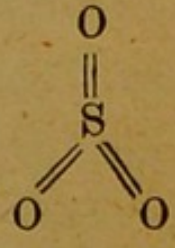
*Artiads (Atomicity even).*

<u>DIADS (2).</u>	<u>TETRAIDS (4).</u>	<u>HEXADS (6).</u>
Oxygen.	Carbon.	Selenium.
Cadmium.	Silicon.	Sulphur.
Mercury.	Lead.	Nickel.
Copper.	Platinum.	Cobalt.
Zinc.	Palladium.	Iron.
Magnesium.	Aluminium.	Manganese.
Barium.	Titanium.	Chromium.
Strontium.	Tin.	
Calcium.		

These bonds are expressed in various ways, both for the atom and the compounds. First, for lecture halls and young students, we have the *Graphic representation*. Thus  $\text{Cl}$  chlorine,  $\text{Zn}$  zinc,  $\text{B}$  boron,  $\text{C}$  carbon,  $\text{N}$  nitrogen,  $\text{S}$  sulphur. Secondly, the method of using dashes and Roman numerals to indicate the atomicity, introduced by Dr. Odling, these are placed *above* and to the right of the symbol; thus,  $\text{Cl}^{\text{I}}$ ,  $\text{Zn}^{\text{II}}$ ,  $\text{B}^{\text{III}}$ ,  $\text{C}^{\text{IV}}$ ,  $\text{N}^{\text{V}}$ ,  $\text{S}^{\text{VI}}$ , the dashes or numerals express the number of bonds possessed by each element.

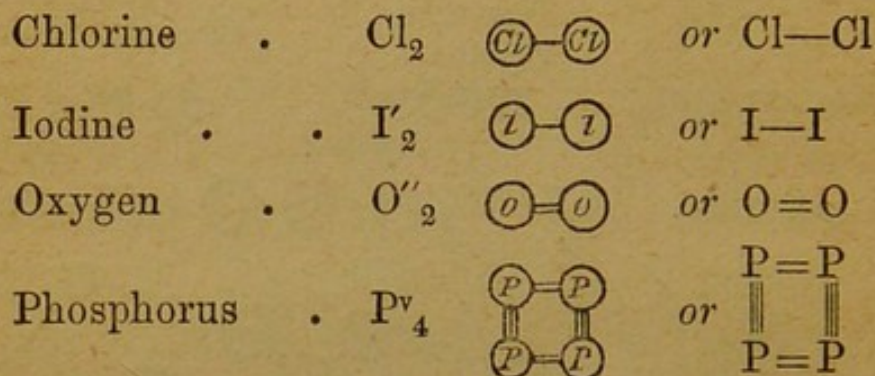
When the elements are in combination the graphic and symbolic formulæ show how they

are severally combined. The following are examples of the two methods of expression:—

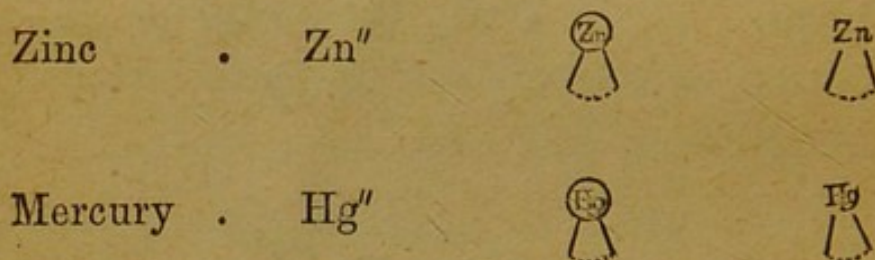
	SYMBOLIC FORMULÆ.	GRAPHIC FORMULÆ.
Potassic chloride,	$K^I Cl$	$\textcircled{K}-\textcircled{Cl}$ or $K-Cl$
Zincic chloride,	$Zn^{II} Cl_2$	$\textcircled{Cl}-\textcircled{Zn}-\textcircled{Cl}$ or $Cl-Zn-Cl$
Boric chloride,	$B^{III} Cl_3$	 or 
Marsh gas,	$C^{IV} H_4$	 or 
Ammonic chloride, $N^V H_4 Cl$		 or 
Sulphuric anhydride, $S^{IV} O''_3$		 or 

In the above cases, all the bonds of the elements are engaged, and the molecules are complete, that is, the bonds of one atom are united to those of another, and not to itself, or an atom of a similar element. There are cases, however, in

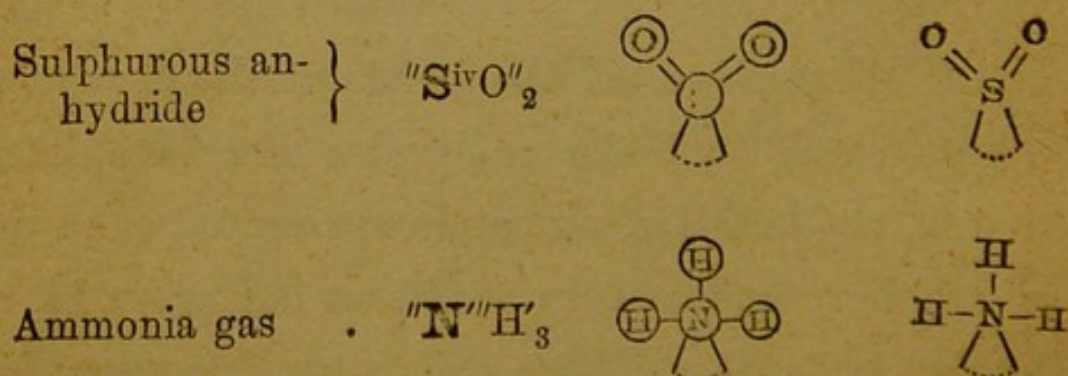
which the bonds are connected to the bonds of an atom of a similar element; for example:—



In some cases we have a diad atom's bonds satisfying each other and forming a monatomic molecule.



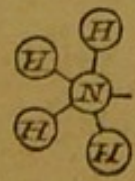
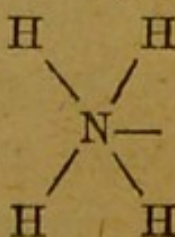


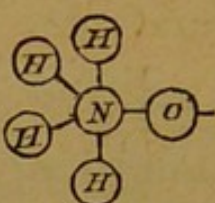
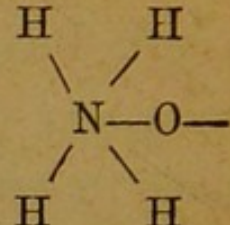
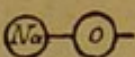
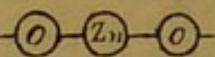
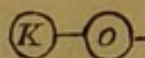
In some instances the elementary bonds are not all united to those of another element, but part to themselves, as in sulphuretted hydrogen and nitrous oxide.



The highest number of bonds that an element is ever known to engage is called its absolute atomicity; those actually engaged in any compound its active atomicity; and those not engaged its latent atomicity. An atom must always have all its bonds engaged by other bonds; therefore if we have either a monad element or a compound with one bond unsatisfied, it cannot exist in a free state. When a compound such as hydroxyl  $\text{H}-\text{O}-$  does occur in a compound in this state, it is called a COMPOUND RADICAL, and is capable of performing all the functions of an elementary body (which is a *Simple Radical*).

The following is a list of the most important of the compound radicals, with their formulæ, graphic and symbolic:—

NAME.	SYMBOLIC.	GRAPHIC.
Hydroxyl	. Ho'	$-\text{O}-\text{H}$ or $\text{H}-\text{O}-$
Hydrosulphyl	. Hs'	 or 
Ammonim	. Am'	 or 

NAME.	SYMBOLIC.	GRAPHIC.
Ammonoxyl	. Amo'	 <i>or</i> 
Sodoxyl	. Nao'	 <i>or</i> Na—O—
Zincoxyl	. Zno''	 <i>or</i> —O—Zn—O—
Potassoxyl	. Ko'	 <i>or</i> K—O—

NOTATION, OR FORMULÆ. There are four different forms of notation, or formulæ, in present use, two of which are graphical, viz., the "*Glyptic*" ( $\gamma\lambda\upsilon\pi\tau\omicron\varsigma$ , carved) and *Graphic* formulæ. The other two, viz., the *Empirical* and *Constitutional*, or *Rational*, are the symbolic representations, of which the *Constitutional* represents the glyptic and graphic, and the *Empirical* gives us only the elements engaged in a particular compound, but gives us no idea as to the mode in which the atoms of the several compounds are linked together.

GLYPTIC FORMULÆ were first introduced by Dr. Hofmann, and consist in representing the atoms by wooden balls, about two inches in diameter, and pierced with as many holes as the atoms which they are intended to represent

have bonds, and in building up a molecule they are joined together by pieces of piping and short pieces of vulcanized indiarubber. Generally, the balls are coloured, to distinguish one element from another, white for nitrogen, blue for hydrogen, green for chlorine, &c.

GRAPHIC FORMULÆ are the representations pictorially of the Glyptic, and in which the bonds are represented by circles as per examples already given; sometimes, however, the circles are omitted and only the bonds given; this method is the best and quickest. Dr. Crum Brown was the first to introduce this form of formula, it has now been adopted by Professor Frankland and generally throughout the kingdom.

Empirical formulæ are forms of symbolic formulæ (which are the most convenient), and in these the symbols are placed, without the slightest regard being made to the mode in which the bonds are satisfied, and only give us the total sum of the atoms contained in the body.

CONSTITUTIONAL OR RATIONAL formulæ are the symbolic representations of the Glyptic and Graphic formulæ, and exhibit not only what is the sum-total of the elements engaged, but also show

how each bond is satisfied. This method of working (which is the best) is that which is now generally used, and is employed throughout this work. For the reason, however, that some students have been accustomed to the Empirical formulæ, the rules of the Constitutional formulæ are given below; students who do not already understand the Constitutional formulæ are strongly advised to obtain a complete knowledge of them, not only as an addition to their knowledge, but because the other is now not recognised by many colleges or allowed in many examinations.

#### RULES FOR FORMULATING.

1. Unless the first element be a *monad*, its symbol is printed in the formula in *thick type*.

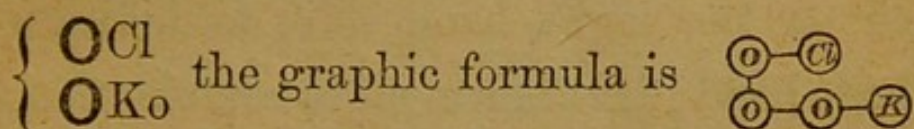
2. The symbols of the elements contained in compound radicals whose available bonds are directly united to the first element, are made to follow on the same line.

3. The symbol of the element having the greatest number of bonds engaged will, as a *rule*, be placed first.

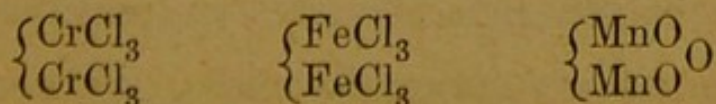
*For example*: Potassic nitrate (saltpetre), whose empirical formula ( $\mathbf{NO}_3\mathbf{K}$ , or  $\mathbf{KNO}_3$ ), when repre-

sented constitutionally, becomes  $\mathbf{NO}_2\mathbf{Ko}$ , which shows that the pentad element, nitrogen, is combined with the four bonds of the first two atoms of oxygen, and also with the remaining bond of the compound radical potassoxyl, and is, therefore, the exact representation of the *glyptic* and *graphic formulæ* of the same compound.

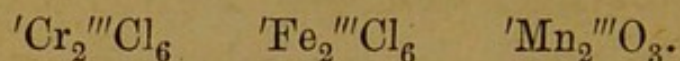
4. When two or more elements are engaged in chemical combination, as by the engagement of their bonds, we place them under one another, and connect them all by a bracket, thus, in—



that is, one bond of the second atom of oxygen is united to one bond of the first atom of oxygen, and one to the potassoxyl. The tetrad aluminium is not found in any other sort of combination, and the hexads, chromium, iron, manganese, nickel, cobalt, and uranium, when found in combination as tetrads, have almost invariably two atoms of the element, united by one each of their bonds; *e.g.*—



We can represent the same kind of combination in one line by using three atomicity marks to the right of the symbol and one to the left, thus:—

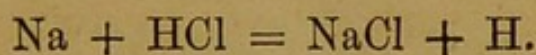


Metals of this description have been called pseudo-triads, but it would appear that they are really tetrads.

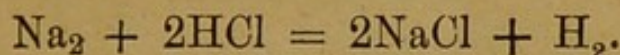
5. In the case of compound radicals we have the last element in combination represented by a small symbol, thus:  $Ko'$   $K—O—$ , a dash being placed on the right hand side, to indicate the available bond; if the element in combination with the last element is a diad, we require two dashes, thus: **Ca $o''$**  is really  $—O—Ca—O—$  or  $(CaO_2)''$ ; so that by the two dashes to the right of the symbol, we understand that the radical has two bonds at liberty, and knowing that oxygen has only two bonds (being a diad), and calcium the same, we therefore know that there must be two atoms of oxygen in combination with the calcium, which is the case.

EQUATIONS OR CHANGES. Chemical reactions or changes are represented by equations and all the elements or compounds engaged are connected

together by a + sign, the — never being used. Equations are of two forms, viz., the atomic and molecular; the atomic gives only the representation of the atomic change taking place, but as atoms cannot always exist as atoms, several becoming united into a molecule, the molecular equation which represents this is the most correct; for example, the atomic equation of the action of hydrochloric acid (HCl) on sodium is—



The Molecular is as under:—



In the case of chemical combinations there are a variety of ways in which the changes take place.

1st. By breaking or opening up of previously existing molecules, as when hydrogen and chlorine unite,  $\text{H}_2 + \text{Cl}_2 = 2\text{HCl}$ ; there are also cases in which one of two molecules upon coming into contact with each other, opens up its latent bonds, uniting itself to the second molecule, as in the formation of ( $\text{N}^{\vee}\text{H}_4\text{Cl}$ ) ammoniac chloride, which, however, suffers dissociation, or separates into two molecules at high temperatures.

2nd. By molecular combinations, or by combining without changing the active atomicity, such as in the case of water of crystallization, (distinguished by placing a comma between the molecules combined): this form of combination is very weak, and is almost always broken up by the application of moderate heat.

Crystallized ferrous sulphate,  $\text{SOHo}_2\text{Feo}''$ ,  $6\text{OH}_2$ .

„        magnesian        „         $\text{SOHo}_2\text{Mgo}''$ ,  $6\text{OH}_2$ .

## WEIGHTS AND MEASURES.

THE weights and measures generally used by chemists, are chiefly those on the French decimal system. The following tables are given to enable the student to convert these into their English equivalents, whenever he may be at a momentary loss.

### FRENCH MEASURES OF WEIGHT.

French.	In English grains.	In troy ounces = 480 grains.	In avoirdupois lbs. = 7000 grains.
Milligramme .....	0·01543	0·000032	0·0000022
Centigramme .....	0·15432	0·000322	0·0000220
Décigramme.....	1·54323	0·003215	0·0002205
Gramme.....	15·43235	0·032151	0·0022046
Décagramme.....	154·32349	0·321507	0·0220462
Hectogramme.....	1543·23488	3·215073	0·2204621
Kilogramme .....	15432·34880	32·150727	2·2046213
Myriogramme.....	154323·48800	321·507267	22·0462126

1 grain = 0·064799 gramme.  
 1 troy oz. = 31·103496 grammes.  
 1 lb. avoir. = 0·453593 kilogr.

### FRENCH MEASURES OF SURFACE.

French.	In English square feet.	In English square yards = 9 square feet.	In English poles = 272·25 square feet.
Centiare, or sq. } mètres .....	10·764299	1·196033	0·0395383
Are, or 100 sq. } mètres .....	1076·429934	119·603326	3·9538290
Hectare, or 10,000 } sq. mètres.....	107642·993418	11960·332602	395·3828959

1 square inch = 6·4513669 square centimètres.  
 1 square foot = 9·2899683 square décimètre.

## FRENCH MEASURES OF CAPACITY.

French.	In cubic inches.	In cubic feet = 1728 cubic inches.	In pints = $\frac{34.65923}{8}$ cubic inches.	In gallons = $\frac{277.27384}{8}$ cubic inches.
Millilitre, or cubic centimètre .....	0.06103	0.000035	0.00176	0.0002201
Centilitre, or 10 cubic centimètres.	0.61027	0.000353	0.01761	0.0022010
Décilitre, or 100 cubic centimètres	6.10271	0.003532	0.17608	0.0220097
Litre, or cubic décimètre ...	61.02705	0.035317	1.76077	0.2200967
Décalitre, or centistère ...	610.27052	0.353166	17.60773	2.2009668
Hectolitre, or décistère ...	6102.70515	3.531658	176.07734	22.0096677
Kilolitre, or stère, or cubic mètre	61027.05152	35.316581	1760.77341	220.0966767
Myriolitre, or decastère ...	610270.51519	353.165807	17607.73414	2200.9667675

1 cubic inch = 16.386176 cubic centimètres.

1 cubic foot = 28.315312 cubic décimètres, or litres.

1 gallon = 4.543358 litres.

## FRENCH MEASURES OF LENGTH.

French.	In English inches.	In English feet = 12 inches.	In English yards = 3 feet.
Millimètre .....	0.03937	0.003281	0.0010936
Centimètre .....	0.39371	0.032809	0.0109363
Décimètre .....	3.93708	0.328090	0.1093633
Mètre .....	39.37079	3.280899	1.0936331
Décamètre .....	393.70790	32.808992	10.9363310
Hectomètre .....	3937.07900	328.089920	109.3633100
Kilomètre .....	39370.79000	3280.899200	1093.6331000
Myriomètre .....	393707.90000	32808.992000	10936.3310000

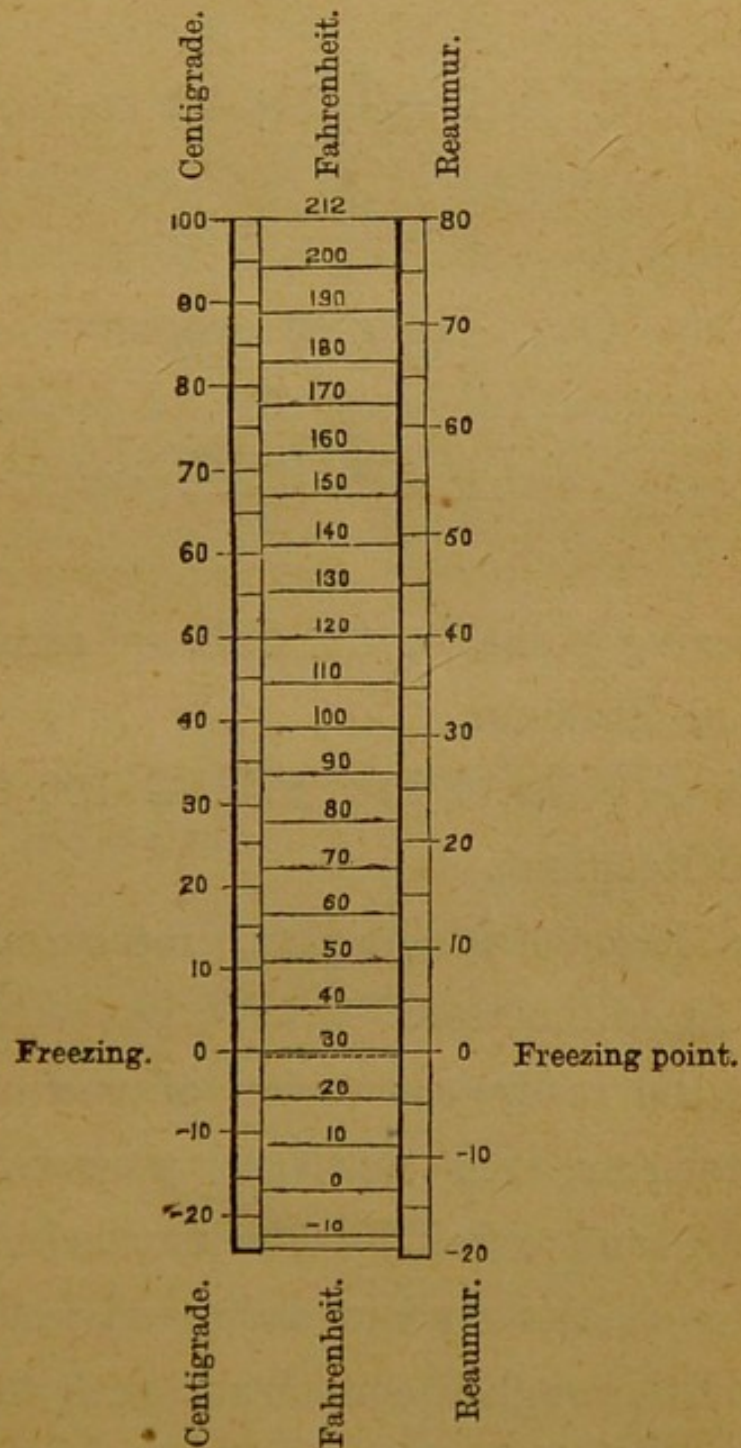
1 inch = 2.5399854 centimètres.

1 foot = 3.0479449 décimètres.

1 yard = 0.9143835 mètres.

1 mile = 1.6093149 kilomètres.

COMPARATIVE SCALE OF ENGLISH, FRENCH, AND  
RUSSIAN THERMOMETERS.



To convert degrees, Centigrade or Reaumur,  
into degrees Fahrenheit:—

Let F = number of degrees Fahrenheit.

C = „ „ Centigrade.

R = „ „ Reaumur.

$$F = \frac{9}{5} C + 32$$

$$F = \frac{9}{4} R + 32$$

$$C = \frac{5}{9} (F - 32)$$

$$R = \frac{4}{9} (F - 32)$$

Freezing point, or 32° Fah. = zero in Centigrade or Reaumur.

Boiling point of 212° Fah. = 100 Centigrade and 80 Reaumur.

For barometric heights, measurements are given in millimètres.

For the ready conversion of gaseous volume into weights, the standard proposed by Dr. Hofman, and called by him the crith, is generally used, the student being advised to make himself thoroughly conversant with the use of it (that is, if he has not already done so), as it will be found of very great service in making various calculations, percentage compositions, &c., &c.

The term crith was given by Dr. Hofman from *κριθῆ*, a Greek word, signifying a barley-corn. The crith is the weight of 1 litre (cubic décimètre) of hydrogen at normal temperature and pressure, and it is used as the standard unit of volume. The weight of the crith is  $\cdot 0896$  gramme.

With the exception of arsenic, phosphorus, zinc, cadmium, and mercury, the densities of the elementary gases are the same as their atomic weights; therefore, hydrogen being taken as unity,

1 litre of oxygen weighs 1 crith, or  $\cdot 0896 \times 16 =$   
1.43 gramme,

1 litre of chlorine weighs 35.5 criths, or  $\cdot 0896 \times$   
 $35.5 = 3.19$  grammes,

and so on.

By Ampère's law all gases and vapours contain the same number of molecules in the same space; therefore the densities of all compound gases and vapours are equivalent to half their atomic weights, consequent upon the fact that all molecules occupy the same space as two volumes or atoms of hydrogen (which is one molecule) at the same temperature and pressure; therefore, as only one volume of hydrogen is used to compare

the densities of elementary gases and vapours, so also must only one volume of compound gases and vapours be used. For example:—

Component atomic weights.

1 litre of  $\mathbf{NO}_2\mathbf{Ho}$  weighs  $\frac{14+32+1+16}{2} = \frac{63}{2} = 31.5$  criths, or  
 $31.5 \times .0896 = 2.82$  grammes.

1 litre of  $\mathbf{HCl}$  weighs . .  $\frac{1+35.5}{2} = \frac{36.5}{2} = 18.25$  criths, or  
 $18.25 \times .0896 = 1.63$  grammes.

## NOTES ON GENERAL ANALYSIS.

The method of detection for qualitative analysis consists in bringing the bodies under examination. 1st. Into the gaseous state, and watching the changes which take place when the substances are changed into a gas. 2nd. By bringing the substances under examination in contact with bodies of known properties, and watching the various phenomena which may follow; these phenomena may consist in an alteration of form, combination, or colour, occasioned by a new arrangement of particles, generally depending upon some chemical change or changes. The change of form most frequent is the formation of a precipitate, by chemical action, of a liquid into a solid; for example, if HCl (Hydrochloric acid) be added to a solution of a silver salt, a white precipitate is formed, consisting of AgCl (Argentio-Chloride), this white precipitate is in reality a white powder. All precipitates are solids in a state of powder. Sometimes, however, the phenomenon may consist in effervescence or deflagration. (Effervescence is the disengagement of

gases from any liquid or solid, as when an acid is added to a carbonate and carbonic anhydride is disengaged. Deflagration is the re-arrangement of the crystals of a substance, and is, in ordinary terms, the crackling of a body when exposed to heat.)

The substances with which the body under examination is brought into contact (to produce some known phenomenon) are called *Reagents* or *Tests*, and the change produced by their use is called the reaction of the test. On the purity of the reagents depends much of the success of practical analysis. The methods of preparing the tests and proving their purity are given on pages 37 and 119. These tests, which are acids, bases, salts, and simple elements, when used to distinguish any separate substance (such as we require for manganese), are called *special tests*, and the resulting phenomena are characteristic of the elements for which the different *special tests* are used. Reagents which are employed for dividing a substance or mixture into groups or divisions, such as **SHAm**, Hydric Ammonic Sulphide, or **SH<sub>2</sub>**, Sulphuretted Hydrogen, are called *Group Reagents*.

In going through an analysis of a substance it has to be proved not only that certain elements are present, but that all the other elements are absent; for this reason we cannot adopt the method of indiscriminately adding reagents or tests to the mixture, but must go through a complete course of rules as laid down in this work; that is, it requires not only that the reagents be added in a certain order, but that the substances (sought for) be separated by the *group reagents* into families or divisions, and afterwards each group divided and subdivided, until the absence or presence of every element is satisfactorily proved by the resulting phenomena. The separation into groups is effected by reason of the same or similar change being common to two or more elements, these elements would then be said to belong to the same group, or division. The student should always, on adding any reagent, pause and consider why such a substance is applied, and what is the chemical or physical change he expects, and what might prevent its appearance. The analyst is in fact a judge before whom all the circumstances are laid bare, and on whose judgment depends the decision; he must

also remember that hardly any amount of reading or lecture-hearing can produce a practical analyst, as only *practice* can make perfect, and therefore the student is strongly recommended to make the experiments *himself*.

The first step is to make a preliminary examination of the substance, as per page 57, and every reaction should be carefully noted down, although the student may not find any direct reference to it in the tables. Mixtures may interfere, and sometimes entirely prevent some of the changes described under the preliminary examination, but it should always precede the systematic. The WET WAY examination, performed with *care*, indicates most if not all the elements present. Before proceeding to the wet way examination the student should try the solubility of the substance, as per page 60. By studying the table of solubilities much information can be obtained with regard to the elements present; thus, all the salts of sodium are soluble in water; if, therefore, we have nothing soluble in water, sodium is absent; or upon adding water, a ppt. is formed, Bismuth is indicated. If we require to fuse the substance,

we know that ( $\text{SiO}_2$ ), Silicic Anhydride is present.

The first table is then gone through, each division being laid on one side as finished, until the whole have been completed, after which the divisions A, B, and C, are commenced, and the individual elements distinguished; it will at first be desirable that the student should thoroughly study the first table, so that, on a substance being given him, he can at once tell to which group or groups the component elements belong. When he can perform this expeditiously, he may proceed to the separation of the various members of the several groups, and, in fact, undertake the complete course of qualitative analysis.

As it is often necessary to ignite or fuse substances, the following facts must be attended to. Platinum crucibles or capsules are generally employed to ignite substances in, because of their infusibility, pliability, and the resistance they offer generally to the action of many powerful elements when in a *nascent condition* (that is, just liberated).

There are, however, many operations in which these expensive utensils might be injured, or even

totally destroyed, and therefore attention to the following directions (given by Berzelius) should be attended to, namely :—

In the dry way :—

Never melt in a platinum crucible,

1. Caustic alkalies or alkaline nitrates ; consequently,

No saltpetre ( $\text{NO}_2\text{Ko}$ ), caustic potash ( $\text{KHo}$ ), or soda ( $\text{CONao}_2$ ). Because in such a case, protoxide of platinum would be formed, of course at the expense of the platinum. The protoxide of platinum dissolves in the alkali, and gives it a green colour.

2. No *alkaline sulphurets*.

Nor *sulphates* mixed with *charcoal* powder ;

Because alkaline sulphurets act upon platinum with still greater energy than caustic alkalies.

3. No *metallic substances* ;

Nor any mixture which may yield a metallic residue. *Gold, silver, and copper* may, however, be heated red-hot in platinum crucibles without risk, provided the heat be not too near the point at which those metals fuse ; but melted lead cannot be poured, even in a cold platinum crucible, without spoiling it.

A drop of lead, tin, or bismuth falling upon a red-hot platinum vessel, invariably makes a hole in it.

4. No *phosphorus*, or *phosphoric acid*, mixed with combustible substances; because they would form *phosphuret of platinum*, in consequence of which the platinum would be melted at that point, or would become shrivelled up.

5. Certain *metallic oxides* must not be heated to a white heat in platinum vessels;

Because it often happens that at such temperatures they part with their oxygen, in consequence of which their oxygen, whilst escaping, leaves the metal in a *nascent* condition, and then combines with the platinum as an alloy. The metallic oxides, which have the most tendency to become thus reduced, are the oxides of lead, tin, bismuth, copper, and nickel. If, however, the heat be moderate, they undergo no change.

6. Platinum combines easily with silica and carbon, so that the contact of platinum crucibles with charcoal, at a very high temperature, must be avoided.

In the wet way:—

Platinum must never be used with substances

capable of disengaging chlorine ; wherefore, *Aqua Regia*, even very dilute, must not be put in platinum crucibles ;

Nor must any substance containing *Manganese* lie dissolved with *Hydrochloric Acid* (HCl), because *Chlorine* would be evolved, which will act upon the platinum.

When, for example, the mineral substance to be analysed and calcined with an alkaline carbonate is green or black, it ought to be mixed with hydrochloric acid in a glass vessel, and evaporated until all odour of chlorine has disappeared. In general, all those substances may be boiled or evaporated in platinum crucibles, which do not contain either chlorine or bromine.

In filtering, the great requirement is rapidity, and to effect this, it is best to have the operation performed in an atmosphere of steam ; to accomplish this the filtering should be conducted over a vessel of hot water, so that the steam may always be present, or we may use an apparatus similar to fig. 1, in which A represents a large glass beaker, from which the bottom has been removed, B, the filtering paper, and C, the common Florence

flask, half full of water, in which steam is generated; a small support receives the beaker into which the filtered liquid falls from the paper

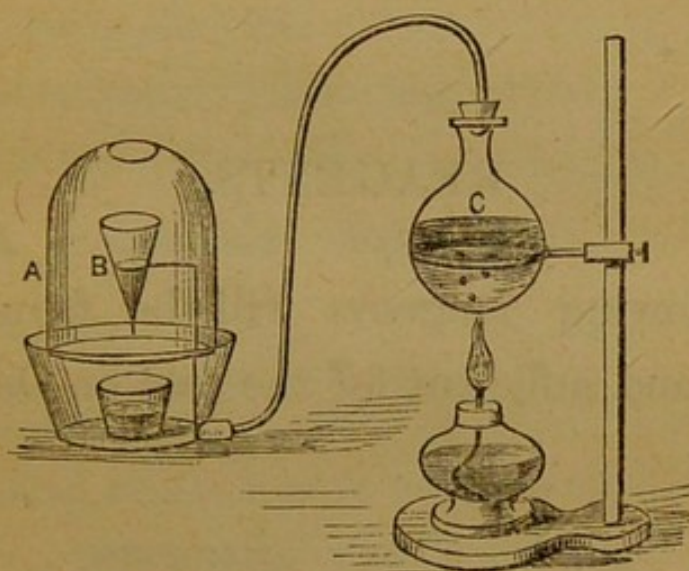


Fig. 1.

filter held by the ring support. The steam from the flask passes into A, and soon fills up the whole apparatus, escaping through the bottomless inverted beaker. By this, or similar contrivances, not only is the filtering proceeded with rapidly, but, whilst taking place, the precipitate, being in an atmosphere of steam, is sheltered from the action of the air.

## REAGENTS.

THE following reagents will be found to be required and sufficient for the part on Qualitative Analysis :

### SOLID OR DRY REAGENTS,

In large quantities.

**CO** $\text{NaO}_2$ , sodic carbonate (soda, carbonate of soda).

**NO** $_2$ **Ko**, potassic nitrate (saltpetre).

**AmCl**, ammoniac chloride.

**CO** $\text{AmO}_2$ , ammoniac carbonate.

**NaHo**, sodic hydrate (used also in solution).

### FLUID REAGENTS,

In quantities of about 4 oz.

**HCl**, hydrochloric acid.

**SO** $_2$ **Ho** $_2$ , sulphuric acid.

**NO** $_2$ **Ho**, nitric acid.

**NaHo**, sodic hydrate.

**CaHo** $_2$ , calcic hydrate, in solution.

- BaHo<sub>2</sub>**, baric hydrate.  
**COAmo<sub>2</sub>**, ammonic carbonate.  
**AmCl**, ammonic chloride.  
**SHAm**, hydric-ammonic sulphide.  
**BaCl<sub>2</sub>**, baric chloride.  
**AmHo**, ammonic hydrate (ammonia).  
**CONao<sub>2</sub>**, sodic carbonate.  
 { **CH<sub>3</sub>**,  
   **COHo**, acetic acid (vinegar pure).  
 { **C(Me)O**,  
   **Pbo''**, plumbic acetate.  
   **C(Me)O**,  
 Methylated spirits free from gum.  
**K<sub>4</sub>FeCy<sub>6</sub>**, potassic ferrocyanide.  
**POHoNao<sub>2</sub>**, hydric disodic phosphate.  
**Fe<sub>2</sub>Cl<sub>6</sub>**, ferric chloride.  
 { **COAmo**,  
   **COAmo**, ammonic oxalate.

In bottles of 1 oz. capacity.

- { **NO<sub>2</sub>**,  
   **NO<sub>2</sub>**, **Coo''**, cobaltous nitrate (pure).  
**PtCl<sub>4</sub>**, platinic chloride.  
**NO<sub>2</sub>Ago**, argentic nitrate.

REAGENTS USED AS SPECIAL TESTS.

- SNa<sub>2</sub>**, sodic sulphide.  
**H<sub>2</sub>SiF<sub>6</sub>**, hydrofluosilicic acid.  
**CaCl<sub>2</sub>**, calcic chloride.  
**SO<sub>2</sub>Cao''**, calcic sulphate.

**Ca(OCl)Cl**, calcic-chloro-hypochlorite (chloride lime).

**Na<sub>2</sub>(OCl)Cl**, sodic-chloro-hypochlorite (chloride of sodium).

**CS<sub>2</sub>**, carbonic disulphide.

**K<sub>6</sub>Fe<sub>2</sub>Cy<sub>12</sub>**, potassic ferricyanide (solution).

**CrO<sub>2</sub>Ko<sub>2</sub>**, potassic chromate ,,

**SO<sub>2</sub>Mgo''**, magnesian sulphate ,,

**COBao''**, baric carbonate ,,

**KI**, potassic iodide ,,

**HgCl<sub>2</sub>**, mercuric chloride.

**AuCl<sub>3</sub>**, auric chloride.

**KCy**, potassic cyanide.

**SnCl<sub>2</sub>**, stannous chloride.

**KCsy**, potassic sulphocyanide (solution).

{ **COHo**,  
**CHHo**, tartaric acid (cream of tartar).  
**CHHo**,  
**COKo**,

{ **C(Me)O**,  
**NaO<sub>2</sub>**, sodic acetate (acetate of soda).  
**C(Me)O**,

**SO<sub>2</sub>Feo''**, ferrous sulphate (in solution).

{ **COHo**, oxalic acid.  
**COHo**,

#### DRY REAGENTS USED AS SPECIAL TESTS.

**POHoAmoNaO**, microcosmic salt.

(**COKo<sub>2</sub> + CONao<sub>2</sub> + C**), black flux.

**MnO<sub>2</sub>**, manganic oxide.

Blue and red litmus and tumeric papers.

## TESTING OF REAGENTS.

All the reagents ought to be tested by some experiment to prove their purity, if not bought at an establishment of high standing; and even then it is well to know how to prove the purity of the reagents used. The following test will be found useful:

## HCl.—HYDROCHLORIC ACID.

Hydrochloric acid, if pure, is perfectly colourless, and leaves no residue when evaporated; it is generally contaminated with  $\text{SO}_2\text{Ho}_2$ , As, Fe, and sometimes Cl &  $\text{SO}_2$ ; its specific gravity should be 1.2. If it be diluted with water (distilled), it gives a precipitate, if  $\text{BaCl}$  or  $\text{SO}_2\text{Ho}_2$  be present. If it imparts a blue tint to a solution of KI (potassic iodide), mixed with starch paste, it is contaminated with chlorine. If it is turned yellow on evaporation, or after adding  $\text{AmHo}$  (ammonia) in excess, and then acidified with

{  $\text{CH}_3$  (Acetic acid), gives a blue colour after  
 {  $\text{COHo}$

the addition of a solution of potassic ferrocyanide,

'Fe''' is present; it should be examined for As (arsenic) by Marsh's test, see page 89.

For use, dilute one part of the concentrated acid with two parts of water.

### **SO<sub>2</sub>Ho<sub>2</sub>.**—SULPHURIC ACID.

Sulphuric acid is most commonly contaminated by As, 'Fe''', Pb, Ca, and the oxides of nitrogen. Lead is present to a much greater extent when the acid is dilute, as Pb is scarcely soluble in concentrated acid, the lead is chiefly got from the lead chambers in which the sulphuric acid is made. There ought to be no residue if the acid be evaporated upon platinum, and if there is a cloud produced on adding spirits of wine, **SO<sub>2</sub>** Pbo'' (plumbic sulphate) or **SO<sub>2</sub>Cao''**, calcic sulphate, is present. Pb can be detected by adding HCl, when, if present, it will be precipitated, as in Group A, Table I. It can also be tested for, by **NO<sub>2</sub>Ho**, nitric acid, or by Marsh's test. The following process for the preparation of pure sulphuric acid is recommended by Fresenius: "Take of strong sulphuric acid 1,000 grm., ammoniac sulphate 3 grm., manganic peroxide, in powder, 5 grm.; put the acid into a porcelain dish, add the

ammonic sulphate, and heat till copious fumes of  $\text{SO}_2\text{Ho}_2$  escape; this is done to destroy any oxides of nitrogen. After cooling, add the manganic peroxide, and heat to boiling whilst stirring, in order to convert any arsenious into arsenic acid. When cool, pour off the clear fluid into a retort, about a litre in capacity, and distil. The neck of the retort must reach so far into the receiver that the distillate may drop directly into its body. The receiver should be cooled with water. To prevent the neck of the retort from touching the receiver, some long asbestos may be used. When about 10c.c have been drawn over, change the receiver, and distil off three-fourths of the contents of the retort." This method depends upon the fact that  $\text{SO}_2\text{Ho}_2$ , sulphuric acid, containing As, in the form of arsenic acid, yields an arsenic free distillate.

The dilute acid is prepared by adding about  $3\frac{1}{2}$  parts of water to every one of acid.

#### $\text{NO}_2\text{Ho}$ .—NITRIC ACID.

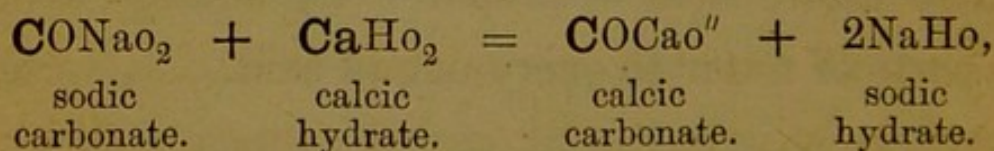
Nitric acid (pure) should leave no residue when evaporated. The impurities most general are  $\text{SO}_2\text{Ho}_2$  and HCl. If, on adding  $\text{NO}_2\text{Ago}$ ,

argentic nitrate, a white precipitate is formed, HCl is present; and if, after diluting with  $\text{OH}_2$  (water), it gives a precipitate with  $\text{BaCl}_2$ ,  $\text{SO}_2\text{Ho}_2$  is present.

For *use*, dilute one part of the concentrated acid with one of water.

#### $\text{NaHo}$ .—SODIC HYDRATE.

Sodic hydrate (caustic soda) should cause no effervescence, or  $\text{SH}_2$  (sulphuretted hydrogen) give a precipitate. The solution should have a sp. gr. of from 1.13 to 1.15, and should be kept in well stoppered bottles. Sodic hydrate can be obtained in sticks; but we can also make it, by the mixing of  $\text{CONaO}_2$  (sodic carbonate), and  $\text{CaHo}_2$  (calcic hydrate), and then boiling the solution. The  $\text{COCaO}''$  is precipitated, and sodic hydrate remains in solution, thus:—



#### $\text{BaHo}_2$ .—BARIC HYDRATE.

Baric oxide mixed with as much water as will dissolve it, should always be mixed fresh,

for, if exposed to the atmosphere, it absorbs carbonic anhydride, which forms a white film of the surface of the solution  $\text{COBa}''$  (baric carbonate).

This must be kept in a well-stoppered bottle. Use a saturated solution.

$\text{COAmo}_2$ .—AMMONIC CARBONATE.

Its solution should give no precipitate or colouration with  $\text{SHAm}$ , hydric ammonic sulphide, or with  $\text{SH}_2$ , sulphuretted hydrogen; nor after acidulating with  $\text{HCl}$  or  $\text{NO}_2\text{Ho}$ ,

(nitric acid); and then adding  $\left\{ \begin{array}{l} \text{NO} \\ \text{Ba}'' \\ \text{NO}_2 \end{array} \right.$ , baric

nitrate, or  $\text{NO}_2\text{Ago}$ , should there be a precipitate.

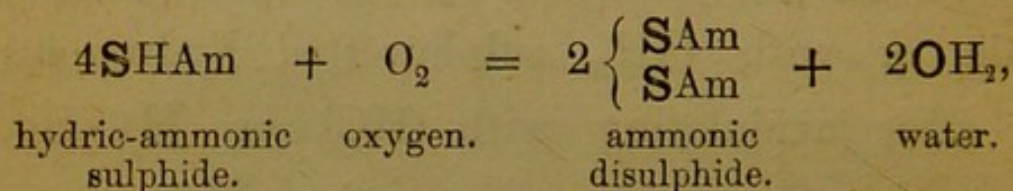
$\text{AmCl}$ .—AMMONIC CHLORIDE.

This salt ought to completely volatilise when ignited on platinum foil, and the solution should be neutral to test papers; there should be no precipitate or colouration upon adding  $\text{SHAm}$ , hydric ammonic sulphide.

Dissolve  $\frac{1}{4}$  oz. in 4 oz. of water for use.

**SHAm.**—HYDRIC-AMMONIC SULPHIDE.

Hydric-ammonic Sulphide is obtained by saturating a solution of ammonia with well washed sulphuretted hydrogen, until it does not produce a precipitate in a solution of  $\text{SO}_2\text{Mgo}''$  (magnesian sulphate). It ought to give no precipitate with lime, and leave no residue on ignition. It should not be used too old, as by exposure to light it becomes yellow, owing to the following decomposition :

**BaCl<sub>2</sub>.**—BARIC CHLORIDE.

The solution of this salt must be neutral to test paper, after precipitation by  $\text{SO}_2\text{Ho}_2$ , sulphuric acid. The filtrate should not have the slightest residue when evaporated on platinum foil.

Dissolve  $\frac{1}{2}$  oz. in 4 oz. of water.

**AmHo.**—AMMONIA.

Ammonia must be colourless, and leave no residue upon evaporation to dryness. No preci-

pitrate should be given when diluted with pure

$\text{NO}_2\text{Ho}$ , and  $\left\{ \begin{array}{l} \text{NO}_2, \\ \text{BaO}'' \text{, baric nitrate, or } \text{NO}_2\text{Ago}, \\ \text{NO}_2, \end{array} \right.$

added. Addition of  $\text{SHAm}$  or  $\text{SH}_2$  ought not to impart the slightest colouration.

Dissolve 1 oz. to 3 oz. of water for use.

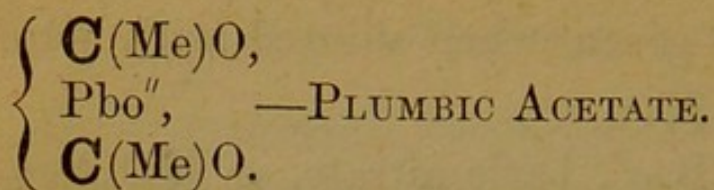
$\text{CONaO}_2$ .—SODIC CARBONATE.

Sodic carbonate must completely volatilise. Its solution should give no precipitate or colouration with  $\text{SH}_2$  or  $\text{SHAm}$ , and ought to behave the same as  $\text{AmHo}$  on treating with the last named reaction for that substance.

$\left\{ \begin{array}{l} \text{CH}_3, \\ \text{COHo}. \end{array} \right.$  —ACETIC ACID.

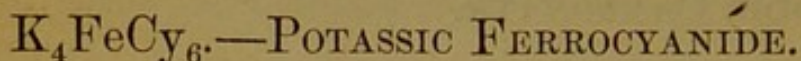
Acetic acid should have no residue on evaporation. After saturation with  $\text{CONaO}_2$ , sodic carbonate, it should give off no empyreumatic odour. It ought to give no colour or turbidness with  $\text{SH}_2$  or  $\text{SHAm}$  after being neutralised by  $\text{AmHo}$ .

The ordinary commercial acid is sufficiently concentrated for analytical purposes.



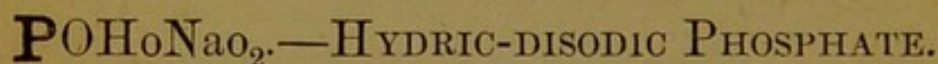
The best commercial acetate is sufficiently pure for use in analytical researches.

Dissolve 1 part, by weight, of the salt in 10 parts, by measure, of water.



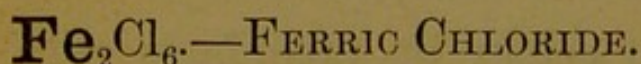
The commercial is sufficiently pure for analytical purposes.

Dissolve 1 drachm in 2 oz. of water.



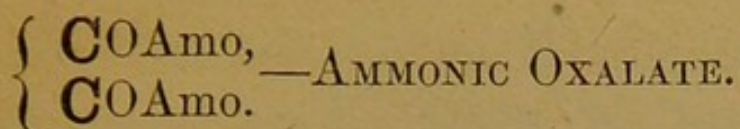
Hydric-disodic phosphate must give no precipitate with ammonia (AmHo).

Dissolve  $\frac{1}{4}$  oz. in 2 oz. of water.



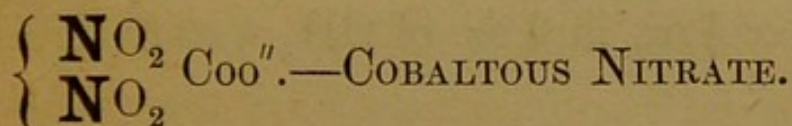
Ferric chloride must be neutral to test paper.

Dilute with an equal volume of water, and filter.



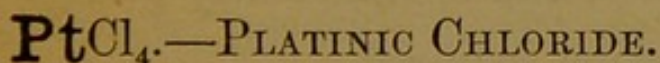
Ammonic oxalate ought to leave no residue after ignition on platinum foil. Upon adding **SH<sub>2</sub>** (sulphuretted hydrogen) or **SHAm** (hydric ammonic sulphide) there ought not to be a precipitate or turbidness.

Dissolve 1 drachm in 2 oz. of water.

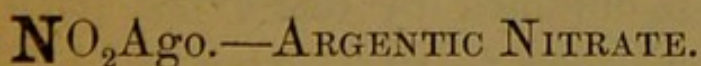


The best commercial is fit for use.

Dissolve  $\frac{1}{4}$  drachm in 1 oz. of water.



Platinic chloride dissolves (if pure) completely in spirits of wine.



Its solution, if pure, will, after the addition of HCl in excess, leave no fixed residue.

Dissolve  $\frac{1}{4}$  drachm in 1 oz. of water.

## SPECIAL REAGENTS.

**S**Na<sub>2</sub>.—SODIC SULPHIDE.

Sodic sulphide is made in Leblanc's method of manufacturing sodic carbonate. The best commercial is pure enough for analytical experiments.

Dissolve  $\frac{1}{2}$  oz. in 2 oz. of OH<sub>2</sub> water.

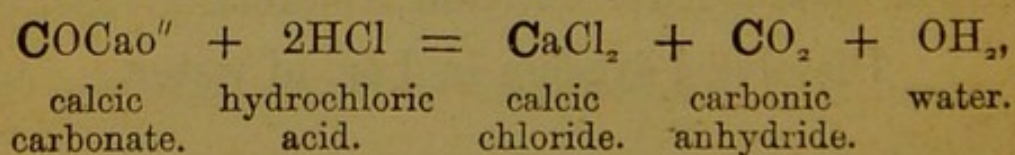
**H<sub>2</sub>SiF<sub>6</sub>**.—HYDROFLUOSILICIC ACID.

This acid can be obtained from the chemists in india-rubber bottles, and is sufficiently pure.

A strong solution required.

**CaCl<sub>2</sub>**.—CALCIC CHLORIDE.

Calcic chloride is obtained by dissolving pure **COCaO''** (calcic carbonate) in dilute HCl (hydrochloric acid). The following is the reaction :



The solution thus obtained must be neutral to test

paper. Upon adding **SHAm** (hydric ammoniac sulphide) there should be no precipitate or colouration.

Dissolve  $\frac{1}{2}$  oz. in 4 oz. of water.

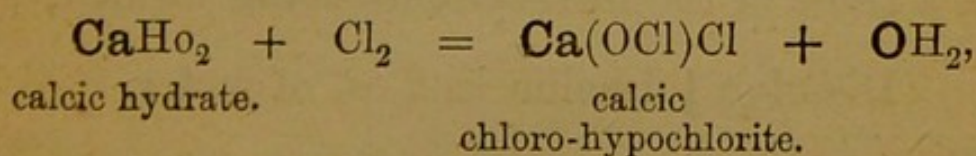
**SO<sub>2</sub>CaO''**.—CALCIC SULPHATE.

Calcic sulphate, or **SO<sub>2</sub>CaO''**, **2OH<sub>2</sub>** (gypsum).

Dissolve as much of the salt as the water will take up.

**Ca(OCl)Cl**.—CALCIC CHLORO-HYPOCHLORITE.

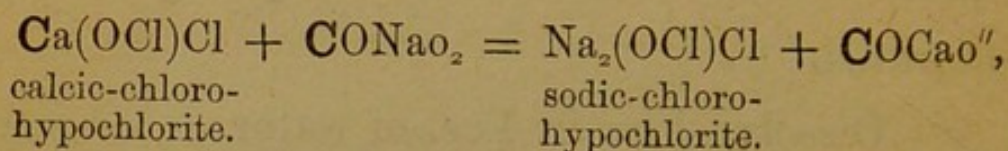
Calcic chloro-hypochlorite (chloride of lime) (bleaching powder). It is made by passing chlorine gas into a low room, on the floor of which is laid a quantity of lime, **CaHO<sub>2</sub>** (calcic hydrate), until no more gas is absorbed.



**Na<sub>2</sub>(OCl)Cl**.—SODIC CHLORO-HYPOCHLORITE.

Sodic chloro-hypochlorite (chloride of soda). To prepare this, precipitate a clear solution of **Ca(OCl)Cl** (bleaching powder) with **CONaO<sub>2</sub>**,

sodic carbonate, and filter the calcic carbonate ( $\text{COCaO}''$ ) off, thus :



**CS<sub>2</sub>.**—CARBONIC DISULPHIDE.

Carbonic disulphide (bisulphide of carbon) is a heavy volatile liquid, boiling at 45° Cent., or 113° Fah., having a specific gravity of 1·2; dissolves gutta percha, caoutchouc, &c.; has a very offensive smell, and must be kept well stopped up.

**K<sub>6</sub>Fe<sub>2</sub>Cy<sub>12</sub>.**—POTASSIC FERRICYANIDE.

Potassic ferricyanide (red prussiate of potash) ought to be made in solution as required, as the ferricyanide decomposes when in a state of solution, a trace of the ferrocyanide being formed.

Dissolve  $\frac{1}{2}$  drachm in 2 oz. of water.

**CrO<sub>2</sub>KO<sub>2</sub>.**—POTASSIC CHROMATE.

Potassic chromate, after acidulating with HCl, the solution ought not to give a precipitate with **BaCl<sub>2</sub>** (baric chloride).

Dissolve  $\frac{1}{2}$  oz. in 4 oz. of water.

**SO<sub>2</sub>Mgo''**.—MAGNESIC SULPHATE.

Magnesian sulphate (Epsom salts). Dissolve one portion in 8 of water.

**COBaO''**.—BARIC CARBONATE.

Baric carbonate may be prepared by dissolving **BaCl<sub>2</sub>** (baric chloride) in hot water, and precipitating with **COAmo<sub>2</sub>** (ammonic carbonate) and **AmHo** (ammonia) then washing the precipitate until the last washings give no turbidness with **NO<sub>2</sub>Ago** (argentic nitrate). Stir the precipitate up with water to the consistence of cream, and keep the mixture in a stoppered bottle, always shaking well before using.

**KI**.—POTASSIC IODIDE.

The commercial is fit for use.

Dissolve  $\frac{1}{4}$  drachm in 1 oz. of water.

**HgCl<sub>2</sub>**.—MERCURIC CHLORIDE.

Dissolve one part in 16 of water.

**AuCl<sub>3</sub>.**—AURIC CHLORIDE.

Auric chloride (if pure) will not give a precipitate with HCl or K<sub>4</sub>Fe<sub>2</sub>Cy<sub>12</sub> when acidified and diluted.

**KCy.**—POTASSIC CYANIDE.

The commercial is fit for use.

Dissolve 1 drachm in 2 oz. of water.

**SnCl<sub>2</sub>.**—STANNOUS CHLORIDE.

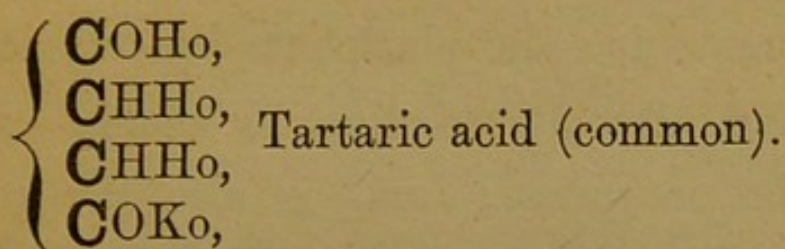
Stannous chloride may be prepared by boiling granulated tin in concentrated HCl in a flask, keeping the tin always in excess until hydrogen has ceased to be evolved, then dilute the solution with four times its volume of water containing a little HCl, and filter. Keep the filtrate in a bottle well stopped, containing some fragment of tin.

**KCsy.**—POTASSIC SULPHOCYANIDE.

Potassic sulphocyanide.

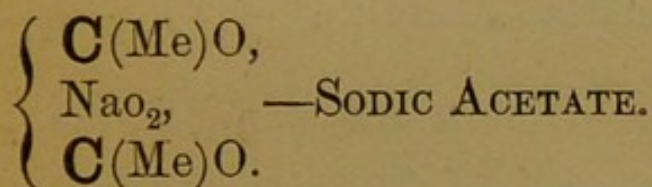
Dissolve one part, by weight, in ten of water.

## TARTARIC ACID.



1 oz. in 4 oz. of water.

## SODIC ACETATE.



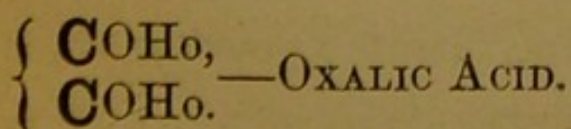
Sodic acetate (acetate of soda) is made by adding acetic acid to a concentrated solution of  $\text{CONaO}_2$  until all effervescence stops.

Dilute one part of the concentrated solution with four of water.

 $\text{SO}_2\text{Feo}''$ .—FERROUS SULPHATE.

The best commercial is sufficiently pure.

Dilute one part with three parts of water.



The commercial is sufficiently pure.

**POHoAmoNao.**—MICROCOSMIC SALT.

Hydric ammoniac sodic phosphate (microcosmic salt) is found, after putrefaction, in human urine and in Ichaboe guano; and is also prepared artificially. Used in the dry state.

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## OTHER TESTS.

REAGENT PAPERS.—Blue litmus paper serves to detect free acid by the paper changing red. Red litmus the presence of free alkalies, by changing blue.

Turmeric paper is changed from yellow to brown by free alkalies.

PREPARATION OF BLUE LITMUS.—“Digest one part of litmus of commerce with six parts of water, and filter the solution; divide the intensely blue filtrate into two equal parts; saturate the free alkali in the one part by repeatedly stirring with a glass rod, dipped in very dilute  $\text{SO}_2\text{Ho}_2$ , until the colour of the fluid just appears red; add now the other part of the blue filtrate; pour the whole fluid into a dish, and draw strips of fine unsized paper through it; suspend these strips over

threads, and leave them to dry. The colour of the litmus paper must be perfectly uniform, and neither too light nor too dark."

PREPARATION OF REDDENED LITMUS PAPER.—  
"Stir blue solution of litmus with a glass rod dipped in dilute  $\text{SO}_2\text{H}_2\text{O}_2$ , and repeat this process until the fluid has just turned distinctly red. Steep slips of paper in the solution, and dry them as directed in the preceding paragraph. The dried slips must look distinctly red."

PREPARATION OF TURMERIC PAPER.—Digest and heat one part of bruised turmeric root with five parts of dilute spirits of wine; filter the tincture obtained, and steep slips of fine paper (similar to that used above) in the solution. The slips, when dry, must exhibit a fine yellow tint.

SOLUTION OF INDIGO.—Add fuming  $\text{SO}_2\text{H}_2\text{O}_2$  to finely pulverised indigo, in small portions at a time, taking care to keep the mixture well stirred. The acid at first gives a brownish tint, from the matter which the indigo contains in admixture, but subsequently turns blue. Heating to any considerable extent partly destroys the blue solution. When sufficient indigo has been added, cover the vessel, and let it stand for two or three

days; then pour its contents into twenty times the quantity of water; mix, filter, and keep the filtrate for use.

DISTILLED WATER ought always to be used in all analytical operations: it should leave no residue on evaporation, nor precipitate or turbidity on the addition of  $\text{BaCl}_2$  (baric chloride),  $\text{NO}_2\text{Ago}$  (argentic nitrate),  $\left\{ \begin{array}{l} \text{COAmo} \\ \text{COAmo} \end{array} \right.$  (ammonic oxalate).

PART I.  
QUALITATIVE ANALYSIS.

THE  
LIBRARY OF THE  
MUSEUM OF NATURAL HISTORY

## QUALITATIVE ANALYSIS.

### SHORT PRELIMINARY EXAMINATION.

[*This preliminary should always precede the systematic Wet-way Examination.*]

REDUCE the substance to a fine powder if given to you as a solid. Evaporate a small portion to dryness, and examine the residue, if given to you as a liquid, and then proceed as follows:—

I.—Place a little of the substance moistened in the HCl, hydrochloric acid (muriatic acid), on a clean platinum wire loop, in the inner blowpipe flame.

- A.** The flame is coloured golden yellow—Presence of . . . . . **Na** (Sodium).  
**B.** The flame is coloured violet; if seen through blue cobalt glass, purple—Presence of . . . . . **K** (Potassium).  
**C.** The flame is coloured green or greenish-blue—Presence of . . . . . **Cu** (Copper), and perhaps **BH<sub>3</sub>** (Boric acid).  
**D.** The flame is coloured greenish-yellow or olive—Presence of . . . . . **Ba** (Barium).

- E.** The flame is coloured intensely crimson—Presence of . . . . . **Sr** (Strontium), or **Li** (Lithium).
- F.** The flame is coloured light-red or orange—Presence of . . . . . **Ca** (Calcium).

II.—Place a small portion of the substance, with a little  $\text{CONaO}_2$ , sodic carbonate (carbonate of soda), in a bulb test tube, and apply heat.

- + **A.** A grey metallic mirror—Presence of **As** (Arsenic).
- B.** Globules of mercury . . . . . **Hg** (Mercury).
- C.** A crystalline sublimate of a whitish colour—Presence of . . . . .  $\left\{ \begin{array}{l} \text{COHo} \\ \text{COHo} \end{array} \right.$  (Oxalic acid).
- D.** Oxygen is given off—Presence of . Nitrate, chlorates, perchlorates, and sometimes peroxides.

III.—Place another portion of the substance in a bulb test tube, and heat in the blowpipe flame.

- A.** Substance swells and then diminishes, and evolution of steam takes place—Presence of . . . . . Salts containing water of crystallization, as phosphates, borates, and alums.
- B.** Reddish-yellow drops in tube—  
Presence of . . . . . A sulphide.
- C.** Evolution of nitrous fumes takes place—  
—Presence of . . . . . Nitrates.
- D.** Substance turns black—Presence of Organic matter.
- E.** Fumes of acetone—Presence of . Acetates.
- F.** A white sublimate—Presence of . Compounds of **Am** (Ammonium), **Hg** (Mercury), and **As** (Arsenic).

- C. Substance does not change—Absence  
of, . . . . . Organic matter, salts  
containing water of  
crystallization, and  
all volatile sub-  
stances.

IV.—Place a portion of the substance on the borax bead, and put it in the outer blowpipe flame.

- A. Bead whilst hot is blue, when cold it is blue  
—Presence of . . . . . **Co** (Cobalt).  
B. Bead whilst hot is green, when cold it is  
green—Presence of . . . . . **Cr**(Chromium).  
C. Bead whilst hot is yellowish-red, when cold  
it fades—Presence of . . . . . **Ni** (Nickel).  
+ D. Bead whilst hot is reddish-brown, when cold  
it is cream colour—Presence of . . . . . **Fe** (Iron).  
E. Bead whilst hot is green, when cold it is blue  
—Presence of . . . . . **Cu** (Copper).

V.—Put a little of the substance in a test tube with HCl, and apply heat.

- A. Reddish-brown fumes of nitrous gas evolved  
—Presence of . . . . . Nitrites or  
Nitrates.  
B. Evolution of Chlorine—Presence of . . . . . Hypochlorites.  
C. Effervescence and evolution of gas, which  
makes lime-water milky—Presence of . Carbonates.  
D. Evolution of  $\text{SH}_2$ , known by its offensive  
smell—Presence of . . . . . Sulphides.  
E. Evolution of Euchlorine, a greenish gas—  
Presence of . . . . . Chlorates.  
F. Evolution of a gas having the smell of burn-  
ing sulphur—Presence of . . . . . Sulphites.

## TABLE OF THE SOLUBILITY OF THE

To understand this Table, look where the oxide column meets resulting salt, and shows

Acids. Bases.	<b>OK<sub>2</sub></b>	<b>ONa<sub>2</sub></b>	<b>OAmo<sub>2</sub></b>	<b>BaO</b>	<b>SrO</b>	<b>CaO</b>	<b>MgO</b>	<b>Al<sub>2</sub>O<sub>3</sub></b>	<b>Cr<sub>2</sub>O<sub>3</sub></b>	<b>ZnO</b>	<b>MnO</b>	<b>NiO</b>	<b>CoO</b>	<b>FeO</b>
<b>F</b>	W	W	W	W	W	W	A	A	A	A	A		A	
<b>SO<sub>2</sub>Ho<sub>2</sub></b>	W	W	W	O	O	W	W	W	W	W	W	W		W
<b>FOHo<sub>3</sub></b>	W	W	W			A	A							A
<b>BHo<sub>3</sub></b>		W				A								
<b>COHo</b> <b>COHo</b>	W	W	W			A								
<b>COHo<sub>2</sub></b>	W	W	W	A	A	A	A			A	A			A
<b>HCy</b>	W													
<b>HCl</b>	W	W	W	W	W	W	W		W	W	W		W	W
<b>HI</b>	W		W											W
<b>HBr</b>	W													
<b>SH<sub>2</sub></b>	W	W	W	W		W				A	A	A	A	A
<b>NO<sub>2</sub>Ho</b>	W	W	W	W	W								W	
<b>OCl</b> <b>OHo</b>	W													
<b>CH<sub>3</sub></b> <b>COHo</b>	W	W	W			W		W		W				W

W stands for soluble in water ; A, soluble in acids ; O, insoluble in acids ; **W** sparingly soluble in water. Line F shows the

PRINCIPAL OXIDES, ACIDS, AND SALTS.

the acid line. The square forming the junction represents the its solubility, as follows:—

	$\text{Fe}_2\text{O}_3$	$\text{O}^{\text{A}}\text{As}_2$	$\text{PbO}$	$\text{Hg}_2\text{O}$	$\text{HgO}$	$\text{CuO}$	$\text{BiO}_3$	$\text{CdO}$	$\text{AuO}_3$	$\text{PtO}_2$	$\text{SbO}_3$	$\text{SnO}$	$\text{SnO}_2$	$\text{As}_2\text{O}_3$	$\text{As}_2\text{O}_5$
	A	A	A	A	A	A	A				A		A	W	W
	W	W	A	W	W	W									
	A	A	A			A									
			A			A									
		A		A											
	W	O	W	A	W	W		W	W	W	W	W	W	W	
		O	W		A			W							
									W						
		A	A		A	A		A			A			A	A
	W	W	W	W	W	W	W								
	W		W			W									

in water and acids; **A** insoluble in water, and sparingly soluble solubility of the bases. The acids are all soluble in water.

QUALITATIVE ANALYSIS OF A SUBSTANCE WHICH  
MAY CONTAIN ONE OR ALL THE BASES AND  
INORGANIC ACIDS.

*Examination of the solubility of the substance, and  
preparation of the solution for analysis.*

Boil the substance with water, HCl, hydrochloric acid (muriatic acid),  $\text{NO}_2\text{Ho}$ , nitric acid (aqua fortis), and *aqua regia* successively, in this order, until the proper solvent is found; pour or filter off the fluid obtained with each solvent from the residue, and evaporate a few drops on a watch-glass, to see if any of the original substance has been dissolved by that solvent. Examine the fluid filtered off from each solvent where it is found to have *dissolved* some portion of the substance under examination, by tables which will be found on the following pages. If there is still a residue after boiling with all the solvents, it must be fused upon platinum foil, with five times its weight of fusion mixture. The fused mass must then be boiled with water and filtered. Now examine residue (after dissolving it as above) for the base, and the filtrate for the acid.

How would you perform the Qualitative analysis of an alloy containing arsenic, copper, silver lead and iron?

Dissolve in  $\text{HNO}_3$  and add  
 $\text{HCl}$  then  $\text{AgCl}$ ,  $\text{PbCl}_2$  } ppt

filter and to solution  
digest with  $\left\{ \begin{array}{l} \text{CuS} \\ \text{As}_2\text{S}_3 \\ \text{PbS} \end{array} \right\}$  on adding  
 $\text{SH}_2$

digest the  $\text{CuS}$  &  $\text{PbS}$  with  $\text{SO}_2$  &  $\text{H}_2\text{O}_3$

TABLE No. 1.

General Table for the examination of a substance which may contain one or more metals.  
 Determination of the group to which the metal or metals belong. Never start with an alkaline solution  
 (see note 1).

GROUP A.	GROUP B.	GROUP C.	GROUP D.	GROUP E.	GROUP F.
<p><i>HCl</i></p> <p>To a portion of prepared solution add <i>HCl</i> in excess.</p> <p>A precipitate is obtained which may contain one or all the following chlorides:</p> <p><math>\text{Hg}_2\text{Cl}_2</math> } A white  <math>\text{PbCl}_2</math> } ppt.  <math>\text{AgCl}</math> }</p> <p>Shake test tube vigorously, and filter. Examine ppt. by Table 2, and filtrate by column B, after it has been diluted with water (see note 2A).</p> <p>No ppt. Dilute the solution with</p>	<p><i>Copper</i></p> <p>To filtrate or solution from column A, add strong solution of <math>\text{SH}_2</math>, and gently warm (see note 2).</p> <p>A ppt. is obtained which may contain one or all the following sulphides (see note 3):—</p> <p>Subdivsn. 1 } <math>\text{HgS}</math> } A black ppt.  <math>\text{PbS}</math> } }  <math>\text{Bi}_2\text{S}_3</math> } }  <math>\text{CuS}</math> } }    Subdivsn. 2 } <math>\text{CdS}</math> } A yellow ppt.  <math>\text{SnS}</math> } }  <math>\text{SnS}_2</math> } }  <math>\text{As}_2\text{S}_3</math> } }  <math>\text{As}_2\text{S}_5</math> } }    A yellow ppt.</p>	<p>To filtrate or solution from B, add <i>AmCl</i> and <i>AmHo</i> in excess.</p> <p>A ppt. is obtained which may contain one or all the following hydrates:—</p> <p><math>\text{Cr}_2\text{H}_6</math> } A dirty coloured ppt.  <math>\text{Al}_2\text{H}_6</math> } A white ppt.  <math>\text{Fe}_2\text{H}_6</math> } reddish brown ppt.</p> <p>Wash and filter off the ppt. Examine ppt. by</p>	<p>To filtrate or solution from C, add <i>SHAm</i>, and gently warm.</p> <p>A ppt. is obtained which may contain one or all the following sulphides:—</p> <p><math>\text{FeS}</math> } A black ppt.  <math>\text{CoS}</math> } " "  <math>\text{NiS}</math> } " "  <math>\text{ZnS}</math> } A white ppt.  <math>\text{MnS}</math> } A flesh-colour ppt.</p> <p>Wash and filter off the ppt. Examine ppt. by Table 6, and filtrate by column E (see note 6).</p>	<p>To filtrate or solution from column D add, <i>AmCl</i>, <i>AmHo</i> in excess, and <math>\text{COAmO}_2</math>.</p> <p>A ppt. is obtained which may contain one or all the following carbonates:—</p> <p><math>\text{COBao}</math> } A white ppt.  <math>\text{COSro}</math> } " "  <math>\text{COCao}</math> } " "</p> <p>Wash and filter ppt. Examine it by Table 7.</p> <p>Add to filtrate (see note 7)</p>	<p>The filtrate or solution from column E may contain <i>Na</i>, <i>K</i>, <i>Am</i>.</p> <p>Examine the solution by Table 8.</p> <p><i>Magnesium</i></p>

<p>water, and examine by column B.</p>	<p>Subst. { <math>Sb_2S_3</math> } { An orange ppt. }          { <math>As_2S_3</math> } { A black ppt. }          { <math>PtS_2</math> } { }</p> <p>Filter off and well wash ppt.; then examine it by Tables 3 and 4.</p>	<p>Boil the filtrate till free from <math>SH_2</math> (see note 4), and add a few drops of <math>NO_2Ho</math>. Evaporate to dryness (see note 5). Moisten residue with <math>HCl</math>, add water, and apply heat. If insoluble white flakes remain, presence of <math>SiO_2</math>. Filter off, and examine filtrate by column C.</p>	<p>No ppt. Treat solution same as directed for filtrate, and examine by column C.</p>	<p>Table 5, and filtrate by column D.          No ppt. Examine solution by column D.</p>	<p>No ppt. Examine solution by column E.</p>	<p><math>POHoNaO_2</math> is a white ppt. which is <math>POAmoMgo</math> (see note 8). Gently heat (see note 9). Wash and filter ppt.; then examine it by Table 7, and filtrate by column F.          No ppt. Examine solution by column F.</p>
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The notes referred to will be found upon page 74.

TABLE No. 2.

Examination of ppt. produced by HCl, Group A.  
After the HCl ppt. has been shaken up in test tube and filtered off, add water and boil.

If there is still a residue, it must be filtered off and examined separately.		
To filtrate or solution add $\text{CrO}_2\text{K}_2\text{O}_2$ .  A yellow ppt. is obtained,  LEAD. Confirm by reaction No. 9.*  <i>Heat with <math>\text{CONaO}</math> on G. yield beads of Lead</i>	If there is still a residue, add AmHo, and if this does not dissolve it all, then filter off residue.	
	To filtrate or solution add $\text{NO}_2\text{Ho}$ in excess.  A white ppt. is obtained,  SILVER. Confirm by reaction No. 15.	If the residue turns black after adding AmHo,  MERCURY. (See note 10.) Confirm by reaction No. 3. <i>Heat with <math>\text{CONaO}</math> grey sublimate of Mercury</i>

TABLE No. 3.

Examination of the ppt. produced by  $\text{SH}_2$ , Group B.  
Gold and platinum must be specially tested for in the original solution, by reactions Nos. 47, 48, and 50.

The well-washed ppt. is transferred to a flask, and boiled with NaHo for a few minutes, and the residue (if any) must be filtered off, and examined by Table 4.			
<i>Subdivision 2.</i> —As, Sn, and Sb (Au and Pt test for as above). Treat the filtrate or solution with HCl in excess, $\text{SH}_2$ being added.			
A yellow ppt. is obtained,  ARSENIC. Confirm by reaction No. 40.	Divide filtrate from As ppt., or solution treated as above, into three parts, and proceed as follows:—		
	1st Portion.—Add $\text{HgCl}_2$ .  A white ppt.  TIN. (See note 11.) Confirm by reaction No. 33.	2nd Portion.—Add $\text{SO}_2\text{Ho}_2$ .  A nearly white ppt.,  TIN. (See note 12.) Confirm by reaction No. 35.	3rd Portion (Marsh's test).— A black mirror upon porcelain.  ANTIMONY. Confirm Mirror not dissolved by a drop of $\text{Na}_2(\text{OCl})\text{Cl}$ . See reaction No. 45.

\* Reactions referred to will be found upon pages 78, 79, &c.

TABLE No. 4.

Examination of the residue from Table 3, Group B, insoluble in NaHo.

<i>Subdivision 1.</i> —Hg (see note 13), Pb, Bi, Cd, and Cu. Wash the residue till free from alkali, and then boil it in a little strong $\text{NO}_2\text{Ho}$ .				
A black residue remains,  <b>MERCURY.</b>  Confirm by reaction No. 18.	To filtrate from Hg residue, or solution treated as above, add $\text{SO}_2\text{Ho}_2$ .			
	A white ppt.,  <b>LEAD.</b>  Dissolve ppt. in ammoniac acetate, and confirm by reaction No. 8.	To filtrate from Pb ppt., or solution treated as above, add <u>AmHo</u> in excess, and boil.		
		A yellow ppt.,  <b>BISMUTH.</b>  Dissolve ppt. in $\text{NO}_2\text{Ho}$ , evaporate to dryness, then confirm by adding water, reaction No. 22.	To filtrate from Bi ppt., or solution treated as above, add $\text{COAmo}_2$ in excess.	
			A straw-coloured ppt.,  <b>CADMIUM.</b>  Confirm by reaction No. 31.	If a blue solution remains, it indicates  <b>COPPER.</b>  Confirm by reaction No. 24.

TABLE No. 5.

Examination of ppt. produced by AmHo in excess, AmCl being present, Group C.

To ppt. add NaHo in excess.		
A light-coloured residue, which turns green and then reddish-brown—  <b>IRON.</b>  (See note 14.) Confirm by reactions Nos. 59 and 60.	Boil filtrate from Fe ppt., or solution treated as above, in a test tube for some time.	
	A dirty-green ppt.,  <b>CHROMIUM.</b>  Confirm by reaction No. 52.	To filtrate from Cr ppt., or solution treated as above, add AmCl.
		A white ppt.,  <b>ALUMINIUM.</b>  Confirm by reaction No. 57.

TABLE No. 6.

$\text{POHo}_3$  is absent. (See note 16.)

Examination of ppt. produced by  $\text{SHAm}$ , Group D.

<p>Add <math>\begin{matrix} \text{CH}_3 \\ \text{COHo} \end{matrix}</math> to ppt.</p> <p>Should there still be a residue, add <math>\text{NO}_2\text{Ho} + \text{HCl}</math>, which will dissolve the remaining residue. Evaporate almost to dryness, then add a concentrated solution of <math>\text{KCy}</math>, after which well boil, and add <math>\text{SO}_2\text{Ho}_2</math>.</p>			
<p>A greenish ppt., which crystallizes after the lapse of time,</p> <p>NICKEL.</p> <p>Confirm by reaction No. 75.</p>	<p>To filtrate from Ni ppt., or solution treated as above, add <math>\text{SHAm}</math>.</p>		
	<p>A black ppt.,</p> <p>COBALT.</p> <p>Confirm by reaction No. 70.</p>	<p>To filtrate from Co ppt., or solution treated as above, add <math>\text{COAmo}_2</math> in excess.</p>	
		<p>A ppt. is obtained which may contain Fe and Mn. Add <math>\text{AmCl}</math>.</p>	
	<p>A white ppt.</p> <p>MANGANESE.</p> <p>Confirm by reaction No. 78.</p>	<p>To filtrate from Mn ppt., or solution treated as above, add <math>\text{K}_4\text{FeCy}_6</math>.</p>	
		<p>A dark blue ppt.</p> <p>IRON.</p> <p>(See note 15.) Confirm by reaction No. 65.</p>	<p>To filtrate from Fe ppt., or solution treated as above, add <math>\text{SHAm}</math>.</p>
<p>A white ppt.,</p> <p>ZINC.</p> <p>Confirm by reaction No. 77.</p>			

TABLE No. 7.

Examination of the ppt. produced by reagents, Group E.

Dissolve ppt. in $\begin{cases} \text{CH}_3 \\ \text{COHo,} \end{cases}$ and divide solution into three parts, and proceed as follows:— To solution No. 1 add $\text{SO}_2\text{Cao}''$ in excess.			
A white ppt. immediately,  <b>BARIIUM.</b> Confirm by reaction No. 82.	If a white ppt. has been obtained immediately after adding $\text{SO}_2\text{Cao}''$ , then filter it off <i>at once</i> .		
	A white ppt. after the lapse of time,  <b>STRONTIUM,</b> Confirm by reaction No. 86.	To solution No. 2 add $\begin{cases} \text{COAmo} \\ \text{COAmo} \end{cases}$	
		A white ppt.,  <b>CALCIUM.</b> Confirm by reaction No. 91.	To filtrate from Ca ppt., or solution treated as above, add $\text{POHoNaO}_2$ .
			A white ppt.,  <b>MAGNESIUM.</b> Confirm by reaction No. 95.

No. 3 solution is reserved for use in case either of the other two get spoilt.

TABLE No. 8.

## Examination of the solution from Group F.

Evaporate solution to dryness, and ignite residue on platinum foil till no more ammoniacal fumes are given off. Dissolve residue in a little $\text{OH}_2$ , and if necessary add a few drops of HCl.		
Take up a drop of the prepared solution upon the loop of a clean platinum wire, and hold in the outer portion of the lower part of the Bunsen, or the inner cone of the blowpipe flame.		
The flame is coloured violet—presence of  POTASSIUM,  and absence of sodium. Confirm by reaction No. 100.	The flame is golden yellow, and appears purple when seen through blue cobalt glass—Presence of both POTASSIUM and SODIUM.  Confirm for K by reaction No. 100.	
	<table border="1"> <tr> <td>The yellow flame cannot be seen through the cobalt glass—presence of  SODIUM,  and absence of potassium.</td> <td>If the presence of Am has not already been determined in the preliminary examination, apply reaction No. 103.</td> </tr> </table>	The yellow flame cannot be seen through the cobalt glass—presence of  SODIUM,  and absence of potassium.
The yellow flame cannot be seen through the cobalt glass—presence of  SODIUM,  and absence of potassium.	If the presence of Am has not already been determined in the preliminary examination, apply reaction No. 103.	

TABLE No. 9.

## Examination for the Inorganic Acids.

It is very difficult to *definitely* classify the acids, but the following Tables will greatly assist the student.

<p><u>A.</u></p> <p>To a neutral solution add <math>\text{BaCl}_2</math>, and cover the end of the test tube with a watch-glass.</p> <p>A reaction takes place. A white ppt. is thrown down,—Indicating Presence of <math>\text{POH}_3</math>, <math>\text{SiHO}_4</math>, <math>\text{BHO}_3</math>, <math>\text{SO}_2\text{HO}_2</math>.</p> <p>Watch-glass becomes corroded—Presence of <math>\text{HF}</math>.</p> <p>A yellow ppt. is thrown down—Indicating Presence of <math>\text{CrO}_2\text{HO}_2</math>.</p> <p>Effervescence takes place—Indicating Presence of <math>\text{COHO}_2</math>.</p> <p>Examine ppts. by Table 10.</p>									
<p><u>B.</u></p> <p>To a neutral solution add <math>\text{NO}_2\text{Ago}</math>.</p> <p>A ppt. is obtained which may contain one or all the following acids :—</p> <table style="margin-left: auto; margin-right: auto;"> <tr> <td>A yellowish-white ppt.—Indicating presence of</td> <td rowspan="4" style="font-size: 2em; vertical-align: middle;">{</td> <td><math>\text{HI}</math>.</td> </tr> <tr> <td>A white ppt.</td> <td><math>\text{HBr}</math>.</td> </tr> <tr> <td>A black ppt.</td> <td><math>\text{HCl}</math>.</td> </tr> <tr> <td></td> <td><math>\text{SH}_2</math>.</td> </tr> </table> <p>Examine ppt. by Table 11.</p>	A yellowish-white ppt.—Indicating presence of	{	$\text{HI}$ .	A white ppt.	$\text{HBr}$ .	A black ppt.	$\text{HCl}$ .		$\text{SH}_2$ .
A yellowish-white ppt.—Indicating presence of	{		$\text{HI}$ .						
A white ppt.			$\text{HBr}$ .						
A black ppt.			$\text{HCl}$ .						
		$\text{SH}_2$ .							
<p><u>C.</u></p> <p>The following acids will remain in neutral solutions after the above group reagents have been added :—</p> <table style="margin-left: auto; margin-right: auto;"> <tr> <td><math>\text{NO}_2\text{Ho}</math>.</td> <td rowspan="2" style="font-size: 2em; vertical-align: middle;">{</td> <td><math>\text{OCl}</math></td> </tr> <tr> <td></td> <td><math>\text{OHo}</math></td> </tr> </table> <p>Examine for them by Table 12.</p>	$\text{NO}_2\text{Ho}$ .	{	$\text{OCl}$		$\text{OHo}$				
$\text{NO}_2\text{Ho}$ .	{		$\text{OCl}$						
		$\text{OHo}$							

The remaining acids will either have been found in testing for the bases in the preliminary, or by specially testing for them, as per general list of reactions given.

TABLE No. 10.

Examination of ppt. from Table No. 9, A.

To ppt. add HCl.				
A residue still remains—presence of	To solution, after filtering, from last column add { $\text{NO}_2\text{Mgo''}$ and AmHo.			
	A white ppt., <b>POHo<sub>3</sub></b> . Confirm by reactions Nos. 109 and 110.	To solution, after filtering, from last column add $\text{CaCl}_2$ .		
A white ppt., insoluble in		A white ppt., soluble in	To solution add	
<b>SO<sub>2</sub>Ho<sub>2</sub></b> . Confirm by reactions Nos. 118 and 119.	{ $\text{CH}_3$ { $\text{COHo}$ Presence of	{ $\text{CH}_3$ { $\text{COHo}$ Presence of	{ $\text{C(Me)O}$ . Pbo''. { $\text{C(Me)O}$ .	
	{ $\text{COHo}$ . { $\text{COHo}$ . Confirm by No. 111.	<b>BHo<sub>3</sub></b> Confirm by No. 112.	A yellow ppt.—presence of <b>CrO<sub>2</sub>Ho<sub>2</sub></b> . Confirm by No. 106.	

HF will be detected in Table 9, A, by the watch-glass being corroded. Confirm by No. 113.

$\text{COHo}_2$  will also be detected in Table 9, A, by the effervescence. Confirm by No. 116.

TABLE No. 11.

Examination of ppt. from Table 9, B.

To ppt. add $\text{AmHo}$ , and filter <i>quickly</i> .			
Add a little $\text{KCy}$ to the residue (after filtering) in a long-necked bulb tube.			
Octahedral crystals volatilize—presence of  <b>HI.</b> Confirm by Nos. 125 and 126.	By holding a small piece of starch-paper over the tube, it will change to a <i>blue</i> colour—presence of  <b>HBr.</b> Confirm by 123 and 124.	To solution after adding $\text{AmHo}$ , add $\text{NO}_2\text{Ago}$ and $\text{NO}_2\text{Ho}$ .	
		A white ppt.,  <b>HCl.</b> Confirm by No. 121.	To solution from last column add { $\text{C(Me)O}$ . $\text{Pbo}''$ . $\text{C(Me)O}$ .  A black ppt.,  <b>SH<sub>2</sub>.</b> Confirm by No. 128.

TABLE No. 12.

Examination for the acids, Table 9, C.

To neutral solution add $\text{SO}_2\text{Ho}_2$ and sulphate of indigo, then boil. The solution is bleached. Add $\text{SO}_2\text{Ho}_2$ and $\text{SO}_2\text{Feo}''$ .	
A brown-coloured ring forms at the top of solution in the test tube—  <b>NO<sub>2</sub>Ho.</b> Confirm by No. 130.	Evaporate solution from last column to dryness, and heat to redness. Dissolve residue in water, and add $\text{NO}_2\text{Ago}$ .  A white ppt. { $\text{OCl}$ Confirm by { $\text{OHo}$ . No. 134.

## IMPORTANT NOTES

*Regarding the Precipitates produced by the Group Reagents, and the Causes which may interfere with the reactions.*

(TABLE 1.)

Before we add HCl to the solution under examination, it is necessary to test the solution as to whether it be acid, neutral, or alkaline. When it is one of the two former, a very few drops of HCl will suffice. If, however, alkaline, it must be made neutral. After the precipitate has been produced, just add a few more drops of HCl, shake the mixture, and filter.

1. If the original solution be alkaline, several of the acids, as well as the sulphides of Subdivision 2, Group B, will be thrown down, HCl will also throw down the oxychlorides of Bi, Sb, and Sn, but these dissolve upon the addition of more HCl.

The precipitation of Ag is prevented under the following conditions:—

When  $\left\{ \begin{array}{l} \text{NO}_2\text{Hgo}'' \\ \text{NO}_2 \end{array} \right.$  is present, although precipitated (that is, changed into the chloride AgCl), it is immediately taken into solution, because AgCl is soluble in a solution of  $\left\{ \begin{array}{l} \text{NO}_2\text{Hgo}'' \\ \text{NO}_2 \end{array} \right.$ , especially if it be hot and concentrated.

If now  $\text{OH}_2$ , water be added, and the solution be allowed to cool, we shall have yellowish-white crystals deposited, which are pure AgCl. To prevent  $\left\{ \begin{array}{l} \text{NO}_2\text{Hgo}'' \\ \text{NO}_2 \end{array} \right.$  from stopping the precipitating of AgCl, ammoniac acetate should be added to the solution, if it be suspected it is present.

As the chlorides of Hg, Pb, and Ag are very heavy, there is no occasion to warm the solution. Indeed it is disadvantageous to do so, as a portion of the  $\text{HgCl}_2$  would be converted into  $\text{Hg}_2\text{Cl}_2$  and the  $\text{PbCl}_2$  be dissolved.

2A. Water is added, because if the solution is very acid, the members of the Group B are not readily precipitated as sulphides, especially Cd.

If, therefore, the addition of water has been overlooked, we may have the Cd thrown down in the next Group C. If, however, there is no acid reaction, Zn, if present, may be precipitated.

2. If only a yellow cloud of sulphur appears, pass on to next column.

3. If arsenic is expected (as arsenic acid), the solution should be previously treated with sulphurous acid.

4.  $\text{SH}_2$  is here expelled, to avoid the precipitation of Ba, Sr, and Ca, as sulphates, owing to the formation of sulphuric acid.

5. Ignite residue on platinum foil if organic matter is present, but not otherwise.

6. Should the filtrate have a brown colour, indicative of dissolved nickelous sulphide, it must be evaporated until the excess of **S**HAm is expelled, then acidified with dilute HCl, the ppt. thrown upon the filter, and examined with the rest previously obtained.

7. Filter off before adding **P**OHoNa $\text{o}_2$ , if there has been a precipitate.

8. If any member of this group be present as a phosphate, group reagent D will throw it down.

9. The solution must not be *boiled*.

10. Mercury as mercurous compound.
11. Tin as a stannous compound.
12. Tin as a stannic compound.
13. Mercury as a mercuric compound.
14. Iron as a ferric compound.
15. Iron as a ferrous compound.
16. If **POHo<sub>3</sub>** be present, expel it by reaction No. 109, throwing it all down and filtering.
17. Always test for the bases before the acids, and by having done so you will have cleared the solution of As and Cr; now add **NO<sub>2</sub>Ho**, and boil to free solution from **SH<sub>2</sub>** and **CO<sub>2</sub>**, and then proceed to test for the acid as per Table No. 9.

## REACTIONS OF THE BASES.

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*The following will be found to be some of the most important Reactions.*

### GROUP A

(TABLE 2.)

Consists of those metals which are precipitated by HCl, hydrochloric acid (muriatic acid), as chlorides.

**Hg**—MERCURY as a mercurous compound.

1. The white ppt. **Hg<sub>2</sub>Cl<sub>2</sub>**, mercurous chloride (subchloride of mercury, calomel), produced by HCl, hydrochloric acid (muriatic acid),\* (see T. 1, Gr. A), turns black when either NaHo, sodic hydrate (caustic soda), AmHo, ammoniac hydrate (solution of ammonia), or KHO, potassic hydrate (caustic potash), is added in excess, owing to the formation of **NH<sub>2</sub>Hg<sub>2</sub>Cl**.

\* T. is used instead of Table, and Gr. instead of Group.

2. When a small piece of zinc, upon which is placed a piece of gold leaf, is put into a vessel containing a mercurial solution, a white stain will be observed on the gold leaf, owing to a portion of the gold having amalgamated with some of the mercury contained in the solution. Upon application of heat, the above stain will disappear.

3. Compounds of mercury, heated with **CONao**", sodic carbonate (soda, carbonate of soda), in a tube closed at one end, yield a grey sublimate of metallic mercury, which collects in the cool parts of the tube.

4. Copper introduced into a solution of a mercurous or a mercuric salt, especially after the addition of **HCl**, becomes covered with a white lustrous coating; when moderately heated, the copper regains its original colour, vapours of mercury being given off. Slips of copper foil or wire, which have been previously dipped in **NO<sub>2</sub>Ho** and then thoroughly washed, are placed in a boiling solution of mercury for three or four minutes, which should be slightly acidulated with **HCl**. If, when the copper is coated with mercury, it be placed in a covered tube, and the mercury be

allowed to vapourize, a coating of *minute* globules of Hg will be found upon the cool part of the tube. If then the copper be removed, a small particle of iodine introduced, and the tube gently heated, the sublimate of mercury will turn yellow, and when rubbed with a glass rod, scarlet, owing to the formation of **HgI<sub>2</sub>**, mercuric iodide, which is used in medicine, and in the preparation of *Nessler's test* for ammonia.

### **Pb.**—LEAD.

5. The white ppt. **PbCl<sub>2</sub>**, plumbic chloride (chloride of lead), produced by HCl, hydrochloric acid (muriatic acid) (see T. 1, Gr. A), remains unchanged in colour after the addition of AmHo, ammoniac hydrate (solution of ammonia), owing to the formation of the white insoluble **PbO**, **PbCl<sub>2</sub>**, plumbic oxychloride (oxychloride of lead). The above ppt. **PbCl<sub>2</sub>** is soluble in large excess of hot water and **NO<sub>2</sub>Ho**.

6. Weak **SO<sub>2</sub>Ho<sub>2</sub>**, sulphuric acid (vitriol), and sulphates, give the white ppt. **SO<sub>2</sub>Pbo''**, plumbic sulphate (sulphate of lead), at once in strong solutions, but only after the lapse of time in weak solutions of lead. **SO<sub>2</sub>Pbo''** is insoluble in

water and dilute acids, but freely soluble in ammoniac acetate and tartrate.

7.  $\text{KI}$ , potassic iodide (iodide of potass), gives a yellow ppt.  $\text{PbI}_2$ , plumbic iodide (iodide of lead).

8.  $\text{CrO}_2\text{Ko}_2$ , potassic chromate (chromate of potash), gives a nice yellow ppt.  $\text{CrO}_2\text{Pbo}''$ , plumbic chromate (chrome yellow, chromate of lead), soluble in  $\text{NaHo}$ , sodic hydrate (caustic soda), and but very slightly soluble in  $\text{NO}_2\text{Ho}$ , nitric acid (aquafortis).

9. Compounds of lead heated with  $\text{CONao}_2$ , sodic carbonate (soda, carbonate of soda), upon charcoal, in the blowpipe flame, yield malleable metallic beads of lead, which make a black mark when rubbed over white paper.

10. The soluble salts redden litmus.

11.  $\text{PbCl}_2$  is *very* slightly soluble in cold water, but soluble in boiling water.

12.  $\text{AmHo}$  throws down, from solutions of plumbic salts, a white basic precipitate, which is not soluble in an excess of the reagent.

13.  $\text{COAmo}_2$  gives, in solutions of lead, the precipitate  $\text{COPbo}''$ , which is insoluble in an excess of the reagent.

**Ag.**—SILVER.

14. The white ppt.  $\text{AgCl}$ , argentic chloride (chloride of silver), produced by  $\text{HCl}$ , hydrochloric acid (muriatic acid) (see T. 1, Gr. A), is soluble in  $\text{AmHo}$ , ammoniac hydrate (solution of ammonia), but insoluble in  $\text{NO}_2\text{Ho}$ , nitric acid (aqua fortis).

15. Compounds of silver, heated with  $\text{CONaO}_2$ , sodic carbonate (soda, carbonate of soda), upon charcoal, in the blowpipe flame, yield shining white malleable metallic beads of silver.

16. "If silver occurs only in traces in slags or complex ores, it can only be detected by the well-known method of cupellation. If, however, the silver compound is not mixed with a very large amount of foreign matter, it can be detected in very minute quantities, by reduction with soda on the charcoal splinter. The white ductile beads dissolve easily on warming in dilute  $\text{NO}_2\text{Ho}$ , and yield  $\text{AgCl}$  with  $\text{HCl}$ , which can then readily be recognised by its behaviour with  $\text{NO}_2\text{Ho}$  and  $\text{AmHo}$ . Less than one-tenth of a milligramme of silver can thus be easily detected with certainty."—*Bunsen*.

## GROUP B

(TABLE 4.)

Consists of those metals which are precipitated by **SH<sub>2</sub>**, sulphuretted hydrogen (hydrosulphuric acid), as sulphides, after separating Group A.

SUBDIVISION 1 consists of those metals whose sulphides are insoluble in alkaline hydrates and sulphides.

**Hg''**.—MERCURY as a mercuric compound.

17. Solution of **SH<sub>2</sub>**, sulphuretted hydrogen (hydrosulphuric acid), added a few drops at a time, gives first a light-coloured ppt., which then changes to a yellowish-red, and lastly into the black ppt. **HgS**, mercuric sulphide (protosulphide of mercury, vermilion) (see T. 1, Gr. B), which is insoluble in HCl, hydrochloric acid (muriatic acid).

18. **SnCl<sub>2</sub>**, stannous chloride (protochloride of tin), added a few drops at a time, gives first the white ppt. **Hg<sub>2</sub>Cl<sub>2</sub>**, mercurous chloride (calomel, subchloride of mercury), and if enough

**SnCl<sub>2</sub>** is added, it will turn grey, owing to its being reduced to metallic mercury, which can be united into globules by agitation and heat; but it is most easily accomplished by boiling the metallic deposit, after decantation of the supernatant fluid, with HCl.

19. Reactions Nos. 2 and 3 will also take place with mercuric as well as mercurous compounds.

**Pb.**—LEAD.

20. Refer to reactions Nos. 6 to 13.

**Bi.**—BISMUTH.

21. **CrO<sub>2</sub>Ko<sub>2</sub>**, potassic chromate (chromate of potash), gives a finely divided yellow ppt. insoluble in NaHo, sodic hydrate (caustic soda), but it is soluble in **NO<sub>2</sub>Ho**, nitric acid (aqua fortis).

22. AmCl, ammoniac chloride (sal ammoniac), added to solutions of bismuth, diluted with large quantities of water, gives a white ppt. **BiOCl**, a basic chloride insoluble in tartaric acid.

23. Compounds of bismuth, heated with **CONao<sub>2</sub>**, sodic carbonate (soda, carbonate of soda), upon charcoal, in the blowpipe flame, yield brittle metallic beads of bismuth.

**Cu.**—COPPER.

24.  $K_4FeCy_6$ , potassic ferrocyanide (ferrocyanide of potass), gives a chocolate-coloured ppt.

$Cu_2FeCy_6$ , cupric ferrocyanide (ferrocyanide of copper), insoluble in HCl, hydrochloric acid (muriatic acid).

25.  $KHo$ , potassic hydrate (caustic potash), in excess gives a light blue ppt.  $CuHo_2$ , cupric hydrate (hydrated oxide of copper), which turns black if the solution is boiled, owing to the formation of  $CuO$ , cupric oxide (black oxide of copper, protoxide of copper).

26.  $AmHo$ , ammoniac hydrate (solution of ammonia), gives a greenish-blue ppt. of  $CuHo_2$ , cupric hydrate (hydrated oxide of copper); if the  $AmHo$  is in excess, this ppt. will dissolve into a splendid clear dark blue solution.

27. Place a piece of platinum foil into a vessel containing a cupric solution, acidified with HCl, hydrochloric acid (muriatic acid), along with a piece of sheet zinc, both being arranged in the vessel so as to touch each other; the platinum becomes coated with metallic copper.

28. Compounds of copper, heated with  $CONaO_2$ , sodic carbonate (soda, carbonate of soda), and

KCy, potassic cyanide (cyanide of potass), upon charcoal, in the inner blowpipe flame, yield beads of metallic copper.

29. If a piece of bright iron be dipped into even a very slightly acidulated solution of a cupric salt, it becomes coated with a red metallic coating of copper.

### **Cd.**—CADMIUM.

30. The yellowish ppt. **CdS**, cadmic sulphide (sulphide of cadmium), produced by **SH<sub>2</sub>**, hydro-sulphuric acid (sulphuretted hydrogen) (see T. 1, Gr. B), is insoluble in alkaline hydrates and sulphides; it is also insoluble in HCl, hydrochloric acid (muriatic acid). **SHAm**, Hydric ammoniac sulphide (hydrated ammoniac sulphide), will also give the above ppt. **CdS**. This precipitate is not volatile when heated, thus being distinguished from **As<sub>2</sub>S<sub>3</sub>**, arsenious sulphide, which it otherwise resembles.

31. Compounds of cadmium heated with **CONa<sub>2</sub>O**, sodic carbonate (soda, carbonate of soda), upon charcoal, in the inner blowpipe flame, produce a yellowish-red incrustation of **CdO**,

cadmic oxide (oxide of cadmium), owing to the reduction and subsequent oxidation of the metal.

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(TABLE 3.)

SUBDIVISION 2 consists of those metals whose sulphides are soluble in alkaline hydrates and sulphides.

**Sn.**—**TIN** as a stannous compound.

32. The chocolate-coloured brown ppt. **SnS**, stannous sulphide (protosulphide of tin), produced by **SH<sub>2</sub>**, hydrosulphuric acid (sulphuretted hydrogen) (see T. 1, Gr. B), is soluble in **NaHo**, sodic hydrate (caustic potash).

33. **AuCl<sub>3</sub>**, auric chloride (chloride of gold), added in excess to weak dilute solutions of stannous salts, gives a brown ppt. of gold; should the stannous salt be in excess, the beautiful purple (purple of cassius) ppt. will result.

34. **HgCl<sub>2</sub>**, mercuric chloride (perchloride of mercury, corrosive sublimate), gives a white ppt. of **Hg<sub>2</sub>Cl<sub>2</sub>**, mercurous chloride (subchloride of mercury, calomel), which turns grey if the stan-

nous salt is in excess, owing to its being reduced to metallic mercury by the stannous salt.

35. Compounds of tin, heated with  $\text{CONaO}_2$ , sodic carbonate (soda, carbonate of soda), and  $\text{KCy}$ , potassic cyanide (cyanide of potass), upon charcoal, in blowpipe flame, yield malleable beads of metallic tin.

**Sn<sup>IV</sup>**.—**TIN** as a stannic compound.

36. The dingy yellow ppt.  $\text{SnS}_2$ , stannic sulphide (bisulphide of tin), produced by  $\text{SH}_2$ , sulphuretted hydrogen (hydrosulphuric acid) (see T. 1, Gr. B), is soluble in  $\text{KHo}$ , potassic hydrate (caustic potash), and  $\text{AmHo}$ , ammoniac hydrate (solution of ammonia).

37.  $\text{NaHo}$ , sodic hydrate (caustic potash), gives a powdery white ppt. of  $\text{SnOHO}_2$ , stannic acid (hydrated binoxide of tin), which is soluble in excess of the reagent;  $\text{AmHo}$ , ammoniac hydrate (solution of ammonia), gives the same ppt., but it is not soluble in excess of  $\text{AmHo}$ .

38. Reaction No. 35 also takes place with stannic as well as stannous compounds.

39. If a strip of zinc be placed in a solution of tin, made slightly acid with  $\text{HCl}$ , it becomes

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coated with metallic tin, either as a spongy mass or a grey lamina. Platinum foil, if in contact with the zinc, does not become blackened.

**As.**—ARSENIC.

40. Marsh's Test.—Introduce a portion of the

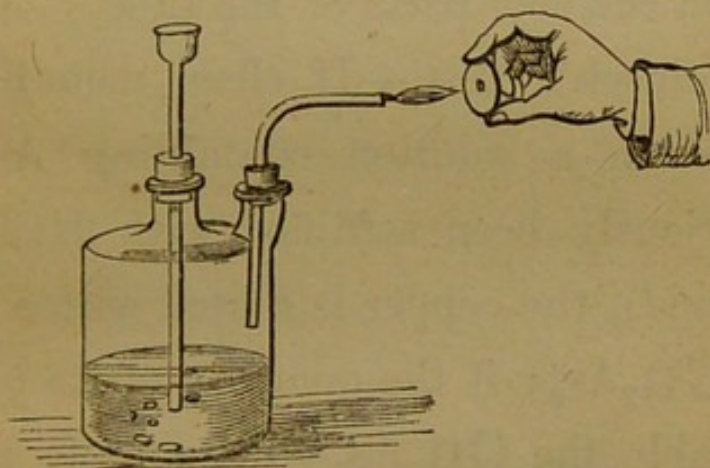


Fig. 2.

substance or solution into an apparatus evolving pure hydrogen, such apparatus having a gas jet attached, as represented; in which are placed the zinc and sulphuric acid together with the substance to be tested. If now the escaping gas  $\text{AsH}_3$ , arseniuretted hydrogen (trihydride of arsenic), be ignited, it will burn with a greenish-blue flame, giving off fumes of  $\text{As}_2\text{O}_3$ , arsenious anhydride (white oxide of arsenic). If whilst ignited a piece of cold porcelain or common dish

be placed over the flame, a black mirror of metallic arsenic will be deposited upon it.

Place a drop of  $\text{Na}_2(\text{OCl})\text{Cl}$ , sodic chloro-hypochlorite (chloride of soda), upon this mirror, *will be dissolved at once.*

41. Compounds of arsenic, heated with  $\text{CONa}$  and  $\text{KCy}$  in a bulb test tube, yield a black metallic ring upon the cool parts of the tube.

42. Reinsch's Test.—If clean metallic copper be boiled in a solution containing As, which has previously been acidified with  $\text{HCl}$  (hydrochloric acid), the copper is coated with a *steel grey* film of  $\text{Cu}_2\text{As}_3$ ; if the quantity of As present be considerable, the  $\text{Cu}_2\text{As}_3$  will separate after boiling for some time, in *large black scales*. The copper wire, or foil, is then carefully washed and heated in an open tube, the arsenic oxidizes and deposits on the cool part of the tube in octahedral crystals of  $\text{As}_2\text{O}_3$ , arsenious anhydride.

43.  $\text{SO}_2\text{Cuo}''$ , cupric sulphate, produces in neutral solutions of the arseniates a greenish-blue precipitate of  $\text{AsHoCuo}''$ , hydric-cupric arsenite (Scheele's green).

arsenic acid =  $Ag_3 AsO_4$  arsenate of silver

Brick red colour arsenite of silver

arsenious acid =  $Ag_3 AsO_3$

Canary colour

sent is extremely small, a *pink tinge* pervades the solution.

48.  $\left\{ \begin{array}{l} \text{COH}_o \\ \text{COH}_o \end{array} \right.$  oxalic acid, gives a dark brown ppt.

of gold, owing to its reduction to the metallic state by the oxalic acid.

49. Ferrous salts precipitate gold from its solutions as a bluish-black precipitate, which, when rubbed, acquires a metallic lustre.

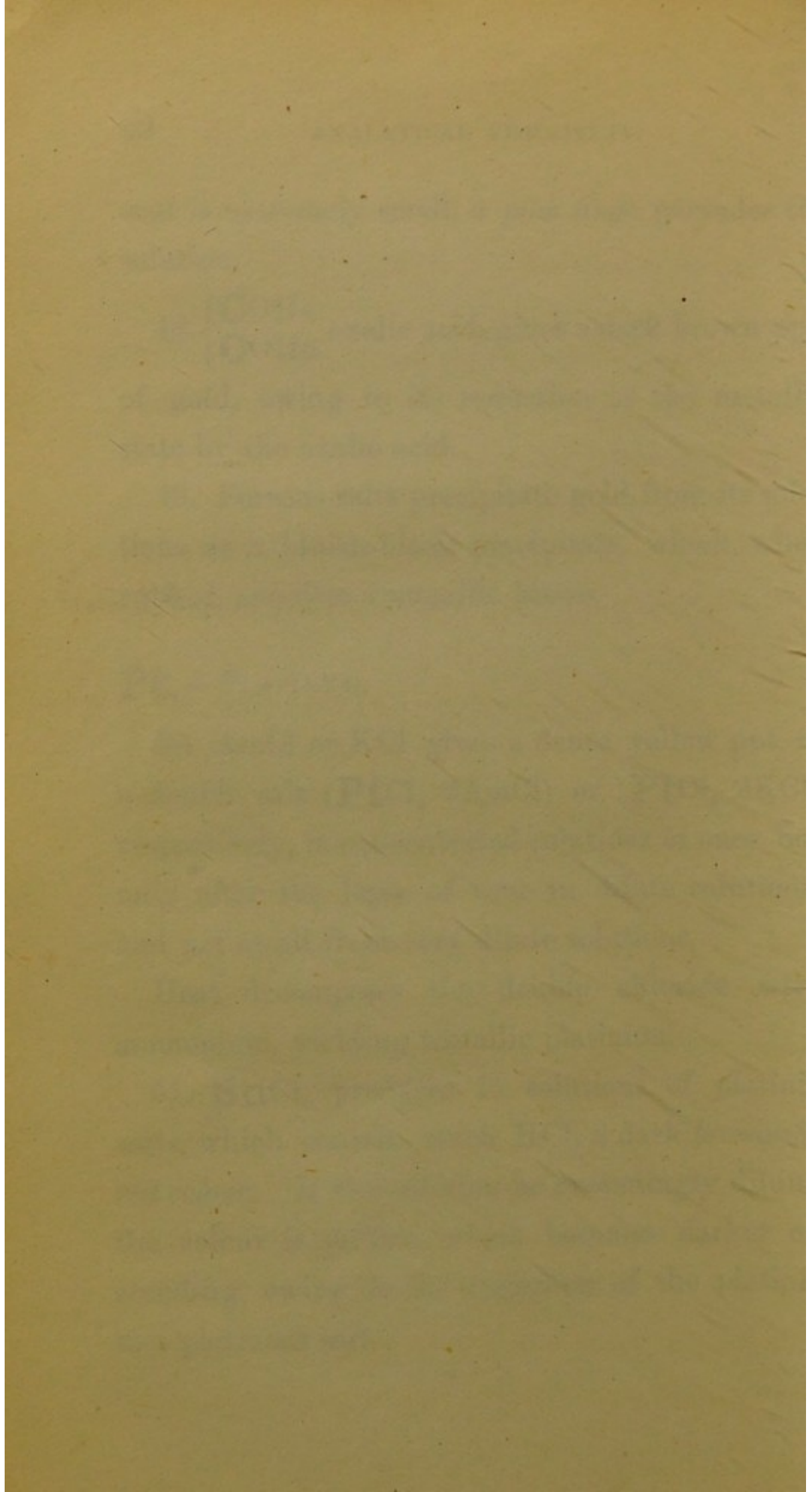
### **Pt.**—PLATINUM.

50. AmCl or KCl gives a dense yellow ppt. of a double salt ( $\text{PtCl}_4 \cdot 2\text{AmCl}$ ) or ( $\text{PtCl}_4 \cdot 2\text{KCl}$ ) respectively, in concentrated solutions at once, but only after the lapse of time in dilute solutions, and not at all from very dilute solutions.

Heat decomposes the double chloride with ammonium, yielding metallic platinum.

51.  $\text{SnCl}_2$  produces in solutions of platonic salts, which contain much HCl, a dark *brownish-red colour*. If the solution be exceedingly dilute, the colour is yellow, which becomes darker on standing, owing to the reduction of the platonic to a platinous salt.

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## GROUP C

(TABLE 5)

(Consists of those metals which are precipitated after the separation of Groups A and B, by AmHo, ammoniac hydrate, in the presence of excess of AmCl, ammoniac chloride, as hydrates.

(Cr.—CHROMIUM.

*being insoluble in*

52.  $\text{Na}_2(\text{OCl})\text{Cl}$ , sodic chloro-hypochlorite (chloride of soda), boiled with solutions of chromium for a period of time, change them into a yellowish colour, owing to the formation of a chromate.

53. NaHo gives a dingy green ppt. of  $\text{Cr}_2\text{Ho}_6$ , chromic hydrate (hydrated sesquioxide of chromium), soluble in excess of the reagent, but it may be reprecipitated by considerable boiling.

54. Compounds of chromium colour the borax bead light green, both in the inner and outer flame.

**Al.**—ALUMINIUM.

55. NaHo, sodic hydrate (caustic soda), gives a gelatinous dingy-coloured white ppt. of  $\text{Al}_2\text{Ho}_6$ ,

aluminic hydrate (hydrate of alumina), soluble in excess of the reagent, but it may be reprecipitated by the addition of AmCl, ammoniac chloride (sal ammoniac).  $KHO + AmCl = AmHo + KCl.$

56. AmHo also gives the ppt.  $Al_2Ho_6$ , which is only soluble in very large excess of AmHo.

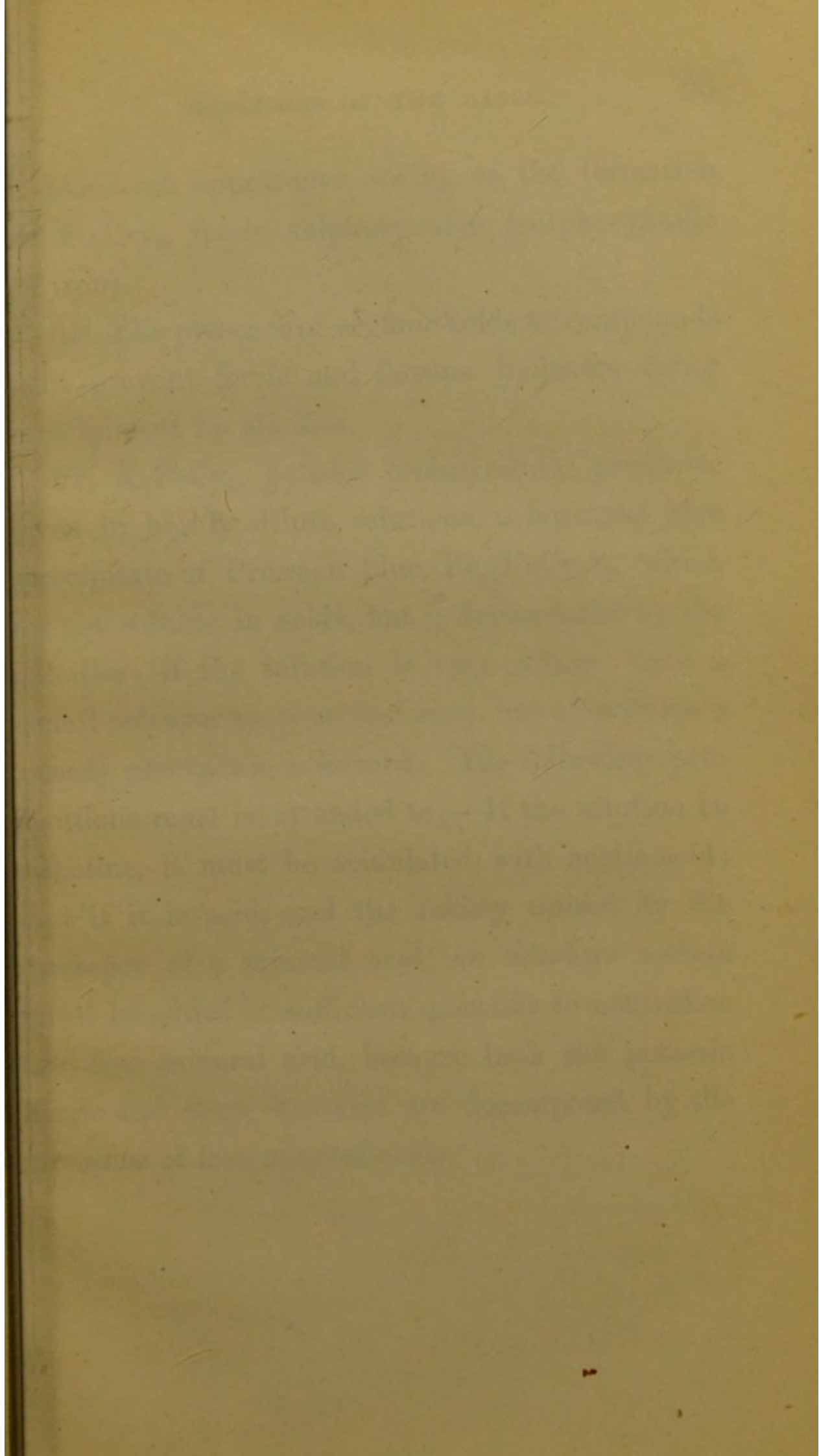
57. Compounds of aluminium, if heated upon charcoal in the blowpipe flame, and then moistened with  $\left\{ \begin{array}{l} NO_2 \\ NO_2 \end{array} \right. CoO''$ , cobaltous nitrate (nitrate of cobalt), and again heated in the inner blowpipe flame, yield a splendid blue substance.

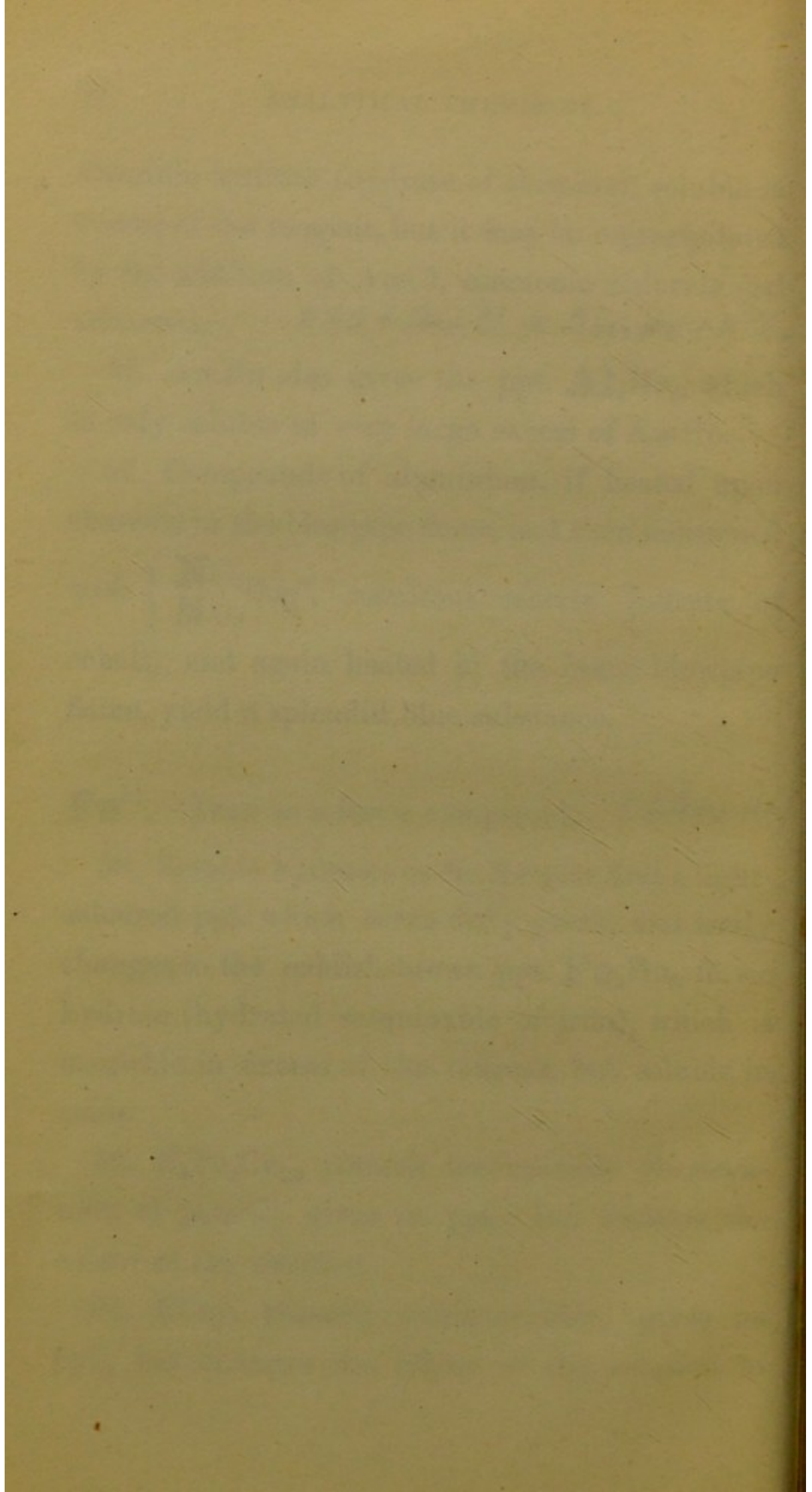
**Fe'''**.—IRON as a ferric compound. *(6AmHo)*

58. Soluble hydrates as NaHo give first a light-coloured ppt. which turns dirty green, and lastly changes to the reddish-brown ppt.  $Fe_2Ho_6$ , ferric hydrate (hydrated sesquioxide of iron), which is insoluble in excess of the reagent, but soluble in acids.

59.  $K_6Fe_2Cy_{12}$ , potassic ferricyanide (ferricyanide of potass), gives no ppt., but darkens the colour of the solution.

60. KCsy, potassic sulphocyanide, gives no ppt., but changes the colour of the solution to





a. blood-red appearance, owing to the formation of  $\text{Fe}_2\text{Csy}_6$ , ferric sulphocyanide (sulphocyanide of iron).

61. The presence of organic acids or compounds will prevent ferric and ferrous hydrates being precipitated by alkalies.

62.  $\text{K}_4\text{FeCy}_6$ , potassic ferrocyanide, produces, even in highly dilute solutions, a beautiful blue precipitate of Prussian blue,  $\text{Fe}_4(\text{FeCy}_6)_3$ , which is not soluble in acids, but is decomposed by the alkalies; if the solution is very dilute, only a small colouration is at first seen, but afterwards a scanty precipitate is formed. The following precautions must be attended to:—If the solution be alkaline, it must be acidulated with acetic acid; but if it is acid, and the acidity caused by the presence of a mineral acid, an alkaline acetate must be added in sufficient quantity to neutralise the free mineral acid, because both the potassic ferric and ferro cyanides are decomposed by the presence of free mineral acids.

## GROUP D

(TABLE 6)

Consists of those metals which are precipitated by **SHAm**, hydric ammoniac sulphide, after separation of Groups A, B, and C.

**Fe''**.—IRON as a ferrous compound.

63.  $K_6Fe_2Cy_{12}$ , potassic ferricyanide (ferricyanide of potass), gives a darkish blue ppt. of **Fe<sub>3</sub>Fe<sub>2</sub>Cy<sub>12</sub>**, ferrous ferricyanide (ferricyanide of iron), which is insoluble in weak acids.

64. **AmHo**, ammoniac hydrate, gives a light straw-coloured ppt. of **FeHo<sub>2</sub>**, ferrous hydrate (hydrated protoxide of iron), which soon changes to a brownish-green, and lastly to the reddish-brown ppt., owing to the absorption of oxygen.

If salts of ammonium are present, they will prevent the precipitation of ferrous hydrate by ammoniac hydrate.

65. **KCsy**, potassic sulphocyanide (sulphocyanide of potass), does neither change the colour of the solution, nor give a ppt.

66. Reaction No. 61 also applies here.

67.  $K_4FeCy_6$ , potassic ferrocyanide, produces at

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First a bluish-white ppt. of  $\text{FeK}_2\text{FeCy}_6$ , which soon turns blue by absorbing oxygen from the air. Free alkalies prevent its formation, and if added when formed, speedily decompose it. It is insoluble in the dilute acids.

### Co.—COBALT.

68.  $\text{KCy}$ , potassic cyanide (cyanide of potass), gives a light slate-coloured ppt. of  $\text{CoCy}_2$ , cobaltous cyanide (dicyanide of cobalt), soluble in excess of the reagent, owing to the formation of  $\text{K}_2\text{CoCy}_4$ , potassic cobalticyanide (cobalticyanide of potass).

When this solution is boiled with  $\text{Na}_2(\text{OCl})\text{Cl}$ , it will give *no* ppt.

69.  $\text{NaHo}$  gives a blue ppt. of  $\text{CoHo}_2$ , cobaltous hydrate (hydrated oxide of cobalt), insoluble in excess of the reagent; if boiled, it changes to a light red.

70. Compounds of cobalt, heated, give a blue colour to the borax bead, both in the outer and inner flame.

71. "If the solution contains Ni as well as Co, the addition of  $\text{HCl}$  to the solution of the cyanide produces a greenish precipitate, which always

contains the whole of the Ni, and under particular circumstances, all the Co, that is, when these two metals are in the proportion of 3 eq. of Ni to 2 eq. of Co. The precipitate consists then of nickelous cobaltic cyanide,  $\text{Ni}_3\text{Co}_2\text{Cy}_{12}$ . In cases of a larger proportion of Ni, the precipitate is a mixture of  $\text{NiCy}_2$  and the former compound; but if the proportion of Ni is smaller, a part of the Co remains in solution, as  $\text{K}_6\text{Co}_2\text{Cy}_{12}$ ."—*Will.*

### **Ni.**—NICKEL.

72.  $\text{KCy}$ , potassic cyanide, gives a greenish ppt. of  $\text{NiCy}_2$ , nickelous cyanide (dicyanide of nickel), soluble in excess of the reagent, changing to a dingy yellow-coloured solution.

73. When this solution is boiled with  $\text{Na}_2(\text{OCl})\text{Cl}$ , it will give a black ppt. of  $\text{Ni}_2\text{O}_3$ , nickelic oxide (sesquioxide of nickel).

74.  $\text{NaHo}$  gives a light green ppt. of  $\text{NiHo}_2$ , nickelous hydrate (hydrated protoxide of nickel), insoluble in excess of the reagent.

75. Compounds of nickel, when heated in the outer blowpipe flame, colour the borax bead yellowish-red whilst hot, but upon cooling it

fades to a lighter colour; if heated in the inner flame, specks of grey metallic nickel are seen in the bead.

### **Zn.**—ZINC.

76.  $\text{KHo}$  gives a gelatinous white ppt. of  $\text{ZnHo}_2$ , zincic hydrate (hydrated oxide of zinc), soluble in excess of the reagent.

77. Compounds of zinc heated upon charcoal in the blowpipe flame, and then moistened with  $\left\{ \begin{array}{l} \text{NO}_2\text{Coo}'' \\ \text{NO}_2 \end{array} \right.$ , cobaltous nitrate (nitrate of cobalt), and again heated, yield a splendid green substance.

### **Mn.**—MANGANESE.

78.  $\text{KHo}$  gives a dirty white ppt. of  $\text{MnHo}_2$ , manganous hydrate (hydrated oxide of manganese), which will darken if exposed to the atmosphere in an open flask.

79. Compounds of manganese heated with  $\text{NO}_2\text{Ko}$ , potassic nitrate (nitrate of potash, nitre) and fusion mixture, upon platinum foil, yield a beautiful green mass.

80.  $\text{AmHo}$ , ammonia, gives the same ppt.,

unless ammoniac salts be present, which prevent the precipitation.

81. If a few drops of a fluid containing a manganous compound in solution, are placed on  $\text{PbO}_2$  or  $\text{Pb}_3\text{O}_4$ , with the addition of  $\text{NO}_2\text{Ho}$ , and the mixture boiled, the  $\left\{ \begin{array}{l} \text{NO}_3 \\ \text{NO}_3 \end{array} \right. \text{NO}_3\text{Mn}$  formed, imparts a purple-red colour to the fluid. Cl must be absent.

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## GROUP E

(TABLE 7)

### SUB-DIVISION No. 1,

Consists of those metals which are precipitated by  $\text{COAmO}_2$ , ammoniac carbonate (sesquicarbonate of ammonia), in the presence of excess of  $\text{AmCl}$  (ammoniac chloride) as carbonates, after the separation of Groups A, B, C and D.

### **Ba.**—BARIUM.

82.  $\text{CrO}_2\text{Ko}_2$ , potassic chromate (chromate of potash), gives a nice yellow ppt. of  $\text{CrO}_2\text{Bao}$ , baryic chromate (chromate of baryta), insoluble in

excess of the reagent and weak acids, but soluble in  $\text{NO}_2\text{Ho}$ , nitric acid (aqua fortis).

83.  $\text{SO}_2\text{Cao}''$ , calcic sulphate (sulphate of lime), gives at *once* a powdery white ppt. of  $\text{SO}_2\text{Bao}''$ , baric sulphate (sulphate of baryta), which is insoluble in alkalies and weak acids.

84. Flame test, as in preliminary, greenish-yellow.

85.  $\text{SiH}_2\text{F}_6$ , hydrofluosilicic acid, added either to a neutral or alkaline solution of Ba, throws down a white precipitate of  $\text{BaSiF}_6$ , which appears only after much agitation and the lapse of some time; addition of alcohol renders the precipitate rapid and complete.

### **Sr.**—STRONTIUM.

86.  $\text{CrO}_2\text{Ko}_2$ , potassic chromate (chromate of potash), gives *no* ppt.

87.  $\text{SO}_2\text{Cao}''$ , calcic sulphate (sulphate of lime), gives, *after the lapse of time*, a white ppt. of  $\text{SO}_2\text{Sro}''$ , strontic sulphate (sulphate of strontia), very slightly soluble in  $\text{NO}_2\text{Ho}$  or HCl.

88. Flame test, as in preliminary, intense crimson.

**Ca.**—CALCIUM.

89.  $\text{CrO}_2\text{Ko}_2$ , gives no ppt. owing to  $\text{CrO}_2\text{Cao}''$ , calcic chromate (chromate of lime), being soluble.

90. Soluble sulphates give, *in concentrated solutions only*, a white ppt. of  $\text{SO}_2\text{Cao}''$ , soluble in excess of water.

91.  $\left\{ \begin{array}{l} \text{COAmo}, \\ \text{COAmo}, \end{array} \right.$  ammoniac oxalate (oxalate of ammonia), gives immediately a white ppt. of  $\left\{ \begin{array}{l} \text{CO} \\ \text{CO} \end{array} \right. \text{Cao}''$ , calcic oxalate (oxalate of lime), insoluble in weak acids, but freely soluble in HCl.

92. Flame test, as in preliminary, light red or orange.

93. "The three alkaline earths may be detected in mixtures containing all three of them, by the different colouration which they severally impart to the flame. To this end the sample under examination is repeatedly moistened with  $\text{SO}_2\text{Ho}_2$ , then cautiously dried, and introduced into the fusion zone of the gas flame. After the evaporation of the alkalies that may chance to be present, the baric colouration will make its appearance alone. After this colouration has completely disappeared, and the sample moistened with HCl, gives, on spirting

no longer, a flame colouration of a bluish-green tint when viewed through the green glass. The sample is moistened again with HCl, and tested for Ca, by viewing through the green glass. The sample is moistened again with HCl, and tested for Sr, as for Ca."—*Fresenius*.

### SUB-DIVISION No. 2

Consists of that metal which is precipitated by  $\text{POHoNaO}_2$ , hydric disodic phosphate, after the separation of Sub-Division 1 as a phosphate.

#### **Mg.**—MAGNESIUM.

94.  $\text{COAmo}_2$ , ammoniac carbonate (sesquicarbonate of ammonia), with application of heat, gives a ppt. of  $\text{COMgo}''$ , magnesian carbonate (carbonate of magnesia), soluble in the presence of AmCl.

95. AmCl and AmHo together do not cause any ppt., but if  $\text{POHoNaO}_2$ , hydric disodic phosphate (hydrated phosphate of soda), be then added, a white crystalline ppt. of  $\text{POAmoMgo}''$ , ammoniac magnesian phosphate (magnesian phosphate of ammonia), will be obtained, which is soluble in acids, but insoluble in AmHo.

96.  $\text{AmHo}$  by itself gives a gelatinous white ppt. of  $\text{MgHO}_2$ , magnesian hydrate (hydrate of magnesia), insoluble in excess of the reagent, but freely soluble in  $\text{AmCl}$ .

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## GROUP F

(TABLE 8)

Consists of those metals which are not precipitated by any of the group reagents.

**Na.**—SODIUM.

97.  $\text{PtCl}_4$ , platinic chloride (tetrachloride or platinum) and tartaric acid give *no* ppt.

98. Flame test, as in preliminary, golden yellow.

**K.**—POTASSIUM.

99. Tartaric acid gives a white crystalline ppt. of hydric potassic tartrate (cream of tartar) from concentrated neutral solutions.

100.  $\text{PtCl}_4$ , platinic chloride (tetrachloride of platinum), gives a dense yellow ppt. of a double salt ( $\text{PtCl}_4, 2\text{KCl}$ ) from concentrated acid solu-

tions. Dilute solutions will only give a ppt. after the lapse of time, and very dilute, not at all. This ppt. is *very* slightly soluble in free acids.

101. Flame test, as in preliminary, violet; if seen through blue cobalt glass, *purple*.

**Am.**—AMMONIUM.

102. **PtCl<sub>4</sub>**, platinic chloride (tetrachloride of platinum), gives a dense yellow ppt. of a double chloride (**PtCl<sub>4</sub>,2AmCl**) in concentrated neutral or acidified solutions, which is decomposed by the application of heat.

103. Compounds of ammonium heated with NaHo, in a glass vessel, give off NH<sub>3</sub>, ammonia gas, known by its smell.

104. Nessler's Test for the detection of ammonia as in natural waters, is as follows:—If a solution of **HgI<sub>2</sub>**, mercuric iodide, in potassic iodide, is added to a solution containing an ammoniacal salt or free ammonia, to which has been added (KH<sub>o</sub>) (potassic hydrate), a brown precipitate or colouration is produced. This is by far the most delicate test for ammonia, and is thus spoken of by Wanklyn and Chapman:—“In the whole range of chemical analysis there is no determination

which surpasses that of ammonia in point of delicacy. It is questionable, indeed, whether any other approaches it. The Nessler test is capable of indicating one part of ammonia in 20,000,000 parts of water."

## REACTIONS OF THE PRINCIPAL ACIDS.

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The acids may be classified as follows:—

### GROUP I.

#### SUB-DIVISION No. 1,

Consists of those acids which are precipitated by **SH<sub>2</sub>**, hydrosulphuric acid (sulphuretted hydrogen), from acid solutions, as sulphides.

**AsH<sub>3</sub>**, **AsOH<sub>3</sub>**.—ARSENIOUS and ARSENIC ACIDS.

105. See Reactions Nos. 40 and 41.

#### SUB-DIVISION No. 2

Precipitated as in Division No. 1, only as the oxide instead of the sulphide.

**CrO<sub>2</sub>H<sub>2</sub>**.—CHROMIC ACID.

106. **NO<sub>2</sub>Ag**, argentic nitrate (nitrate of

silver), gives a purple-coloured ppt. of  $\text{CrO}_2\text{AgO}_2$ , argentic chromate (chromate of silver).

107.  $\left\{ \begin{array}{l} \text{C}(\text{Me})\text{O}, \\ \text{Pbo}'' \\ \text{C}(\text{Me})\text{O}, \end{array} \right.$  plumbic acetate (acetate of

lead), gives a nice yellow ppt. of  $\text{CrO}_2\text{Pbo}''$ , plumbic chromate (chromate of lead), insoluble in weak acids.

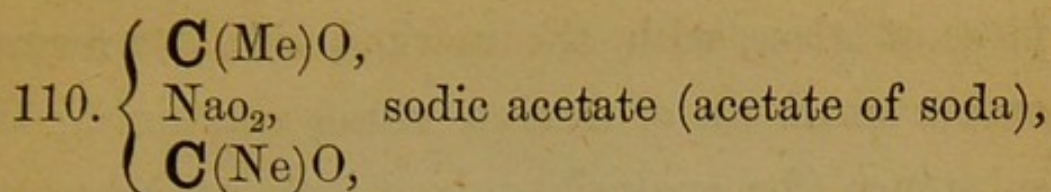
## GROUP II.

Consists of those acids which are precipitated from neutral solutions by  $\text{BaCl}_2$ , baric chloride (chloride of barium), after the separation of Group I.

$\text{POH}_3$ .—PHOSPHORIC ACID.

108.  $\text{NO}_2\text{AgO}$ , argentic nitrate (nitrate of silver), gives a cream-coloured ppt. of  $\text{POAgO}_3$ , argentic phosphate (phosphate of silver).

109.  $\text{AmHo}$ ,  $\text{AmCl}$ , and  $\text{SO}_2\text{Mgo}''$ , magnesian sulphate (sulphate of magnesia), added to an aqueous alkaline phosphate, give a crystalline white ppt. of  $\text{POAmoMgo}''$ . See Reaction No. 95.



added in excess with a drop of  $\mathbf{Fe}_2\text{Cl}_6$ , ferric chloride (hexa-chloride of iron), to a  $\text{HCl}$  solution of a phosphate which must not contain much free acid, gives a gelatinous cream-yellow ppt. of ferric phosphate, insoluble in weak acid, such

as  $\left\{ \begin{array}{l} \mathbf{C}\text{H}_3, \\ \mathbf{C}\text{O}\text{H}_0, \end{array} \right.$  acetic acid.

$\left\{ \begin{array}{l} \mathbf{C}\text{O}\text{H}_0, \\ \mathbf{C}\text{O}\text{H}_0. \end{array} \right.$ —OXALIC ACID.

111. Soluble lime salts give a white ppt. of  $\left\{ \begin{array}{l} \mathbf{C}\text{O} \\ \mathbf{C}\text{O} \end{array} \right. \text{Cao}''$ , calcic oxalate (oxalate of lime). See Reaction No. 91.

$\mathbf{B}\text{H}_0_3$ .—BORIC ACID.

112. Acidulate with a little  $\text{HCl}$ , place a small strip of turmeric paper about a third in the solution, carefully withdraw, and slowly dry the immersed part, after which it will present a crimson appearance.

$\text{HF}$ .—HYDROFLUORIC ACID.

113. Trace a mark upon a piece of glass, covered with a thin layer of wax, after which place this

piece of glass, with the marked side downward, over a small tin or leaden vessel containing the substance (for analysis) mixed with concentrated  $\text{SO}_2\text{H}_2\text{O}_2$ . After having rubbed the wax off the glass, it will be found to be etched where the mark was traced.

### $\text{SiH}_4$ .—SILICIC ACID.

114. Add HCl in excess to any soluble silicate, then evaporate to dryness, and after cooling moisten it with HCl, and warm, after which add a quantity of water; white flakes of  $\text{SiO}_2$ , silicic anhydride (oxide of silicon), are left undissolved.

115. Insoluble silicates must be fused with four times their weight of fusion mixture, upon platinum foil, until no more  $\text{CO}_2$ , carbonic anhydride, is evolved; after which boil the mass with water, and treat as in Reaction No. 114.

### $\text{COH}_2$ .—CARBONIC ACID.

116. HCl added to carbonates causes effervescence, owing to the evolution of  $\text{CO}_2$ , carbonic anhydride. If this gas is decanted into a vessel containing clear lime-water, and well shaken, the lime-water will become milky, owing to the for-

mation of  $\text{COCaO}''$ , calcic carbonate (carbonate of lime).

$\text{SOHO}_2$ .—SULPHUROUS ACID.

117. By adding a little zinc and  $\text{HCl}$ , evolution of  $\text{SH}_2$  takes place, known by its smell.

$\text{SO}_2\text{HO}_2$ .—SULPHURIC ACID.

118.  $\left\{ \begin{array}{l} \text{C(Me)O}, \\ \text{Pbo}'' \\ \text{C(Me)O}, \end{array} \right.$  plumbic acetate, gives a white

ppt. of  $\text{SO}_2\text{Pbo}''$ , plumbic sulphate (sulphate of lead).

119.  $\text{BaCl}_2$ , baric chloride (chloride of barium), gives a white ppt. of  $\text{SO}_2\text{Bao}''$ , baric sulphate (sulphate of barium). See Reaction No. 83.

120. Heated with  $\text{CONaO}_2$  upon charcoal, in the inner blowpipe flame, yields  $\text{SNa}_2$ , sodic sulphide (sulphide of sodium), which, if treated with an acid, will evolve  $\text{SH}_2$ .

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### GROUP III.

Consists of those acids which are precipitated by  $\text{NO}_2\text{Ago}$ , argentic nitrate (nitrate of silver), from neutral solutions.

## HCl.—HYDROCHLORIC ACID.

121. Add **MnO<sub>2</sub>**, manganic oxide (black oxide of manganese), and **SO<sub>2</sub>Ho<sub>2</sub>**, then apply heat; evolution of chlorine takes place, known by its smell, colour, &c.

122. **NO<sub>2</sub>Ag**o gives a curdy-looking white ppt. of AgCl, argentic chloride (chloride of silver), soluble in AmHo, but insoluble in weak **NO<sub>2</sub>Ho**; exposure to the sunlight always darkens this ppt.

## HBr.—HYDROBROMIC ACID.

123. Add **MnO<sub>2</sub>** and **SO<sub>2</sub>Ho<sub>2</sub>**, then apply heat; evolution of bromine vapour takes place, which colours moistened starch paper *yellow*.

124. **NO<sub>2</sub>Ag**o gives a cream-coloured yellow ppt. of AgBr, slightly soluble in AmHo, but insoluble in **NO<sub>2</sub>Ho**.

## HI.—HYDRIODIC ACID.

125. Treat as in Reaction No. 123; evolution of iodine vapour of a violet or purple colour takes place, which colour moistened starch paper *blue*.

126. **NO<sub>2</sub>Ag**o gives a light yellow ppt. of AgI, argentic iodide (iodide of silver), soluble in KCy, but insoluble in weak **NO<sub>2</sub>Ho**.

127.  $\begin{matrix} \text{NO}, \\ \text{NO}, \end{matrix} \text{Hgo}''$ , mercuric nitrate (nitrate of mercury), gives a greenish-yellow ppt., insoluble in excess of the reagent.

**SH<sub>2</sub>**.—HYDROSULPHURIC ACID.

128. Addition of HCl and application of heat causes evolution of **SH<sub>2</sub>**, known by its nasty smell; sometimes a piece of zinc is required in the case of those sulphides insoluble in HCl.

129.  $\begin{matrix} \text{C(Me)O} \\ \text{Pbo}'' \\ \text{C(Me)O} \end{matrix}$  gives a black ppt. of **PbS**, plumbic sulphide (sulphide of lead), with soluble sulphides.

$\left\{ \begin{matrix} \text{CH}_3, \\ \text{COHo.} \end{matrix} \right.$  —ACETIC ACID.

(This acid is only partly precipitated by **NO<sub>2</sub>Ag**o.)

129A. Add **Fe<sub>2</sub>Cl<sub>6</sub>**, ferric chloride (perchloride of iron), to acetic solutions; it will change them to a darkish-red colour. A brownish ppt. will be obtained if this solution is well boiled.

## GROUP IV.

Consists of those acids which are not precipitated by  $\text{BaCl}_2$  or  $\text{NO}_2\text{Ag}$  from neutral solutions.

$\text{NO}_2\text{Ho}$ .—NITRIC ACID.

130. Add a concentrated solution of  $\text{SO}_2\text{Feo}''$ , ferrous sulphate (protosulphate of iron), in a small test tube, and slowly pour down the inside of the tube, held slanting, a little strong  $\text{SO}_2\text{Ho}_2$ , sulphuric acid; a brown-coloured ring forms at the top of the solution if  $\text{NO}_2\text{Ho}$  is present.

131. Heated with copper turnings and  $\text{SO}_2\text{Ho}_2$ , nitric oxide fumes are given off, which become red in the upper part of the tube by absorbing oxygen.

132. A nitrate, when heated, deflagrates, though less violently than a chlorate.

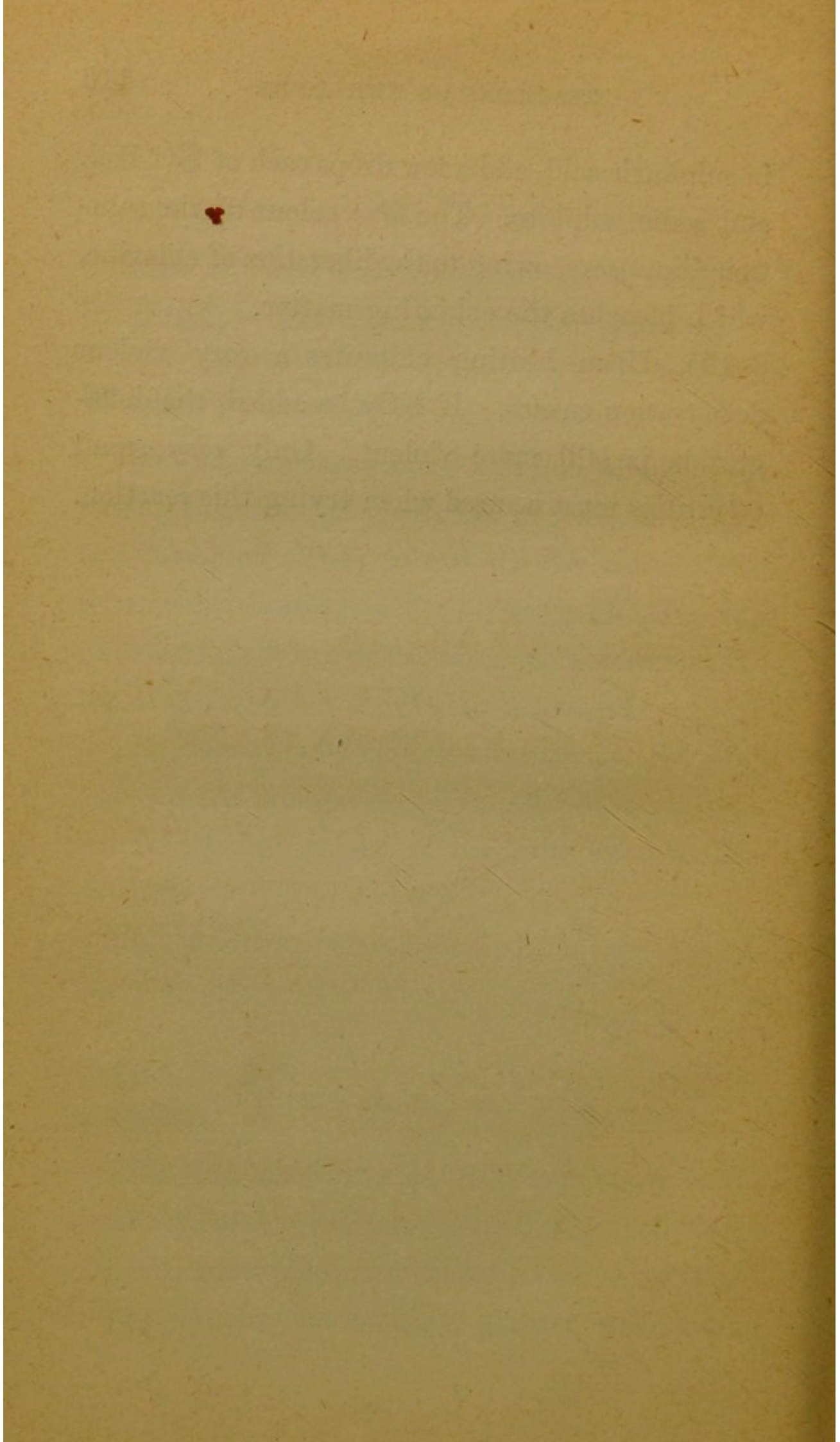
$\left\{ \begin{array}{l} \text{OCl}, \\ \text{OHo}. \end{array} \right.$  —CHLORIC ACID.

133. Add concentrated  $\text{HCl}$  and apply heat; evolution of a greenish-coloured gas, called *euchlorine*, takes place, known by its offensive smell.

134. Colour the solution slightly with indigo

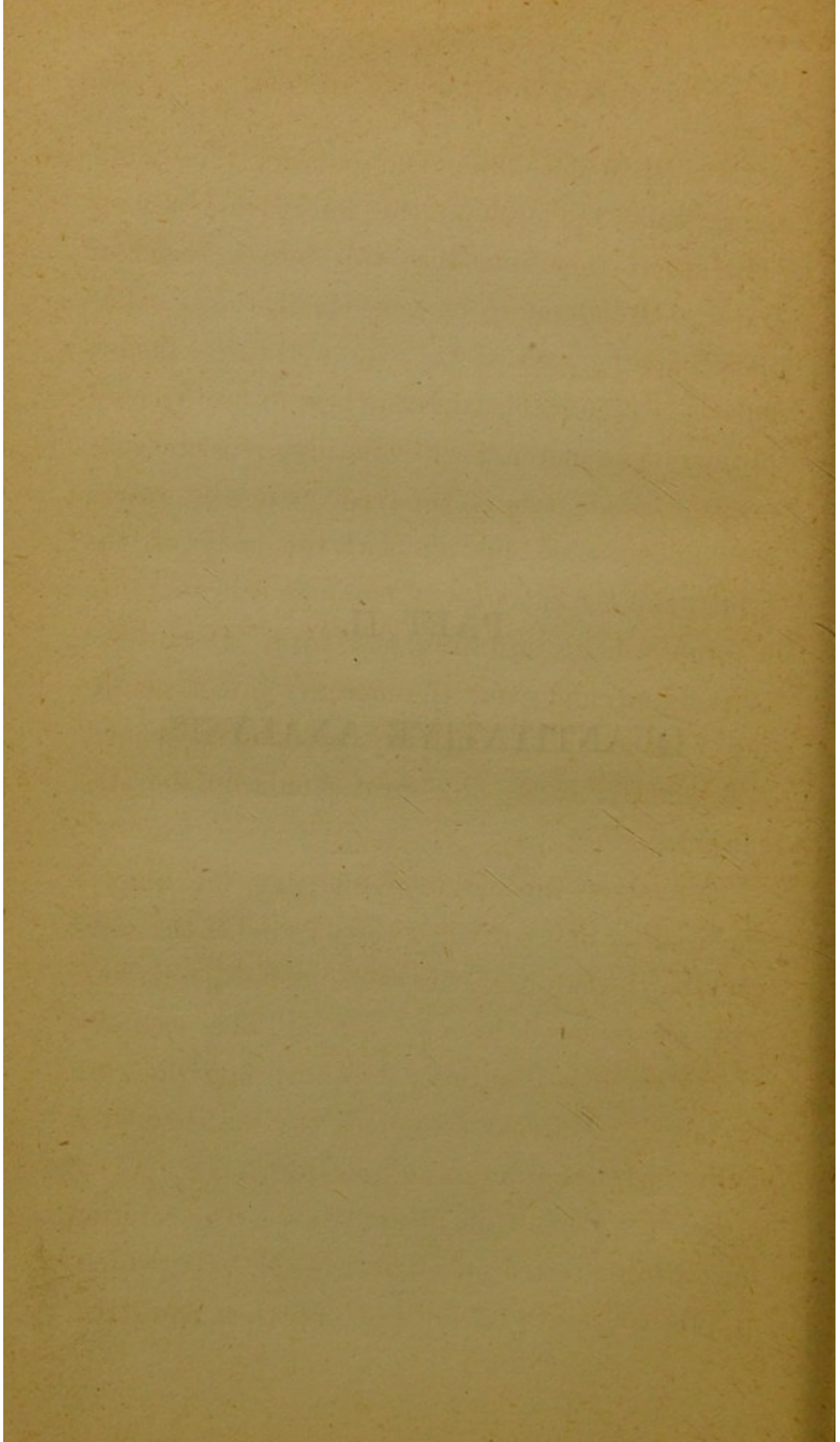
in sulphuric acid, add a few drops each of  $\text{SO}_2\text{Ho}_2$ , and sodic sulphite. The *blue* colour of the solution disappears, owing to the liberation of chlorine, which bleaches the colouring matter.

135. Upon heating chlorates a very violent deflagration ensues. If  $\text{KC}_y$  be added, the deflagration is still more violent. Only *very* small quantities must be used when trying this reaction.



PART II.

QUANTITATIVE ANALYSIS.



## QUANTITATIVE ANALYSIS.

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### REAGENTS.

THE following is a list of the reagents required, in addition to those for Qualitative Analysis:—

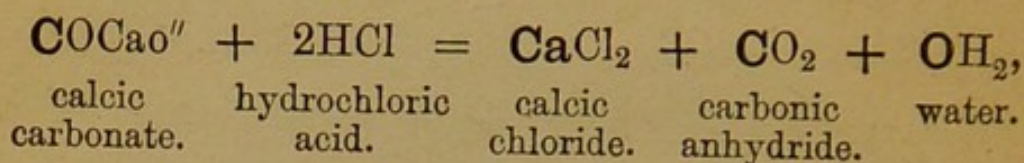
- CaCl<sub>2</sub>**, calcic chloride.
- Cl<sub>2</sub>**, chlorine gas.
- O<sub>2</sub>**, oxygen gas.
- H<sub>2</sub>**, hydrogen gas.
- Zn**, zinc granulated.
- Cu**, copper.
- CuO**, cupric oxide.
- CrO<sub>2</sub>Pbo''**, plumbic chromate.
- HF**, hydrofluoric acid.
- CrO<sub>2</sub>Ho<sub>2</sub>**, chromic acid.
- Uranic acetate.

### TESTING AND PREPARING OF REAGENTS.

#### **CaCl<sub>2</sub>**.—CALCIC CHLORIDE.

Calcic chloride (muriate of lime) is produced

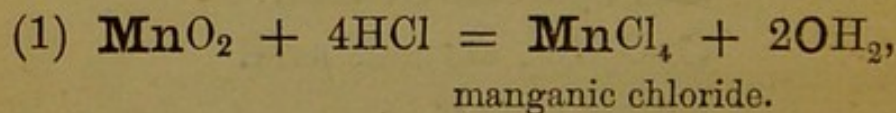
by the action of HCl, hydrochloric acid, on calcic carbonate,  $\text{COCaO}''$ , thus:—



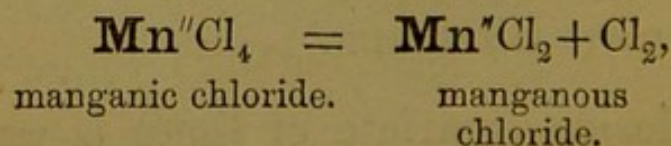
It crystallizes in six-sided prisms, which produce cold on solution in water as  $\text{CaCl}_2, 6\text{OH}_2$ . This reagent is also used for extracting the moisture from the air, owing to its great attraction for water.

#### $\text{Cl}_2$ .—CHLORINE.

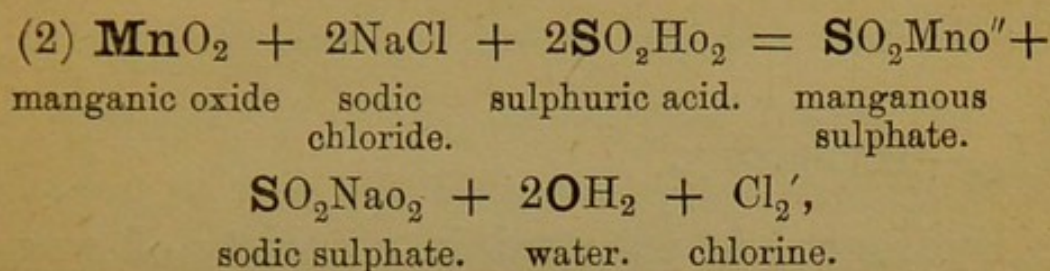
Chlorine is prepared by heating a mixture of manganic oxide,  $\text{MnO}_2$ , and hydrochloric acid, or a mixture of salt (sodic chloride), hydrochloric acid, and manganic oxide. This last,  $\text{MnO}_2$ , has no chemical reaction in the last equation. The following are the reactions:—



The manganic chloride under the influence of heat breaks up, thus:—



And for the second reaction—

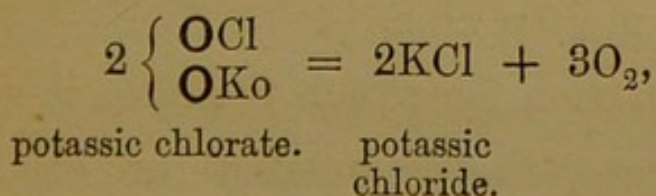


The readiest way to obtain Cl is to add some acid to bleaching powder.

The gas should then be passed through a solution of nitrate of lead, to free it from HCl, hydrochloric acid, which may have been given off, as the solution gives off gas on the application of heat.

### O<sub>2</sub>.—OXYGEN.

Oxygen can be prepared by heating potassic chlorate, thus:—



A little **MnO<sub>2</sub>**, manganic oxide, or  $\left\{ \begin{array}{l} \text{FeO}^{\text{O}} \\ \text{FeO}^{\text{O}} \end{array} \right.$  (peroxide, sesquioxide, or red oxide of iron), added, reduces the heat required for the giving off of the oxygen, but is liable to contaminate

it by traces of Fe or Mn, and from the potassic chlorate.

### **H<sub>2</sub>.**—HYDROGEN.

Hydrogen, is prepared in a variety of ways, but the one most generally employed is the addition of HCl to Zn, and then well drying the gas as it passes out of the flask.

### **Zn.**—ZINC.

If zinc be only required free from iron, the crude product obtained from the reduction of the ore may be used, as this generally contains only an inconsiderable amount of iron. The ordinary zinc of commerce contains much more iron, because the zinc is obtained by melting the crude material in an iron pan, and iron is slowly dissolved by molten zinc.

If the operation for which the zinc is required is very delicate, the commercial or crude zinc must be re-distilled from a clay or black-lead retort. The neck of the retort should hang down quite perpendicular over a vessel of water, and the neck constantly cleaned with a piece of iron wire.

**Cu.**—COPPER.

Copper is best prepared by the electrotype method, or by precipitating the metal from a solution of the sulphate, and then freeing from iron by boiling in hydrochloric acid, after which wash, dry, and roll into sheets.

If copper be pure, it should dissolve completely in  $\text{NO}_2\text{Ho}$ , nitric acid, and leave no trace of lead, iron, &c., when ammonia is added; and if the solution be precipitate by  $\text{SH}_2$ , sulphuretted hydrogen, the filtrate should leave no residue on evaporation.

**CuO.**—CUPRIC OXIDE.

Cupric oxide is obtained by treating pure copper scales with pure  $\text{NO}_2\text{Ho}$ , and then heating (after the effervescence has ceased) in a sand bath; and afterwards in a clay crucible to moderate red-

ness, till no more fumes of  $\left\{ \begin{array}{l} \text{NO}_2 \\ \text{NO}_2 \end{array} \right.$  nitric peroxide (nitrous gas) are given off, stirring from time to time with a hot glass rod.

**CrO<sub>2</sub>Pbo''.**—PLUMBIC CHROMATE.

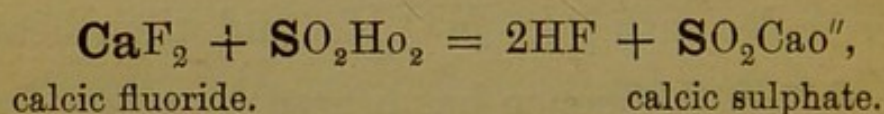
Plumbic chromate may be prepared by pre-

precipitating a filtered solution of acetate of lead with excess of bichromate of potassium; well washing the precipitate, by decantation, and then thoroughly on a linen strainer; dry, and afterwards heat to bright redness in a clay crucible, and then powder up.

#### HF.—HYDROFLUORIC ACID.

Hydrofluoric acid, when condensed, is a volatile colourless liquid, which emits suffocating white fumes in the air. It unites with water, and even very dilute solutions attack glass, and requires, therefore, to be kept in gutta-percha bottles.

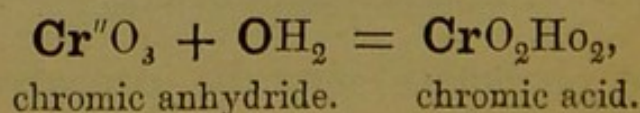
The following is the decomposition :—



It should give no precipitate on neutralization with potash.

#### $\text{CrO}_2\text{Ho}_2$ .—CHROMIC ACID.

Chromic acid is obtained by adding  $\text{Cr}''\text{O}_3$ , chromic anhydride, to water, thus :—



Great care must be taken with this reagent, as it is a very powerful oxidizing agent; for example, if thrown upon alcohol it sets it on fire.

URANIC ACETATE.—Heat finely-divided pitch-  
uline with nitric acid, and filter; then treat with  $\text{SH}_2$ , sulphuretted hydrogen; evaporate and treat with water, and again filter. Uranic nitrate crystallizes from the filtrate; purify this by recrystallization, and then heat the crystals with acetic acid, and allow the filtrate, after again filtering, to crystallize out. These crystals contain the uranic acid and the mother-liquid, the undecomposed nitrate.

On the addition of  $\text{SH}_2$  no change should take place.

## MANIPULATION.

For getting insoluble substances into solution, one of the most important things is that the substances should be very finely pulverized, especially those which are insoluble in acids; for example, felspar, trachyte, and granite will lose silica, and alkali glass, three or four per cent. of matter when in a finely pulverized condition and treated with water.

To powder substances up, a small steel anvil and hammer will be found convenient; then sifting on a piece of fine, well-washed, and thoroughly dry linen, and afterwards again pulverizing the parts which are left.

Care must be taken to prevent any loss in the substance under examination, more especially as the loss is not likely to be equally distributed among the component parts.

---

  
PRECIPITATES.

For filtering, the same apparatus as described on page 32 should be used, and the paper to be

of the Swedish description, which gives about .25 per cent. of ash, the paper being washed by acid before a delicate analysis. The following is an analysis of Swedish paper by Plantamour:—

Ferric oxide . . . . .	13.92
Lime . . . . .	12.83
Magnesia . . . . .	6.21
Alumina . . . . .	2.94
Silica . . . . .	63.23
	<hr/>
	99.13

In washing, it is preferable to use hot water. When it is intended to ignite the filtering paper, care must be taken that all water is carefully expelled, as any moisture that is left will be expelled with such rapidity that particles of solid matter may be easily carried away.

If the weight of the filtering paper is to be subtracted from the total weight, it is generally convenient to weigh between two watch-glasses.

For ignition of precipitates in crucibles, see below. The ignition is best performed over a Bunsen burner. When the precipitate requires to be ignited in crucibles or tubes, an apparatus

similar to fig. 3 should be used for the crucible, and fig. 4 for the tubes.

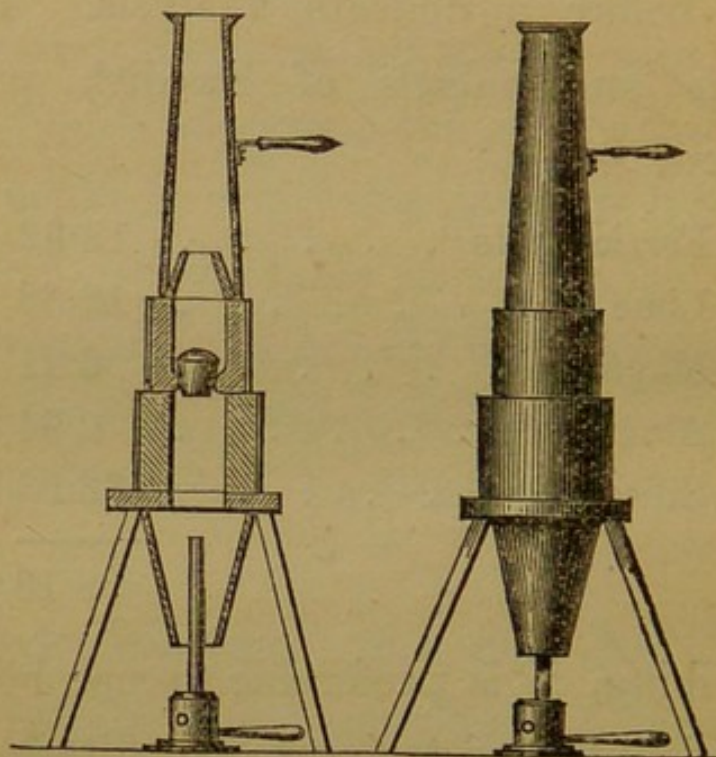


Fig. 3.

The whole of the furnace is represented, in section and in full; the power is sufficient to

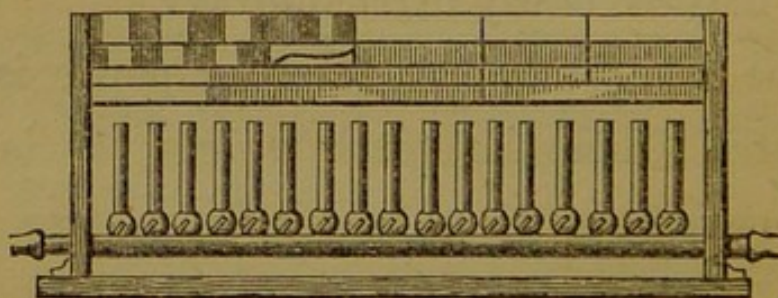


Fig. 4.

fuse 1,000 grains of anhydrous carbonate of soda in ten minutes, and will take in a crucible of  $2\frac{1}{3}$  by  $2\frac{1}{2}$  in.

Sometimes it is necessary to ignite in hydrogen. We then employ an ordinary crucible,

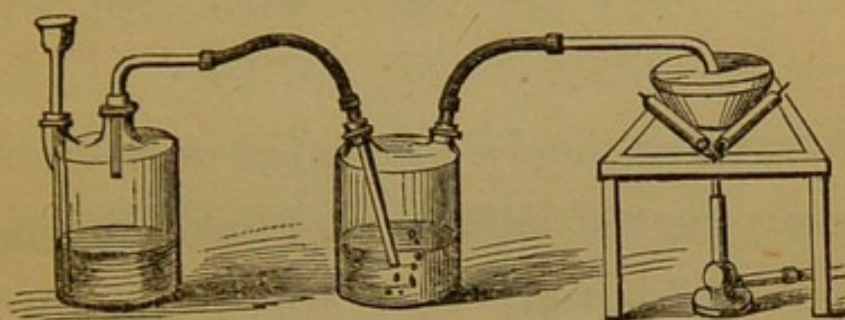


Fig. 5.

into which a tube connected with a Woolf's bottle is fitted.

MEASURING FLASK can be more easily obtained than made, although everything, wherever obtained, should always be carefully tested. If, however, the student has some practice in manipulating, measuring flasks are simple articles.

Measuring flasks are flasks that have *one mark* placed on the neck, which denotes when the quantity specified is contained in the flask. The standard flasks, which contain the normal test quantities, are three—1st, the litre, to be used with burettes that are graduated into centimetre cubes; 2nd, the decigallon, to be used with burettes which are graduated into septems; and 3rd, a flask to contain ten thousand grains of water, which is used with burettes graduated into decems.

It is also desirable that we should have some smaller, such as one-fourth, one-half, &c. These measuring flasks are made with and without stoppers.

BURETTES, or finely graduated test tubes, for measuring the test liquors used in each analysis. The forms of burette most frequently used are

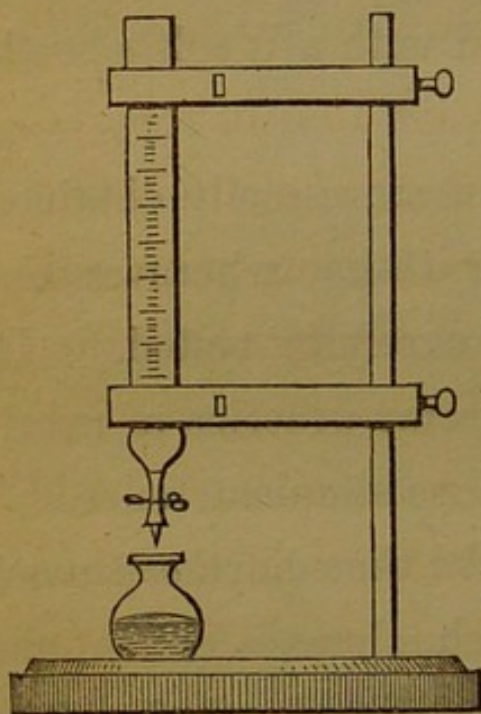


Fig. 6.

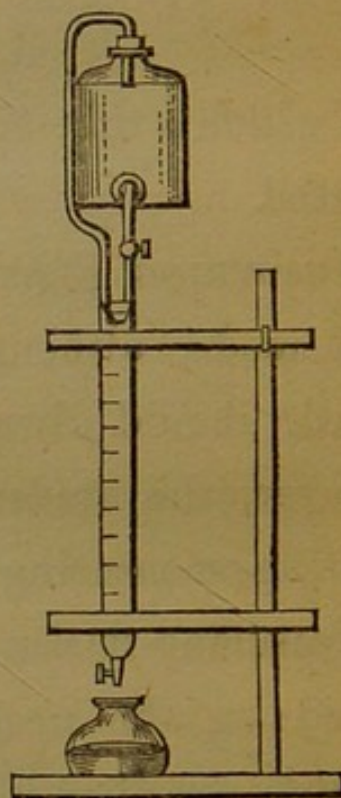


Fig. 7.

those known as Mohr's. Of these the most useful is that in fig. 6, but this cannot be used with certain test solutions, such as that of permanganate of potash, which is decomposed by contact with vulcanised caoutchouc, and we consequently have to use such as in fig. 7. This apparatus,

however, is not so convenient in use as the former kind, and requires great care in manipulating the stop-cock.

SIZES of burettes differ greatly. The most common capacity contains 50 centimètre cubes, or nearly 770 grains of water; or 100 septems, equal to 700 grains of water; or 100 décems, equal to 1,000 grains of water. But for special operations we require other sizes. The length of scale of the burette ought not to exceed fifteen or sixteen inches, whatever be the capacity, or else the instrument is inconveniently long. You ought also to be able to read the line  $0^{\circ}$  at the top of the scale, and the lowest number, without inconvenience.

The GRADUATION of the burette is usually made into centimètre cubes, septems, or décems, and each of these units into halves, fifths, or tenths. The facility for the exact reading of the scales on burettes is afforded by the use of Erdmann's float. The graduations of any tube for test should not be closer than one-twentieth of an inch. The reading of even millimètre scales, which are twenty-five to the inch, almost requires the use of a cathetometer, or magnifying instrument.

ERDMANN'S FLOAT is represented in fig. 8. It ought to float upright, and not lean against the sides of the burette. It should be of such a width as nearly to fill the tube, but yet allowing it freely to rise and fall with the liquid. To set the instrument at  $0^{\circ}$ , zero, the liquor must be first filled a little above the mark, and then run out till the ring on the float and the  $0^{\circ}$  division exactly coincide.



Fig. 8.

The ring must, on taking the observation, appear like a straight line. The absolute height of the liquor in the burette is to be disregarded. For any time reading the measurable height of the liquor, notice the division at which the height of the ring on the float indicates.

PINCHCOCKS are used for regulating, by means of screws or pressure, the amount of liquor which shall escape from a burette in a given time. There are generally two varieties, A and B, in fig. 9. A is only used when the drops are wanted at once; B, by means of the regulating screw,

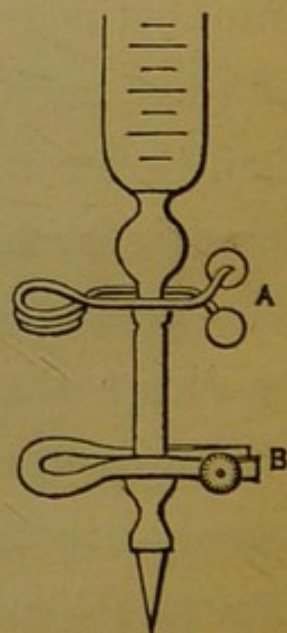


Fig. 9.

can be adjusted to give drops of the required sizes, and at certain intervals. This last is useful in the assay of silver coins and other cases, when small quantities of test are used which require to be of a determined size.

PIPETTES, for measuring small quantities of solutions, &c., are of two kinds—such as deliver one fixed quantity of liquor, or such as, being tubular and graduated from end to end, measure various quantities. We may distinguish these two forms as *bulb pipette* and *scale pipette*. The chief use of a pipette is to measure with precision

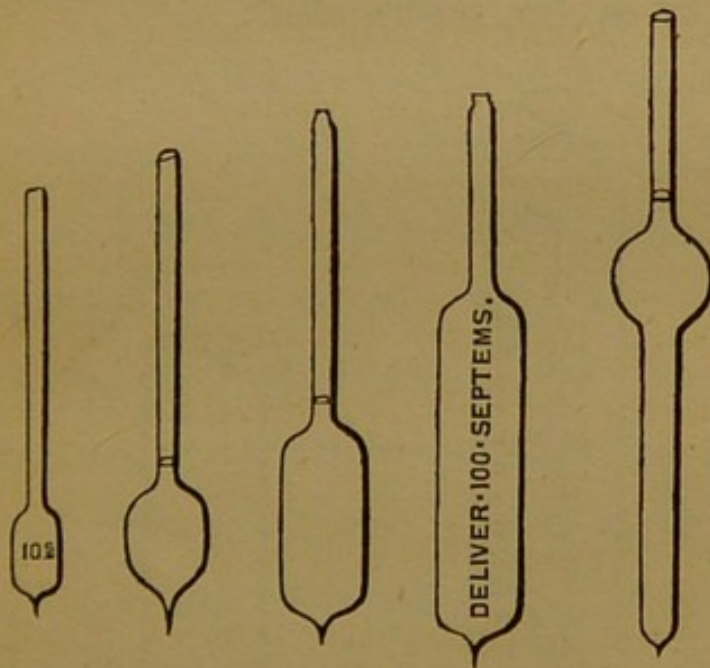


Fig. 10.

that quantity of a solution which is to be subjected to volumetric analysis, or that dose of a graduated test liquor which is required for a

specific operation. The picking and management of a pipette requires a few observations. The lower opening, from which the liquor escapes, should not be more than one-twenty-fifth of an inch ( $= 1$  millimètre) in diameter. The upper end should be closed by the wet finger, and the pressure relaxed as the liquor is wanted, until the desired point be reached.

Fig. 10 represents the bulb pipettes, or those which serve to deliver one quantity.

SCALE PIPETTES, which are graduated from end to end, and can deliver any given quantity within the capacity of the instrument.

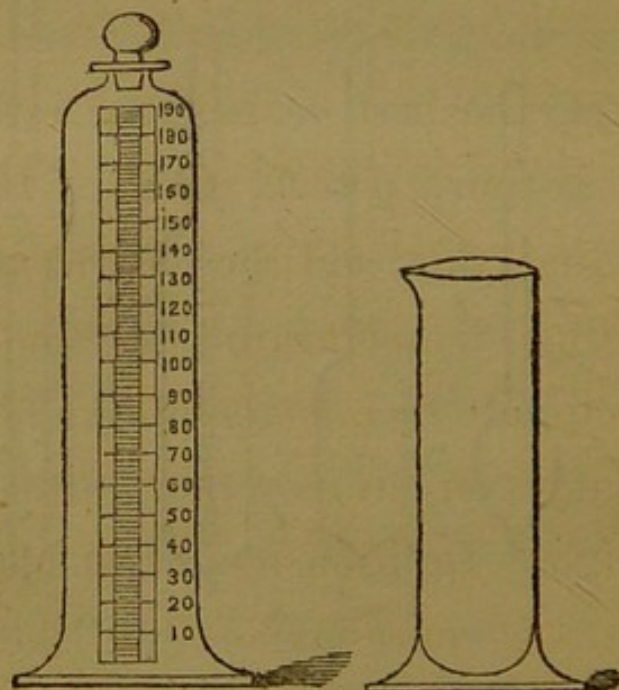


Fig. 11.

TEST MIXERS are tall cylindrical jars with

broad feet, and are used for preparing test acids, and differ from the measuring flask in the fact that in the latter a weighed equivalent of a solid chemical is dissolved in water and diluted until it forms a standard measure, such as a litre, a décigallon, or 100 décems, whereas in the latter an equivalent of a dissolved chemical is diluted from a standard measure. Test mixers are represented in fig. 11.

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### TESTING THE BALANCE, AND WEIGHING.

In Quantitative Analysis it is of the first moment that the balance should be correct to the least amount. A good and practically useful balance should turn very distinctly with a weight of 1 milligramme, or 0.0154 grain. If there is any little variation in the weights required for either scales to weigh a certain quantity, it must be regulated by means of small pieces of tinfoil (not paper, as that will absorb moisture).

The following experiments should be made to test the scales:—

1st. Load both scales with the greatest load

they are constructed to carry, the balance then carefully adjusted, and a milligramme added to one scale-pan. This ought to turn the beam to the same extent as if the scales were empty. In most cases, however, it will show somewhat less on the index.

2nd. When both scales are loaded and in equilibrium, the weights are changed, so as to transfer that of the right scale to the left, and *vice versâ*. If the balance is true, and has perfectly equal arms, this will not affect its stability.

3rd. When a balance is disturbed from a state of rest, and made to oscillate, it should always return to its original position and equilibrium.

The balance must stand all these tests, though the least important is a slight variation in the length of the arms, as it does not interfere with the accuracy of the weighing. To keep the edges of the balance free from rust, it is a very good practice to place inside the case a beaker half-filled with sulphuric acid;  $\text{SO}_2\text{Ho}_2$ , or  $\text{BaCl}_2$ , baric chloride. The balance should not be kept in the laboratory.

In weighing, any small variation in the lengths of the arms of the balance will not inter-

ferre with the correct results, because, as a rule, we have always one scale-pan for the weights and another for the substance to be weighed; but of course any perceptible error must not be allowed to pass.

The following considerations must be attended to :—

1st. If the substance to be weighed is liable to attract moisture, it should be weighed in a closed vessel, or between two watch-glasses.

2nd. No substance should be weighed whilst hot, as the ascending currents of air caused by the heat of the body under examination will cause the apparent weight to vary every instant.

3rd. In weighing, the weights should be added with care and judgment, not placing a great weight on at first at a venture.

4th. The same plan as is recommended for the qualitative examination of a substance should also be strictly carried out in weighing and all operations connected with the quantitative analysis of a substance—viz., that every operation and step should be carefully noted down in a note-book. The weight of the vessel should be entered, together with the substance weighed, and

then the weight of the vessel subtracted, to leave the real weight of the substance.

It is sometimes convenient to weigh by the method of substitution, and this method will generally give very true results, and is used where great accuracy is required, as in the determination of atomic weights. The process is conducted as follows: the material to be weighed is placed in one of the pans, and weights or a counterpoise in the other, just to equipoise the beam. The material is then removed, and the equilibrium of the balance restored by substituting weights for the removed substance. The weights, it is perfectly evident, must be exactly that of the material to be weighed. In practice the process is materially shortened by adding a tare (or weight) in one pan, and then placing weights with the substance under examination in the other. The weight of the tare, minus the extra weight required to be placed with the substance, equals the true and required weight.

The weights should be carefully tested by placing a tare or counterpoise in one pan, and the weight to be tested in the other, until there is just equilibrium; then replacing the weight with

one known to be correct, and watching to see if there be any variation. In the purchasing of weights, it is invariably the safest way for the analytical chemist to test every weight, no matter how high the reputation of the maker may be. No great importance need be placed in the weights being slightly tarnished, the coating of oxide being so extremely thin it will make no perceptible difference in weighing.

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### ESTIMATION OF WATER.

ESTIMATION OF WATER can be performed in two ways—either by driving off the water and calculating the loss, or by driving off the water and calculating the amount collected. The first method can only be adopted when the substance loses nothing else but water, by the application of heat. Silicates often retain water with such obstinacy that they require the use of the blow-pipe. If the substance loses other parts besides water by the process of driving moisture off, then modifications are resorted to, which will be mentioned subsequently. For the indirect method, or that of calculating the quantity of

water driven off, heat the substance in a current of dry air, and the water collected by chloride of calcium, or pieces of pumice stone saturated with sulphuric acid. Here, again, if the substance loses other things besides water, modifications have to be adopted.

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## ESTIMATION OF THE METALS.

### **Hg.**—MERCURY.

As a metal, in common with most of the others, it is tested for, whenever practicable, by the dry way, or without the mixture of any reagents; 2. In the wet way, or with solutions (tests); and 3. The solution of some of its salts, as the chloride, sulphide, with various reagents, &c.

**IN THE DRY WAY.**—The quantity of mercury contained in a substance may often be determined by separating it from the other constituents by distillation, but requires many precautions, and is not so much to be relied upon as the wet or humid way. The operation should be conducted as follows:—Place the substance into a small retort or combination tube 50 in. long, mixed

with a little pure lime and potash; this being done, draw the neck of the retort into a long thin spout, then introduce this into a vessel of water. If the retort be very small, the distillations may be carried on over a Bunsen flame; but when practicable, it is the best to use a small furnace. The mercury will volatilize over and condense at the bottom of the receiver in the water; the tube is then cut off near the body of the retort, and the globules of mercury collected into a large one by agitating the flask. Decant the clear liquor, and pour the mercury into a weighing flask; dry the mercury by blotting paper, and then dry in the air—heat should not be employed.

To analyze the iodide of mercury, the lime is replaced by finely divided copper, and if the mercury be the sulphate, the calcic and sodic carbonates are replaced by magnesite. The accuracy of the method is entirely dependent on the care bestowed. Marchand receives the mercury in a bulb apparatus containing gold-leaf.

IN THE HUMID OR WET WAY.—The solution must be first tested for nitric acid,  $\text{NO}_2\text{Ho}$ , which, if present, must be got rid of; then add  $\text{HCl}$ ,

hydrochloric acid, and  $\text{SnCl}_2$  stannous chloride; boil for some time, and allow to cool. After cooling, the clear supernatant liquor is decanted from the globules of mercury, which are washed with slightly acidulated water, and dried as in the dry way.

*Test Analysis.*—2 grms. HCl give 73 to 73.88 per cent. of metal.

If the black precipitate will not unite into metallic globules, boil, after decanting the supernatant liquid off, with HCl acid. This process becomes uncertain if much free  $\text{NO}_2\text{Ho}$  be present. If this is found to be so, by test 130, page 114, HCl should be added, and the solution concentrated by heat, till no more fumes of HCl or Cl are given off.

It is sometimes convenient to use phosphoric acid, as its action is not interfered with by  $\text{NO}_2\text{Ho}$ , and the globules of mercury are much larger. In fact, if it were not for the difficulty of obtaining it, we should always use phosphoric acid in preference to stannous chloride.

**MERCUROUS AND MERCURIC SALTS.**—If the mercury be required as mercurous salts, add sodic chloride, and dry the precipitate in a temperature

of  $100^{\circ}$ ; if as a mercuric salt, add, in addition to sodic chloride,  $\text{SO}_2\text{Feo}''$ , ferrous sulphate, then  $\text{OK}_2$ , potassic oxide (potash), and dilute  $\text{SO}_2\text{Ho}_2$ ; agitate occasionally till the precipitate has turned white—that is, till the mercurous oxide is completely changed into the chloride; then filter and weigh as before.

**MERCURIC OXIDE.**—Place in a bulb-tube one end of which is drawn out to a point, and allow dry air to pass over the heated oxide until it is quite decomposed; then calculate as given in the general dry method.

### **Pb.**—LEAD.

**AS METAL.**—By ignition in hydrogen.—Reduce the compound in a current of hydrogen, minding that the heat is not too great. The metal substance, with the exception of the sulphide, may be fused with cyanide of potassium. Ignite cautiously in a porcelain crucible till the weight remains constant.

**AS OXIDE.**—By Precipitation as Carbonate.—Warm the solution gently, add carbonate of ammonia, and then ammonia slightly in excess; wash dry, remove the precipitate as completely

as possible to a porcelain crucible, and ignite. This generally gives a trifle too low results, owing to the carbonate of lead not being completely soluble. Rose recommends the use of oxalate of ammonia as the best reagent for precipitating lead from its solutions.

AS CHLORIDE.—The solution is mixed with hydrochloric acid, HCl, in excess, and the precipitate washed with alcohol and ether, and then dried at a temperature of  $100^{\circ}$ , or ignited. In the latter case a porcelain crucible must be used.

### **Ag.**—SILVER.

AS a METAL.—Silver is more easily separated from other substances, and its quantity quantitatively estimated, than most metals. As a metal it can be ignited in a porcelain crucible; and in the wet way, it can be precipitated by HCl (hydrochloric acid), which precipitates an insoluble chloride in water which must not have the presence of ammonia (see page 82, note 14). Chloride of silver must be washed on as small a filter as possible, and after washing must be dried with great care, and introduced into a porcelain crucible, and the filter burnt within the crucible.

When the chloride is thoroughly dry, an ejection often takes place, occasioned by the silver imbibing oxygen, and then allowing it to escape (called occultating). This may cause some loss. According to M. Mohr, chloride of silver may be reduced to metallic silver by ignition with resin. Chloride of silver is mixed with half its weight of resin, and moderately heated in a crucible until the flames cease to have a greenish-blue colour, and the reduced silver is found as a button at the bottom of the crucible.

When the operator wishes to determine the quantity of silver contained in a certain alloy, we generally make use of the method of *cupellation*, the essential object of which is to oxidize all the baser metals, and leave the silver unacted upon. This method, although not giving strictly accurate results, is often sufficient for various technical purposes. The quantity of the other metals contained in the alloy cannot be thus estimated.

According to MM. Haidlen and Fresenius, silver may be separated from lead by cyanide of potassium. Solution of cyanide of potassium is added to that of the two metals, and heat applied. The lead remains as a residue, and the silver is

reduced in the state of double cyanide of potassium, which is decomposed by nitric acid; nitrate of potash is formed, and cyanide of silver is precipitated, and from this the weight of silver is calculated.

### **Bi.**—BISMUTH.

The estimation of bismuth is directly performed by fusing in a porcelain crucible one part weight of the substance with four or five of cyanide of potassium. Treat with water, and wash the metallic particles with water, then alcohol, and dry.

The best reagent for precipitating bismuth as the carbonate is to add ammoniac carbonate in a very slight excess; heat for some time, nearly to boiling; filter, dry the precipitate, transfer it as complete as possible to a porcelain crucible, and ignite. The solution must be free from hydrochloric acid, or else we shall have some portion of the bismuth precipitated, as the chloride; or if HCl, hydrochloric acid, is present, the bismuth should be precipitated as a sulphide, which can be done by adding hydric-ammoniac sulphide, or sulphuretted hydrogen. Then put the precipitate and filter together in a beaker; add  $\text{NO}_2\text{Ho}$  (nitric

acid); heat gently; dilute, filter, and wash the filter with very dilute nitric acid, then add ammoniac carbonate as before.

We can also precipitate bismuth as the chromate by placing it in a beaker containing a warm solution of bichromate of potassium. The reagent must be added till the precipitate assumes an orange-yellow colour, and dense throughout. Then boil whilst stirring, and wash the precipitate by repeated boiling with water; then decant into a weighing filter.

### **Cu.**—COPPER.

Copper can be estimated by the test 27, page 85, by placing a piece of pure zinc in a solution of copper contained in a platinum vessel, and adding HCl (hydrochloric acid), meanwhile regulating the evolutions of hydrogen by the addition of water. After the evolution of the hydrogen has ceased, add a little more hydrochloric acid, and then, if no more hydrogen is produced, collect the copper (which will be in part a spongy mass, and in part adhering to the sides of the vessel) together, wash it with water till free from hydrochloric acid, and dry in an oven. If this

operation be performed in a porcelain vessel, all the copper will be obtained in the spongy form, but it will take considerably longer.

By direct precipitation as an oxide, first heat the liquor in a porcelain dish, and then add pure  $\text{OK}_2$  (potash). The whole is then allowed to boil moderately for some minutes, and the precipitate, which is of a heavy, brownish-black colour, separated through a filter, and washed with hot water *thoroughly*, till a few drops of the washings give no residue when evaporated to dryness. Finally, ignite intensely, and weigh as *soon* as cool in the crucible used, which must be kept well closed throughout the experiment, because without great precautions it attracts moisture, which interferes with the results. If the solution, however, be *ammoniacal*, the precipitate must be weighed whilst hot, as if allowed to cool a small portion of the precipitate would re-dissolve.

The method of separating copper from its solution, test 26, page 85, is not to be relied on for a quantitative analysis, as the copper is changed whilst drying in the oxide. It is also contaminated with impurities which the iron leaves on dissolving.

To precipitate as a sulphide, add sulphuretted hydrogen; filter off and wash the precipitate with water containing a little sulphuretted hydrogen; dry, and ignite over the blow-pipe.

### **Cd.**—CADMIUM

Is best precipitated as an oxide from its solution by the addition of  $\text{ONa}_2$ , or  $\text{OK}_2$ , sodic or potassic oxide. A white precipitate is formed, which is dried and ignited, during which it evolves water and carbonic anhydride, and the oxide remains in the form of a brown powder. Reducing gases and carbon must be carefully avoided, as they reduce this precipitate (see test 31, page 86).

Cadmium can also be estimated as the sulphate by the use of sulphuretted hydrogen; then dry the precipitate, and weigh.

### **Sn.**—TIN.

If tin be combined, in the metallic state, with other substances, the method best employed to determine its quantity is to boil with  $\text{NO}_2\text{Ho}$ , nitric acid, which converts it into an oxide of tin; when all action has ceased, wash, filter, ignite and weigh.

Tin is also precipitated by  $\text{NO}_2\text{Amo}$ , ammoniac nitrate and sodic sulphate,  $\text{SO}_2\text{NaO}_2$ , as stannic hydrate; allow the precipitate to settle, and wash by decantation *thoroughly*; then collect the precipitate, dry, and ignite.

Or we may completely separate tin as sulphate by saturating the solution with sulphuretted hydrogen, then allowing to stand, and filter. Sulphuretted hydrogen gives two coloured precipitates in solutions of tin (see tests 32 and 36, pages 87 and 88), according to whether the tin be in the stannic or stannous form. Stannic sulphide is inclined to pass through the filter, and should be washed with a strong solution of sodic chloride, or ammoniac acetate containing a little acetic acid. Dry the precipitate, and then heat in an open porcelain crucible until all the odour of sulphur is given off. Finally, ignite strongly, adding ammoniac carbon towards the end.

Tin can also be estimated by the oxidation of stannous chloride, but the operation is attended with peculiar difficulties, for the stannous chloride absorbs oxygen from the air, and also water used for its dilution. The energy of the oxidation varies greatly with the amount of acid present.

**As.**—ARSENIC.

The best way of estimating arsenic is to obtain an arseniate of lead; but if arsenious acid is present, it must be changed into arsenic acid by adding *aqua regia*, and concentrating by evaporating to a small bulk. The method of adding *aqua regia* is preferable to that of nitric acid, which is sometimes done. Then add a weighted quantity of oxide of lead, evaporate to dryness, and heat to gentle redness. The residue consists of plumbic arsenite and oxide of lead, or of oxide of lead and arsenic oxide combined. Subtract the weight of the plumbic oxide, and the remainder equals that of the arsenic acid from which the quantity of arsenic present is calculated.

If the solution contains HCl, hydrochloric acid, the acid must be removed, or else the arsenic precipitated with sulphuretted hydrogen; the arsenic sulphide thus obtained is collected upon a filter, washed, dried, and then weighed. It is extremely necessary in this operation to take care to ascertain if there is any undissolved sulphur left in the solution or with the filter, as this will

very materially alter the calculation. This process is rather difficult to perform.

ESTIMATION BY STANDARD BICHROMATE OF POTASSIUM.—Arsenious acid is oxidized by the addition of the solution of bichromate of potassium, and the excess estimated by ferrous sulphate. The solutions are prepared as follows:—

*Bichromate of potassium*, 3 grms. in 1 litre of water.

*Ferrous sulphate*, 42 grms. in 1 litre of water.

*Arsenious acid*, 5 grms. in solution of slightly acidulated  $\text{OK}_2$ , potash, the acid to be of 1.12 specific gravity, and dilute to 1 litre.

### **Sb.**—ANTIMONY.

Evaporate carefully in a platinum vessel, having before added  $\text{NO}_2\text{Ho}$ , and then ignite.

For the precipitation as a sulphide, add hydrochloric acid, then tartaric acid, after which water may be added as required; but it is advisable to add the tartaric acid first, as otherwise water precipitates antimony from its solution. Then precipitate with sulphuretted hydrogen, and warm gently. Collect the precipitate on a filter, wash with water containing a little sulphuretted hydrogen, and then dry. If the precipitate con-

tains any sulphur, extract it by the aid of carbonic bisulphide (bisulphide of carbon). Transfer to a porcelain crucible, add nitric acid, and evaporate. The sulphur is completely oxidized during the operation. The white residue consists of antimoniac and sulphuric acids, and must be converted into the binoxide by ignition.

### **Au.**—GOLD.

Gold is best estimated by ignition in a porcelain crucible, very gently at first, and afterwards to redness; or by first precipitating with sulphuretted hydrogen, filtering, drying, and then igniting the precipitate.

Or by *precipitation with ferrous sulphate*.—Add to the solution a little hydrochloric acid, and then a clear solution of ferrous sulphate. Heat for some time, gently filter, wash dry, and ignite. We may also use oxalic acid, but this requires some considerable time.

### **Pt.**—PLATINUM.

By ignition, before or after the addition of sulphuretted hydrogen, and by the addition of ferrous sulphate.

As chloride of platinum, we can alter the precipitate by adding potassic chloride and alcohol, allowing to stand, and then drying and weighing. Ignite in a current of hydrogen, allow to cool, and then wash the reduced platinum cautiously by decantation. Finally, dry the tube and weigh.

### **Cr.**—CHROMIUM.

By igniting in a platinum crucible, and applying heat until the weight is constant. Add ammonia slightly in excess, and boil till the supernatant fluid loses its colour. Then wash the precipitate, dry, and ignite in a covered crucible.

If the chromium exists as the acid, it may be precipitated as chromate of baryta or lead, by adding nitrate of lead or baryta, or it may be changed into the oxide by a current of sulphuretted hydrogen. It is necessary, after having passed the current of sulphuretted hydrogen through the liquor, to warm and leave it to settle; the sulphur will settle off.

### **Al.**—ALUMINIUM.

By ignition as before.—By precipitating from its solution with carbonate of ammonia, and then

ammonia, afterwards apply heat to gentle boiling, until the steam ceases to brown turmeric paper. Allow the precipitate to settle, wash well, and then ignite. The heat should be very gentle at first, and the crucible kept well covered to guard against spilling and consequent loss of the substance. Towards the end, the heat may be increased to redness.

### **Fe.**—IRON.

The substance is heated gently in a covered crucible, and then ignited in the blow-pipe flame.

Iron is generally determined in the ferric condition, and hardly ever, with the exception of the ferrous sulphate, estimated in the ferrous condition; and if it originally is in the ferrous form, it ought to be changed into the ferric by adding strong nitric acid, which will at first produce a black colour, but on continual heating, with addition of acid, it will turn pale yellowish-brown, with considerable effervescence. Add ammonia in excess, wash the decantation, transfer the precipitate to the filter, dry *thoroughly*, and ignite the precipitate with the filter ash.

Iron may also be determined in the dry way by means of cyanide of potassium. The ore is first reduced to the state of a fine powder, and mixed with a mixture of cyanide of potassium and carbonate of potash (potassic carbonate), and then the whole exposed to a strong red heat in a porcelain crucible. The silica and albumina remain in the scoriæ, but the reduced iron may be separated by washing with cold water. It may now be weighed.

*As a basic ferric acetate or formate.*—Add ammonia till the solution has acquired a deep-red colour, then sodic acetate. Wash with hot water *thoroughly*, which may contain a little ammoniac acetate; then dry, and weigh. The residue should give no alkaline reaction.

### **Co.**—COBALT.

Ignite in a current of hydrogen till the weight remains constant, and then add hydric-ammonic sulphide, the solution being slightly ammoniacal. The precipitate, which is black, must be well washed with water containing a little hydric-ammonic sulphide, dried, and then digested with nitric acid until nothing remains but the sulphur.

Then dilute with water, filter, and the filtrate precipitated by  $\text{OK}_2$ , potassic oxide. The oxide thus obtained is ignited and weighed.

*Yellow Nitrate of Cobalt.*—Add a strong solution of potassic oxide, and then acetic acid—just a little more than to dissolve the precipitate produced at first by the potassic oxide on the carbonate. Collect the precipitate on a filter, and wash thoroughly with solution of potassic acetate, and then alcohol; dry thoroughly, and weigh.

### **Ni.**—NICKEL.

By ignition, in the absence of all reducing gases.

As for nickel, the best reagent for estimating it as a metal is pure potash,  $\text{OK}_2$ . Heat the solution to boiling, then add the potash, and keep near the boiling temperature for some time. Wash the precipitate *thoroughly*, until a few drops leave no residue on evaporation, after which ignite and weigh. Ignition renders it black, and it then consists of pure oxide of nickel. The weight is not altered by ignition and cooling. The use of hydrogen is therefore not required, as with cobalt.

*As Sulphide.*—Place the solution in a flask, and if necessary neutralise it with ammonia. Then add ammoniac carbonate and hydric-ammoniac sulphide in slight excess. Wash the precipitate with water containing a little hydric-ammoniac sulphide. (The filtrate and washings should be either colourless or slightly yellow.) Dry the precipitate, and remove it as completely as possible to a beaker. Incinerate the filter, and add the ash to the precipitate. Then add *aqua regia* until the sulphur is separated (or the precipitate has lost its black colour). Dilute, filter, and precipitate by potassic hydrate.

### **Zn.**—ZINC.

By Ignition.—Place the substance in a covered platinum crucible, apply gentle heat, and afterwards very intense, until the weight remains constant. The action of reducing gases must be avoided.

The best reagent for precipitating oxide of zinc is the sodic, or potassic carbonate, which should be added when the solution is nearly boiling. It is preferable to use the sodic carbonate, because potassic carbonate generally contains small

quantities of silicic acid. The precipitated zinc carbonate is placed on the filtrate, and washed well with hot water. Dry, ignite, and weigh.

*As Sulphide.*—To the solution in the flask add (ammonic-chloride) sal-ammoniac, ammonia, and then hydric-ammonic sulphide. Slightly warm, and allow to stand for some time. Filter and wash the precipitate with water containing a little hydric ammonic sulphide. Then digest along with the filter in concentrated hydrochloric acid, which dissolves it under disengagement of sulphuretted hydrogen until odour of sulphuretted hydrogen has ceased to be evolved. The liquor is then filtered, and the oxide of zinc precipitated by sodic carbonate. Or we may, in place of this method, ignite with sulphur, after adding the hydric-ammonic sulphide in a current of hydrogen, raising the heat gradually to intense redness.

### **Mn.**—MANGANESE.

By precipitating as a carbonate by sodic carbonate is the best method. This should be added to the solution when nearly boiling. Then filter and well wash by decantation; afterwards dry and ignite in an open crucible till the weight

remains constant, care being taken to exclude reducing gases. Now heat with hydrochloric acid; afterwards, dry and weigh.

*As Sulphide.*—Add sal-ammoniac (ammonic chloride), and ammonia, and then hydric-ammonic sulphide. Filter and wash the precipitate with water containing a little hydric-ammonic sulphide. Finally, dry and ignite with sulphur in a current of hydrogen.

### **Ba.**—BARIUM.

By adding pure sulphuric acid to a solution in a platinum dish, and evaporating to dryness, expel the excess of sulphuric acid and ignite the residue, and weigh.

BY PRECIPITATION.—*As Sulphate.*—The solution must not be very acid. Add sulphuric acid, and keep for some time near the boiling point. Filter, wash, expose to a low heat, and weigh.

*As Carbonate.*—Add ammonic carbonate, afterwards a little ammonia, and then gently heat in a sand bath. The carbonate thus obtained is washed with a *small quantity* of water, dried, ignited, and weighed. This second method is not so much

recommended as the first, as it does not give such exact results.

**Sr.**—STRONTIUM.

The best method is by evaporation, or, after adding sulphuric acid slightly in excess, evaporate and expel the sulphuric acid by cautiously heating, and ignite the residue. The method of washing with water cannot here be applied except with great restriction, because the strontic sulphate is partly soluble in water, and if the precipitate be required, it should be washed with dilute alcohol.

**Ca.**—CALCIUM.

This may be estimated in the same manner as strontium, or we may add oxalate of ammonia or oxalic acid to the solution. The precipitate, which is the oxalate of lime, calcic oxalate, takes a considerable time to settle, and should remain at rest for twelve hours; and as oxalate of lime is soluble in acids, care must be taken that the solution is throughout slightly alkaline. The precipitate is then ignited in a platinum crucible, upon which the oxalate burns with a

blue flame, owing to the reduction of the oxalate into the carbonate. After the blue flame has ceased, weigh; then moisten with a few drops of solution of sal-ammoniac, again ignite, and re-weigh. If this second weighing agrees with the first, it may be taken; if not, proceed as after the first weighing.

### **Mg.**—MAGNESIUM.

Ignite the substance, then carefully pour a little sulphuric acid, and then evaporate the whole mass to dryness. The residue, which consists of sulphate of magnesia, is weighed and calculated.

Also we may precipitate magnesia from its solutions by adding sal-ammoniac, or ammoniac carbonate, and allowing the mixture to stand for twelve hours. This precipitate, although long in forming, is *nearly* insoluble, but should not be washed *too* long. Then dry and ignite in a covered crucible, as the precipitate will on ignition deflagrate.

### **Na.**—SODIUM.

To the substance add sulphuric acid in slight excess, filter, dry, and ignite, adding ammoniac

carbonate towards the end of the experiment. The precipitate will be the sulphate.

*Nitrate.*—Add excess of nitric acid, and then fuse the precipitate after well drying.

*Chloride.*—Add excess of hydrochloric acid, and heat thoroughly in a closed vessel to dryness, bearing in mind that if sodic chloride is not thoroughly dried before ignition, it will decrepitate, and there will be danger of losing part of it. Intense heat must, however, be avoided, as it will cause the chloride to volatilize.

### **K.**—POTASSIUM.

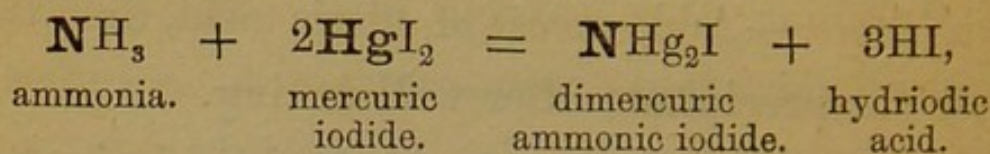
The estimation of potassium is performed exactly similar to sodium.

### **Am.**—AMMONIUM.

*As Chlorides of Ammonium and Platinum.*—To the solution add HCl, hydrochloric acid, and platinic chloride in excess; evaporate nearly to dryness. Add alcohol; wash thoroughly with alcohol, dry thoroughly, and weigh.

*Nessler's Test.*—When a solution of mercuric iodide in potassic iodide is added to a solution containing free ammonia or an ammonical salt,

to which caustic potash has been before added, a brown precipitate or colouration is produced.



And the unknown quantity of ammonia is estimated by imitating the tint by a known quantity of ammonia. The test cylinders must be colourless, and of equal shape and size. Also the distilled water must be carefully tested to prove the absence of ammonia, and always allowed to stand for a few minutes before deciding whether the tests are equal or not. When the solution contains more than one mgrm. in 100 c.c. of water, the tint becomes too dark for estimation.

### **Pd.**—PALLADIUM.

*As Sulphide.*—Precipitate the acid solution with sulphuretted hydrogen. Wash the filter with hot water, and ignite over the blow-pipe.

*As Chlorides of Palladium and Potassium.*—Add potassic chloride and nitric acid; then evaporate to dryness, allow to cool, and wash with alcohol; dry, and weigh. This is not so much recom-

mended as the separation in the form of a *sulphide*, because traces of the precipitate are apt to be carried away in the washing.

**Li.**—LITHIUM.

*As Sulphate.*—Add excess of sulphuric acid, and ignite.

*As phosphate.*—Add pure sodic phosphate and ammonia; warm gently, and then digest at a gentle heat; allow to stand for some time; filter, dry thoroughly, and weigh. This process will always give results under the true value, and is not to be recommended.

**Mo.**—MOLYBDENUM.

Ignite at a gentle redness in a current of hydrogen, till the weight remains constant.

**U.**—URANIUM.

Ignite at a gentle red heat after the addition of a little ammoniac carbonate.

**Ti.**—TITANIUM.

Add ammonia; wash the precipitate well, dry, and ignite, after adding a little ammoniac carbonate. The precipitate is slightly hygroscopic.

## ESTIMATION OF THE NON-METALS.

**S.**—SULPHUR.

Digest in fuming nitric acid or *aqua regia*, to convert it into sulphuric acid as much as possible. The remaining sulphur is collected, weighed, and estimated with that in the sulphuric acid formed.

Or we may mix with potassic nitrate and sodic carbonate, and then fuse. The fused mass is treated with hot water, and then dissolved in hydrochloric acid and treated as above.

**P.**—PHOSPHORUS.

Add a quantity of recently ignited oxide of lead, evaporate to dryness, and then heat gently to dryness, the residue consists of plumbic phosphate, and plumbic oxide, and subtract the weight of the oxide of lead, estimate the phosphorus. We may also estimate it as pyrophosphate of magnesium, but this requires a very considerable time.

**Si.**—SILICON.

*As Silicic Acid.*—Place the substance in a platinum dish, treat with nitric or hydrochloric

acid, and then digest at a gentle heat, at the same time constantly stirring with a glass rod. This is sometimes a lengthy operation. The silica separates either in the gelatinous or pulverulent form. Evaporate to dryness, and dry the residue thoroughly, breaking up the lump as it proceeds. Moisten with hydrochloric acid, and then wash the precipitate well; dry, and ignite over the blowpipe.

*By fusion with Baric Carbonate.*—Add about three parts for one of the substance, &c., then fuse at a moderate red heat. Allow to cool, transfer to a beaker, add a considerable quantity of water and some hydrochloric acid, warm, and allow to stand. Then filter, evaporate to dryness, moisten with hydrochloric acid, warm, dilute, and wash the precipitate; dry thoroughly, and ignite over the blow-pipe.

We may also fuse with potassic carbonate or baric hydrate.

### **C.**—CARBON.

In order to determine the quantity of carbon contained in a substance, the carbon is first converted into carbonic anhydride, and the volume

of this acid determined, or passed through lime-water or baryta-water, the quantity of the carbon is estimated from the earthy carbonates. This is especially the case if volatile bodies are present in the original substance.

### **B.**—BORON.

The estimation of boron is attended with several difficulties. Boric acid cannot be determined by evaporating to dryness, because a portion always volatilizes along with the aqueous, and especially if alcohol be used. Also there is no method of determining immediately the quantity of boric acid in solution by precipitating. In order, therefore, to estimate, we have to determine what quantity of the base or substance is combined with the nitric acid, and the quantity of the acid is estimated from the loss.

### **F.**—FLUORINE.

The best way for determining fluorine is to weigh a certain weight of the substance, and then pour concentrated sulphuric acid upon it, heating till the whole of the fluorine has been expelled,

and likewise the excess of the sulphuric acid, and then calculating accordingly. This experiment must be performed in a platinum crucible.

### **Cl.**—CHLORINE.

If the substance is in solution, argentic nitrate should be used, and the chlorine calculated from the resulting argentic chloride. If the chlorine is in the free state, and we wish to estimate it, it must be passed into water containing ammonia, and care taken that no gas escapes. Then calculate the resulting ammoniac chloride.

### **Br.**—BROMINE.

This is estimated in exactly the same way as chlorine.

### **I.**—IODINE.

This is also estimated similar to chlorine and bromine.

### **N.**—NITROGEN.

The estimation of nitrogen in the state of a gas only occurs when it has to be separated from other gases. Generally we absorb the other gases

with which it may be accompanied by various reagents, and then determine its volume.

*As Nitric Acid.*—Boil the nitrate with excess of standard potash, dilute to a definite bulk, allow to settle, draw off a portion, and titrate back with standard acid.

Or we may convert it into nitric oxide, and then estimate. This is done in the estimation of nitrogen in water.

## **H.**—HYDROGEN.

The estimation of water in which hydrogen is contained is given on page 139, and we can always estimate dry free hydrogen by changing it into water.

## **O.**—OXYGEN.

This is estimated in the same method as hydrogen.

## DIALYSIS.

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THE process of analysis discovered by Thomas Graham, Esq., Master of the Mint, and described in his memoir on "Liquid Diffusion Applied to Analysis," which appeared in the report of the "Transactions of the Royal Society" for 1861, page 183, may often be conveniently applied for the separation of arsenic, arsenious acid, metallic salts, strychnine, and other poisons, mineral and organic, from the organic solutions they may be contained in.

The dialyser, or apparatus for effecting analysis by diffusion, consists of a species of sieve, having sides and top of gutta-percha and bottom of parchment.

"The dialyser serves also for the separation and decomposition of many metallic salts; for the separation of urea and other crystallisable salts from urine; for the separation in general of all

*crystalloids* or crystallisable bodies from all *colloids* or gelatinous bodies; and for the preparation and purification of such colloids as hydrated silicic acid, hydrated alumina, gelatine, albumen, starch, caramel, tannin, gummie acid, &c. In short, the dialyser is an instrument of great practical utility to all chemists, physiologists, &c., who are engaged in researches into the composition of organic or mixed liquors."—*Griffin*.

In using the dialyser, the parchment paper is stretched over the bottom of the gutta-percha vessel and tied with a string above the flange

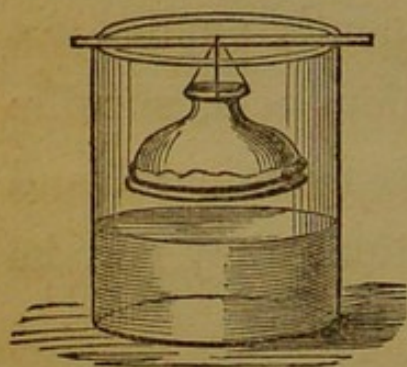


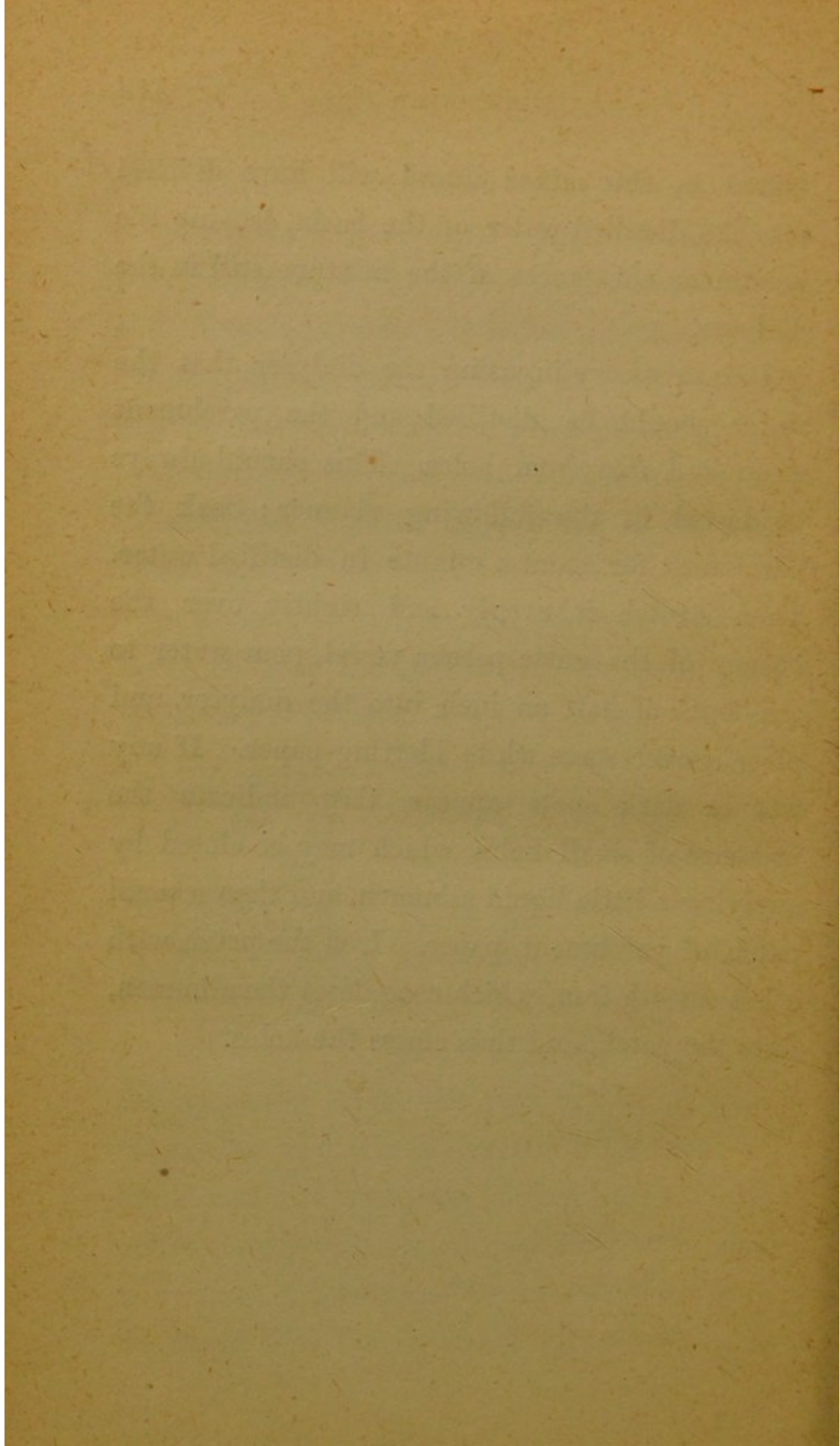
Fig. 12.

which is always left at the bottom. The vessel is then suspended over a glass rod by string tied round the neck. The height of the dialyser in the cylindrical jar must be so arranged that the water in

the jar must cover the face of the parchment paper, but not rise above the outer edge. The liquid under examination having been introduced under the above condition, the dialyser is allowed to remain for twenty-four hours, when it will be found that the crystallisable substance con-

tained in the mixed liquid will have diffused into the distilled water of the basin, leaving the gelatinous substances of the mixture still in the dialyser.

It is necessary in using the dialyser that the water should be distilled and the parchment clean and free from holes. This should always be tested in the following manner: Soak the parchment for about a minute in distilled water. Then stretch it evenly and tightly over the bottom of the gutta-percha vessel, pour water to the depth of half an inch into the dialyser, and place it over some white blotting-paper. If any wet or dark spots appear, they indicate the presence of small holes, which may be closed by applying a little liquid albumen, and then a small patch of parchment paper. Iron the patch with a hot smooth iron, which coagulates the albumen, fixes the patch, and thus closes the holes.



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