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EXERCISES

IN

PRACTICAL CHEMISTRY

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EXERCISES

IN

PRACTICAL CHEMISTRY

BY

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AND

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LONGUM ITER EST PER PRAECEPTA, BREVE ET EFFICAX PER EXEMPLA.

Seneca. Ep. VI.

SERIES I

Qualitative Exercises

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PREFACE.

THE object of this volume is to furnish a systematic course of study to those who are beginning to learn Chemistry practically. The course commences with a series of exercises upon the preparation and properties of some of the most familiar substances, in which precise directions are given as to the apparatus and materials to be used, and as to the manner in which each experiment is to be made. In the earlier exercises, especially, the directions given extend to minute details; the aim of the authors being to provide, as far as possible, all the guidance that a beginner, working by himself or with only occasional supervision, may require. A few exercises have been introduced on parts of manipulation which, from their importance, it seemed desirable to treat separately; but, as a rule, each operation is described where the occasion for its use first arises: and the subjects have been so chosen as to furnish examples of all the usual chemical operations. The attempt has been made to arrange each exercise so as to provide the student with continuous occupation and economise his time by giving two or three operations which can be carried on simultaneously and to which he should direct his attention in turn. The time required for the completion of different exercises cannot but vary considerably, since some operations, too important not to be included, do not admit of being hastened; but in most cases about two hours will be found sufficient.

In Part I, Sect. V, which gives directions for the performance of experiments upon salts of the principal metallic and non-metallic radicles, the operations are described in less detail, both because they are simpler and more uniform in character, and because something may fairly be left to the experience the student has gained. In this section the experiments described are chiefly such as find some application in the course of qualitative analysis which follows. The division into exercises is not continued, since the experiments with each radicle form a separate group, and one or more of these can be performed at the same time, as may be found convenient.

Part II gives the course to be followed in the analysis of a single salt, for the outline of which the authors are mainly indebted to Fresenius' 'Qualitative Analysis.'

The list of apparatus given at the outset shows what it is desirable that a student should have for the performance of the exercises; but those who have not access to a chemical laboratory and who are unwilling to incur the expense of providing themselves with a complete set of apparatus from a chemical dealer, will find in the Appendix a number of suggestions for the construction of pieces of apparatus which can be made very cheaply.

The authors have endeavoured, by repeating nearly every experiment in exact conformity with their written description, to ensure that the student who carefully follows their directions shall command success; but as they cannot but fear that many errors and omissions may, nevertheless, have escaped them, they will be grateful for any suggestions from those engaged in teaching who may make use of their book. It is only by a large experience of the errors into which beginners are liable to fall, that the many ways of going wrong can be discovered and stopped.

The question of chemical nomenclature is at present in such a condition that every lecturer or writer must choose for himself the names he considers least objectionable. The names assigned to substances in this volume are, with a few exceptions (made with a view to consistency), the same as are used in Roscoe's 'Lessons in Chemistry,' and Watts' edition of 'Fownes' Manual[1].' The classification of hydrogen with the metals in the analytical course no longer, perhaps, needs an apology since the publication of Mr. Graham's researches on its combination with palladium.

The illustrations in the text have, with very few exceptions, been drawn directly on the wood from photographs, taken by one of the authors, of the apparatus actually used.

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[1] Sheets of labels for the reagents with which the student is recommended to provide himself may be obtained from Messrs. J. J. GRIFFIN & Sons, 22, Garrick Street, London, W.C.

MEMORANDA.

1. BE orderly and neat in manipulation. Cleanliness stands at the head of the chemist's scale of virtues. All messes must be cleared away with the zeal of a sanitary inspector.

Never go to work, or continue to work, with the table covered with a litter of bottles, flasks, basins, and test-tubes; but replace each bottle on its shelf as soon as you have done with it, and have a basin at hand in which to put dirty test-tubes, &c.

Always wash your test-tubes twice: once, before they are put away; and again, with distilled water, immediately before they are used. Probably more puzzling reactions occur from the use of dirty test-tubes than from any other cause.

2. Do not begin work in a hurry. What is expended in time is very often gained in power, in grasp of a subject. Yet, on the other hand, learn to be economical of time. Several filtrations and evaporations, for instance, may be going on at once. The chemist may sometimes, in spite of the proverb, do more than one thing at a time, by allowing things to do themselves.

3. Be economical of materials. In analysing a substance, do not (without the strongest reasons) use up at once the whole quantity at your disposal. Reserve at least one-fourth of it in a corked tube or covered watch-glass, in case unforeseen accidents should occur and the other portion should be lost. In making a gas, the residue left in the generating vessel will often be of use, at any rate interesting as a specimen. It should not, as a rule, be thrown away, but purified by recrystallisation or otherwise.

4. Never begin an experiment until you have looked over all the preparations for it, to make sure that you have everything that is necessary within reach. You will not then have the mortification

of seeing the half-performed experiment fail for want of some requisite which cannot be procured at the moment.

5. Never add one chemical substance to another without considering for what purpose you add it, and what various effects may be produced.

6. Do not think merely of what will *do*, but what is *best*, of the means at your disposal.

7. Be exact and methodical. Let nothing pass unnoticed, although you may not see its significance at the moment. Make written notes of everything that you do, analyses of lectures, sketches of apparatus. Whatever is worth doing is worth recording.

8. Do not attempt to devise a modification of an experiment until you have tried it in exact accordance with the directions given. Then, and then only, if you fail, you will find it possible to blame the book and not yourself.

9. Do not expose yourself needlessly to vapours which you know to be injurious, e.g. chlorine, hydrogen sulphide, hydrogen arsenide. Remember that the bad effects may not be perceptible immediately.

10. Finally, do not look upon Chemistry as a mere amusement, as a means of getting up a few explosions, creating a few unsavoury smells, producing a few striking changes of colour. Chemistry is worthy of better treatment; it is no longer a 'black art,' but a refined science, and should be thoughtfully and reverently studied.

Nor, again, give up hopes of making discoveries in the science because the land appears to be already highly farmed, and you have not all the refined apparatus which the optician and operative chemist can supply. Records of close and accurate observations of some of the (apparently) simplest phenomena of Chemistry are much needed; and such it is in the power of every student to contribute.

H. G. M.

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PART II.

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LIST OF APPARATUS[1]

Required for the Course of Practical Work contained in Parts I and II of this Book.

1. A Pneumatic Trough, about 36 cm. long, 24 cm. wide, and 16 cm. deep, Fig. 1. This is an apparatus for collecting and experimenting upon gases, and consists of a cistern for holding water or any other fluid, furnished with a movable shelf placed across it about 5 or 6 cm. below the

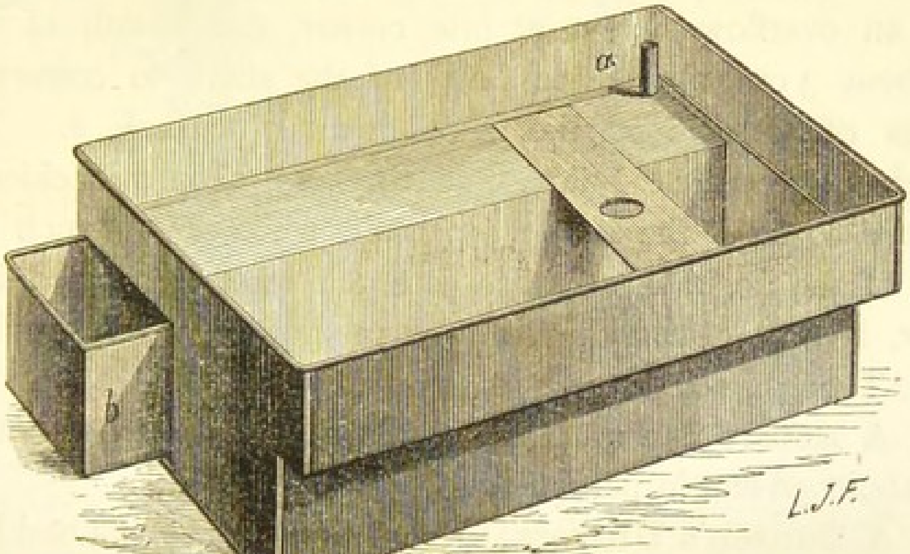

Fig. 1.

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[1] In making this List it has been thought right to err rather on the side of completeness than of deficiency. Some pieces of apparatus, e.g. a Bunsen's holder, are not absolutely necessary; and in several cases the student will be able, by the aid of a few tools and a little ingenuity, to make substitutes for himself. Some suggestions for the construction of economical apparatus will be found at the end of the book, but no direct reference to such substitutes is made in the text, since the student, who is ingenious enough to make a piece of apparatus for himself, will be at no loss to bring it in where it is wanted.

top. In the centre of the shelf is a hole, which should not be less than 2 cm. in diameter, and immediately underneath this hole is soldered a broad, very shallow funnel, the mouth of which is of the same width as the shelf: the accompanying figure represents a section of the shelf at this point. This funnel serves to catch bubbles of gas, in case the delivery tube is not exactly under the centre of the hole, and to direct them upwards through the hole into a jar placed above it. In addition to these essentials, the trough represented in the figure has a ledge running along the whole length of one side on which jars may stand when filled with gas, and also an overflow-pipe *a* at one corner, the mouth of which is about 3 cm. above the level of the shelf, to convey any excess of water into the supplementary trough *b*. These troughs are made of japanned tin, and it is a decided advantage to have the inside japanned white, as a much better view of the position of tubes, jars, &c. when immersed in the water, is thus obtained. For the method of using the Pneumatic Trough, see p. 54.

2. **A Retort Stand**, Fig. 2, with rectangular iron foot, iron rod about 36 cm. in height, and three brass rings.

3. **A Bunsen's Universal Holder**, Fig. 3, of stained birchwood, the smaller of the two sizes which are sold. This, although rather expensive (about 5*s*.), will be found a most useful piece of apparatus, both for chemical and physical experiments. Its construction will be sufficiently evident from the engraving. Care must be taken not to use undue force in tightening the screws, or the threads may be torn away. If the screws work stiffly, a little black-lead, in preference to tallow, should be applied to them.

4. **A Bunsen's Burner**, Fig. 4, small size, with rose top. This burner is a great improvement on the wire-gauze gas-burner, and is constructed on the same principle as the latter, the upright tube representing one of the meshes of the wire-gauze. It gives a blue non-luminous flame, which deposits

no soot on vessels held in it, and possesses a very high temperature, approaching that of the blowpipe-flame. The gas enters through a peculiarly-shaped jet near the bottom of the upright tube, and as it rises in the tube mixes with the air which is admitted by the four holes near the base of the lamp. When a light is applied to the mixture of gas and air

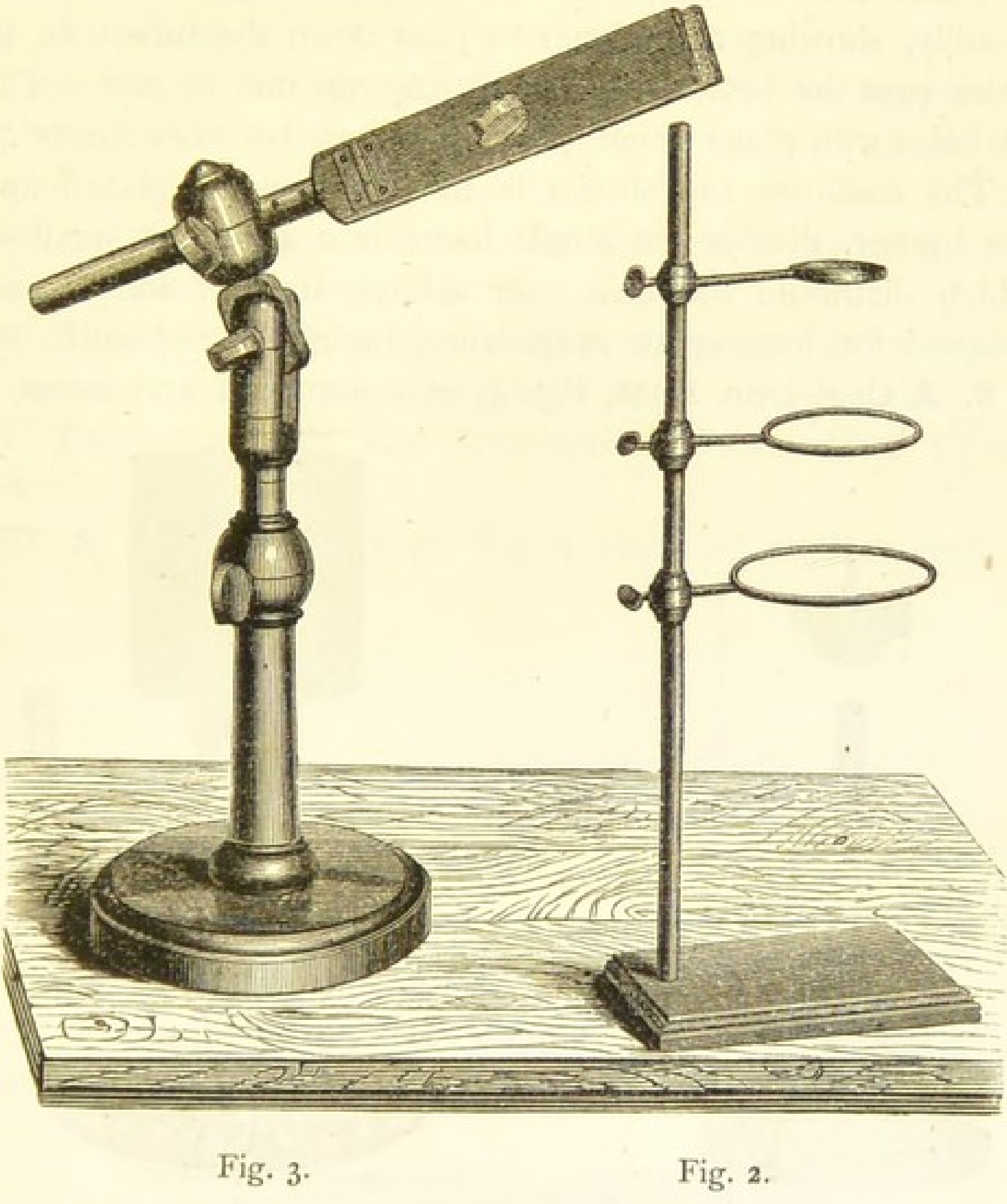

Fig. 3. Fig. 2.

as it issues from the top of the tube, it burns steadily without descending in the tube, so long as the current of gas is uniform and the supply of air is not too great. If, however, the stream of gas is checked, or the supply of air is increased beyond a certain amount, the explosive mixture ignites within the tube, and the gas then burns at the jet near the bottom, with a smoky flame, the heat of which is mainly expended on the tube itself. In fact, the relative proportions of gas and air must be nicely adjusted, or the burner is of little use. If the gas is in excess, the flame burns white, and deposits soot, as may be shown by stopping all the four air-holes with the fingers, when the ordinary luminous gas-flame is produced. If the air is in excess, the flame burns unsteadily, showing a tendency to pass down the tube. In this latter case the best remedy is, to stop up one or more of the air-holes with plugs of cork, until the flame becomes steady[1].

The cast-iron cap shown in the figure, when placed upon the burner, divides the single flame into a ring of small jets which distribute the heat over a large surface, and are well adapted for heating an evaporating basin or sand-bath.

5. **A Cast-iron Foot**, Fig. 5, with stop-cock and screw, on

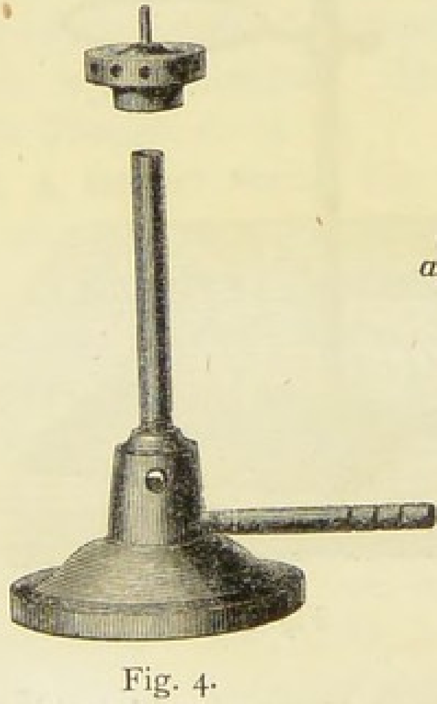
Fig. 4.

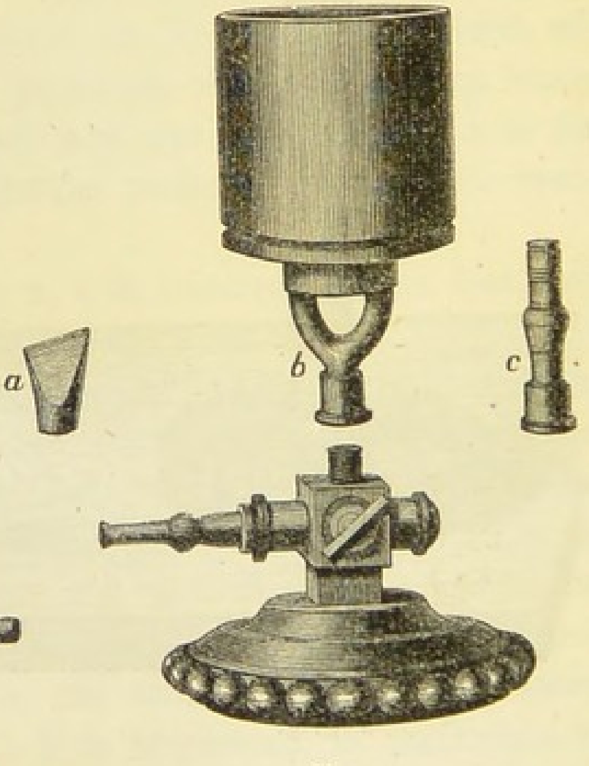

Fig. 5.

which may be fitted the blowpipe-jet *a*, the Argand burner *b*, and the fish-tail burner *c*. The blowpipe-jet is described in

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[1] In the larger sizes of this burner a perforated cap is fitted over the air-holes, by turning which the supply of air can be regulated with great facility.

Part I. Sect. 4. The Argand burner, although not absolutely necessary, is extremely convenient for applying a gentle heat to a flask (e.g. in making oxygen gas), since it is more under control than the Bunsen's burner, and may be regulated to give the least possible flame. The fish-tail burner is chiefly intended for bending tubes, p. 26.

6. **A Set of five Wooden Blocks,** about 12 cm. square, and respectively 2, 5, 7, 10, 12 cm. in thickness.

7. **A Test-tube Stand,** of the usual form, for supporting test-tubes while in use. It should have twelve holes in one row, and a strip of slate should be fitted in front of the holes, on which may be written the contents of each test-tube when it is placed in the stand.

8. **A small Dish,** of tinned iron or copper, about 12 cm. in diameter, for use as a sand-bath.

9. **Two pieces of fine Iron-wire Gauze,** about 12 cm. square.

10. **A Mouth Blowpipe,** Fig. 6, about 20 cm. in length.

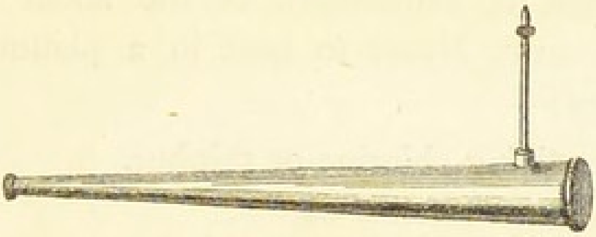

Fig. 6.

11. **An Iron Spoon,** or a small ladle, with bowl about 7 or 8 cm. in diameter.

12. **A pair of Crucible Tongs,** Fig. 7, about 20 cm. in length.

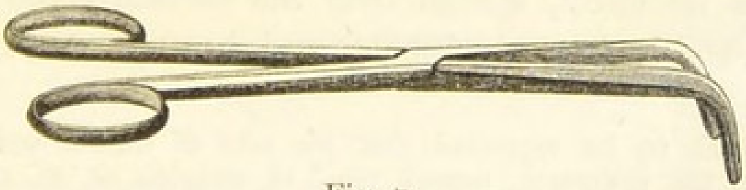

Fig. 7.

13. **A Set of Scales and Weights,** for ordinary purposes. A pair of well-made grocers' scales, with beam about 25 or 30 cm. in length, will answer the purpose. A set of

common gramme weights, from 1 kilog. to 1 grm. should be procured, if possible[1].

14. A Set of four Cork-borers, from 3 mm. in diameter upwards. These are short pieces of thin brass tube, sharpened at one end, and having a thick collar soldered to the other end, to afford a better hold. For the method of using them, see p. 32[2].

15. A piece of Platinum Foil, about 2 cm. × 5 cm. It should weigh about 0.4 grm. This is used chiefly as a support for substances on which we wish to try the effect of a high temperature, in order to test their fusibility, volatility, &c. If the edges of the foil are turned up round a spherical mould, such as the end of a pestle, we obtain a very convenient capsule for fusions on a small scale, e.g. for the decomposition of barium sulphate by sodium carbonate.

It should, however, be a rule—1st. Never to use a platinum vessel when a piece of porcelain will do as well. A bit of a broken evaporating dish will serve for almost every purpose, except when silicon, aluminium, or the alkali metals are to be tested for. 2nd. Never to heat in a platinum vessel the following bodies:—

Substances evolving chlorine or sulphur.
Caustic alkalies or alkaline earths.
Cyanides, chlorates, nitrates.
Easily reducible metallic salts, or their corresponding metals, e.g. lead, silver, tin.

When a platinum vessel is dirty, try first to clean it by boiling it in a dish with a little strong hydrogen chloride. If this has no effect, spread over the surface some powdered potassium-and-hydrogen-sulphate, and heat it over a Bunsen's

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[1] It is much to be regretted that no sets of metric weights corresponding to the ordinary 'pound pile' of weights at 2*s*. the set, can yet be procured in this country. It would seem hardly possible to bring the metric system into use, when the cheapest sets of gramme weights cost 12*s*. or 14*s*.

[2] When the cutting edge becomes blunt or bent, it should be sharpened on a hone, or by a very fine file, the borer being constantly rotated while the hone or file is passing over it.

burner until the salt fuses, inclining the vessel so that the liquid salt may flow over every part of it. Finally, boil it with water in a dish.

When a piece of platinum foil becomes creased or wrinkled, place it between folds of glazed writing-paper on a smooth surface, such as a plate of glass, and pass over it with strong pressure a rounded burnisher, such as the handle of a paper-knife.

16. A piece of Platinum Wire, about 25 cm. long and 0.32 mm. in diameter (No. 26 iron-wire-gauge). This is chiefly for use in blowpipe experiments, Part I, Sect. 4.

17. Two pieces of Brass or Copper Wire, about 30 cm. long and 1 mm. in diameter. These should be filed to a point at one end and then bent, the one into the form of Fig. 8, the other into the form of Fig. 9, and finally a small piece of wax taper about 3 cm. in length should be stuck upon the pointed end of each.

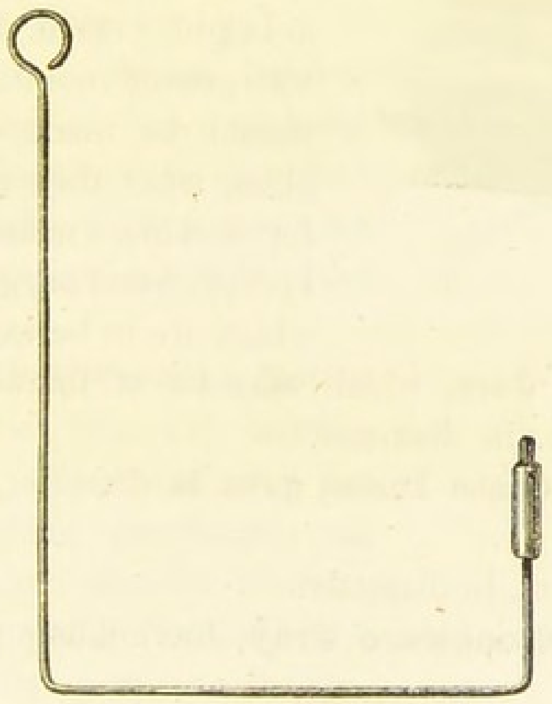

Fig. 8.

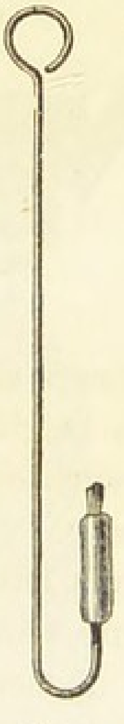

Fig. 9.

18. A Deflagrating Spoon, see Fig. 10. This consists of a brass or iron bowl about 1.5 cm. in diameter, screwed to a piece of stout iron-wire which passes rather stiffly through a cork stuffing-box attached to a tin flange. It is intended to hold substances which are to be burnt in gases.

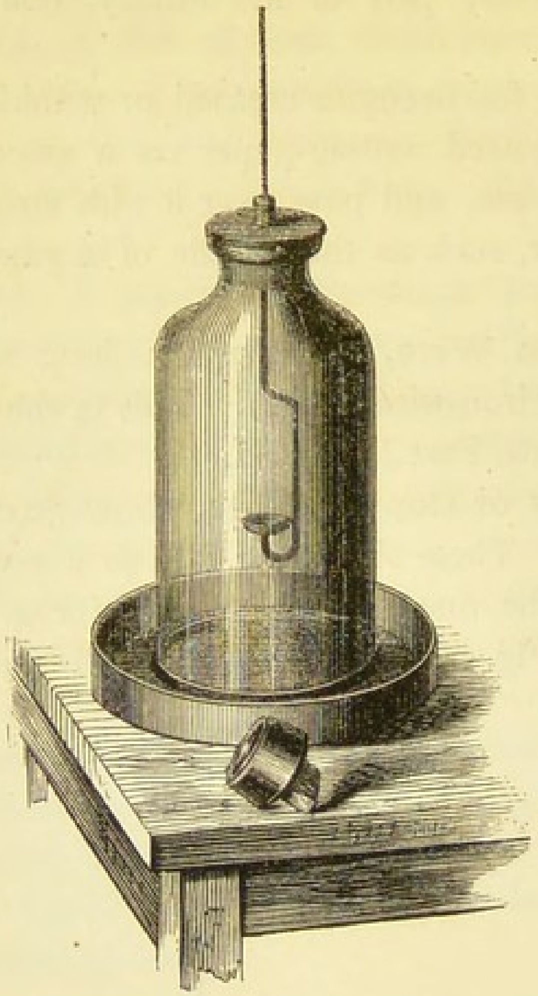

Fig. 10.

19. A Deflagrating Jar, Fig. 10. This is a wide-mouthed stoppered jar, open at the bottom, about 28 cm. in height and 15 cm. in diameter. The top should be ground flat, in order that it may be accurately closed by a glass plate.

20. Two strong cylindrical Glass Jars, for collecting gases; 10 cm. in height, 3 cm. in diameter, with ground mouths. They should be made of thick glass, since they are used for holding mixtures of hydrogen and oxygen gases which are to be exploded.

21. Three similar Jars, which may be of thinner glass, 18 cm. in height, 5 cm. in diameter.

22. Two circular Glass Discs, 5 cm. in diameter, ground on one side.

23. Two Ditto, 8 cm. in diameter.

24. One shallow Stoneware Tray, for holding gas jars, 8 cm. in diameter.

25. One Ditto, 18 cm. in diameter (see Fig. 10). This, though convenient, is not necessary, as a common dinner-plate may be substituted for it.

26. Four Florence Flasks. These may be procured from any oilman, and should be selected of uniform thickness, free from air-bubbles, and with even mouths not chipped away at one side. In order to cleanse them, pour a little strong hydrogen nitrate into each and heat it gently over a lamp, turning it round so as to bring the acid in contact with every part; finally, rinse it thoroughly, first with common water and then with distilled water, and place it to drain mouth downwards in the ring of a retort-stand.

27. Three Flasks with flat bottoms, holding respectively 200 c.c., 250 c.c., 400 c.c.

28. One plain Retort, holding about 200 c.c.

29. One stoppered Ditto, of the same size.

30. One cylindrical graduated Glass Measure, to contain 200 c.c., graduated into spaces of 5 c.c.

31. Two wide-mouthed stoppered Bottles, of white glass, holding about 700 c.c., for use in experiments on gases.

32. Four Ditto, holding about 300 c.c.[1]

33. Two common corked Bottles, with moderately wide mouths, holding about 200 c.c., for use as washing bottles for gases (see Fig. 39).

34. Two Ditto, holding about 300 c.c., for containing water which is to be saturated with a gas, such as chlorine or hydrogen sulphide (see Fig. 41).

35. One Washing Bottle with tubes (Fig. 11), holding 600 c.c. This is of great use for washing precipitates on a filter, and also for containing a supply of distilled water for general purposes in analysis. Its construction is sufficiently plain from the engraving[2]. When air is blown

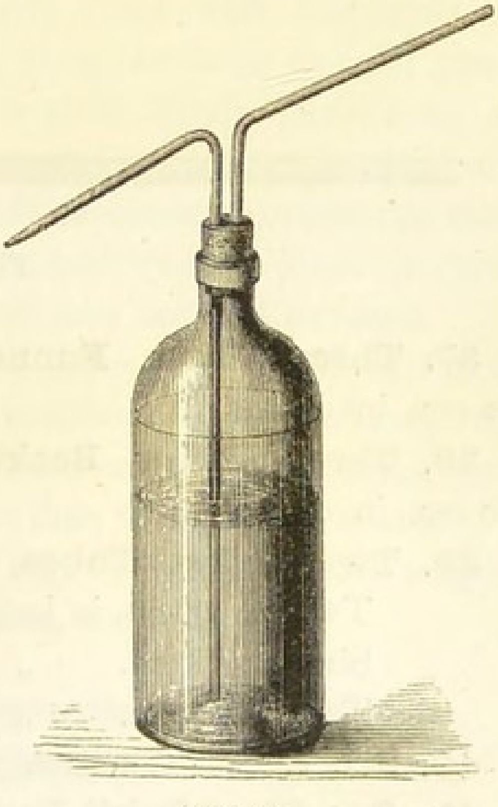

Fig. 11.

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[1] It is a great advantage to have the stoppers of these gas bottles made much more conical than usual. They are then far less liable to become fixed in their places if the volume of gas in the bottle should contract.

[2] This washing bottle may be easily fitted up by the student himself. Instructions for doing this are given at p. 31.

from the mouth into the up-turned tube, a stream of water is forced through the jet at the extremity of the other tube, and may be directed upon a filter, or into a test-tube. If a larger quantity of water is required at once, as in filling an evaporating dish or small flask, the bottle should be inverted so as to bring the blowing-tube lowermost, from which a stream of water will flow while air enters through the other tube.

36. Two Thistle Funnels, *a*, Fig. 12, about 32 cm. in length.

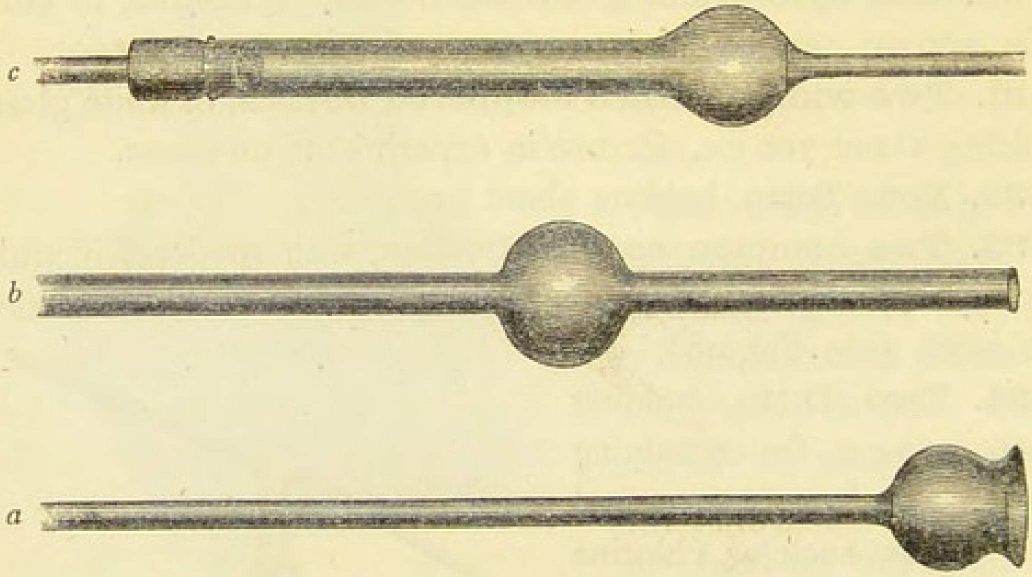

Fig. 12.

37. Three Glass Funnels, respectively 4 cm., 7 cm., 10 cm. in diameter.

38. Three Glass Beakers, respectively 5 cm., 7 cm., 10 cm. in diameter.

39. Twenty Test Tubes, of the following sizes:—

Twelve, 15 cm. in length, 1.5 cm. in diameter.
Six 18 cm. ,, 2 cm. ,,
Two 20 cm. ,, 2.5 cm. ,,

40. Six Watch Glasses, 5 cm. in diameter.

41. One Glass Spirit Lamp.

42. One Glass Mortar, 6 cm. in diameter, with pestle.

43. One Porcelain Mortar, 10 cm. in diameter, with pestle [1].

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[1] This mortar should not be glazed inside.

44. Three Porcelain Evaporating Basins, respectively 5 cm., 9 cm., 12 cm. in diameter[1].

45. One Porcelain Crucible, with cover, about 3 cm. in diameter.

46. Two common Cornish Crucibles, respectively 6 cm. and 8 cm. in diameter.

47. One Reduction Tube, *b*, Fig. 12, about 1 cm. in diameter.

48. Two Drying Tubes, *c*, Fig. 12, about 18 cm. in length. This form of tube is intended to contain calcium chloride, or other hygroscopic substance, in small fragments, for the purpose of removing moisture from gases which are passed through the tube. It is filled in the following way. After removal of the cork, a small tuft of cotton-wool or tow is pushed down into the bulb by means of a glass rod, until it lies across and protects the opening of the narrow tube. The rest of the bulb and the wide tube is then nearly filled with fragments of thoroughly dry calcium chloride, about as large as split peas. Another piece of cotton-wool is then lightly pushed in, to keep the calcium chloride in its place, and finally the cork with its short tube is replaced. It is essential, when the tube is not in use, to keep the ends stopped by little plugs of cork, in order that the moisture of the air may not find entrance.

The calcium chloride for these tubes should not be fused, but only thoroughly dried at a temperature of 200°–300° on a sand-bath. It is in this condition much more porous, and exposes a larger surface to the gas than the fused substance.

49. Twelve Sealed Tubes, Fig. 13, about 6 cm. in length. For directions for making such tubes, see p. 37.

Fig. 13.

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[1] These should be thin in substance (that there may be less risk of their cracking when heated over a lamp), and highly glazed both inside and outside. The Meissen ware is much the best, both as to shape and quality.

50. Two kilogrammes of soft Glass Tubing, free from lead, of different sizes, but chiefly about 6 mm. in external diameter[1].

51. Half a kilogramme of hard Glass Tubing, 5 mm. in external diameter, for making sealed tubes (No. 49) and arsenic tubes.

52. Two or three pieces of Combustion Tubing, about 30–35 cm. in length, and 12 or 14 mm. in diameter.

53. Three or four pieces of Glass Rod, free from lead, about 25 cm. in length, and 4 mm. in diameter: for stirring-rods.

54. A piece of vulcanized India-rubber Tubing[2], about 1 metre in length, and 4 mm. in external diameter: chiefly for use in connecting glass tubes.

55. Two or three pieces of similar Tubing, about 60 or 70 cm. in length, and 7 mm. in external diameter: chiefly for connecting lamps with the gas supply.

56. Three packets of circular Filters, respectively 7 cm., 14 cm., 20 cm. in diameter, suited to each size of funnel, No. 37.

57. One box of Test Papers, containing books of blue litmus, reddened litmus, turmeric, and Brazil-wood paper.

58. Two Brushes for cleaning tubes, one about 3 cm. in diameter, for test-tubes; the other about 5 mm. in diameter for smaller tubes.

59. A light Hammer, of the form known as riveting-hammer.

60. A small Anvil, about 6 cm. square, and 2 cm. in thickness.

61. A pair of cutting Pliers.

62. A three-square File, about 12 cm. in length.

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[1] The French soda glass is usually very good; but some specimens show a great tendency to devitrify when heated.

[2] Tubing of non-vulcanized india-rubber, which is also manufactured, adheres more closely to glass than the vulcanized tube, and is in many respects preferable to the latter. It has the disadvantage of losing its elasticity in cold weather, but after being warmed and stretched a little, it regains all its good qualities.

63. A round (or 'rat-tail') File, about 20 cm. in length. These files should be fitted into handles.

64. A few pieces of Charcoal, for blowpipe experiments. These may generally be selected from the ordinary rough beech-wood charcoal. Sticks about 3 cm. in diameter, free from knots, should be picked out and sawn across the grain into pieces about 3 cm. in length, or rather less. When thus cut, they should show a surface free from cracks and of close, sound texture.

65. A common China Jug, holding about 2 litres.

66. Half a quire of White Blotting-paper, and some sheets of glazed writing-paper.

67. Two or three dozen good Corks of various sizes, from 1 to 4 cm. in diameter. Those should be selected which are free from fissures and cavities, and in which the grain runs *across* not *along* the cone.

68. A common Metre Rule: the first decimetre divided into millimetres, the rest into centimetres.

The following pieces of apparatus are also extremely useful, and access to most of them will be assumed in the exercises:—

69. A Herapath's Gas Blowpipe, provided with a pair of double bellows, or with one of the small French india-rubber blowing machines, Fig. 14. In this form of blowpipe the gas issues from a brass tube about 1 cm. in diameter, in the axis of which a jet is permanently fixed, through which a blast of air is directed through the centre of the gas-flame. By altering the size of the air-jet and the quantity of gas, any kind of flame may be obtained from a large brush-like flame 16 or 18 cm. in length, to a small pointed cone of flame, such as is required for analytical experiments.

The india-rubber blowing machine alluded to, consists of two parts: 1st, The blower, *a*—a pear-shaped vessel of strong vulcanized india-rubber, having a valve fitted at each end enclosed in a small wooden box; 2nd, The regulator, *b*—a spherical vessel of thinner india-rubber, with two necks, one of which is connected by an india-rubber tube with the valve-box at one end of the blower, while the other neck is similarly connected with the blowpipe air-jet. When the blower is placed on the floor and compressed with the foot, the valve at one end closes, and the air contained in the vessel is forced through the other valve into the regulator, which becomes distended and forces the air through the blowpipe-jet. When the blower is relieved from the pressure of the foot, it recovers its shape, the valve nearest the regulator closes and prevents the return of the air, while a fresh supply of air enters through the other valve. When full, the blower

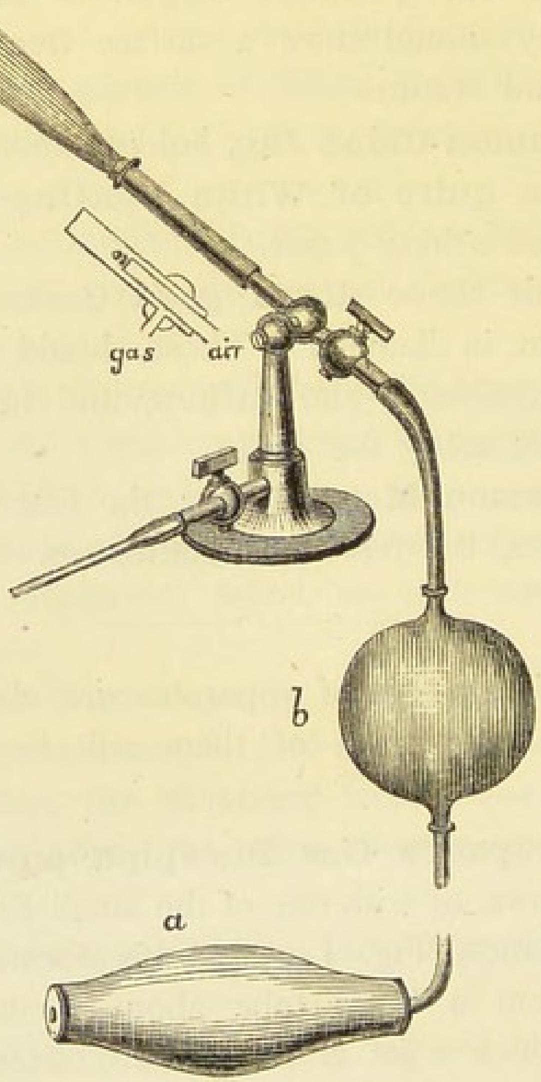

Fig. 14.

is again compressed with the foot so as to force another supply of air into the regulator.

This blowing machine is much cheaper and more portable than any form of double bellows, and is very effective. The regulator is usually made too small, and thus there is a slight variation in the strength of the blast. The india-rubber must be of the best quality; and it is advisable to enclose the regulator in a net, to prevent its becoming so far distended as to burst.

A substitute, which can be made without much difficulty, will be described at the end of the book.

Although such a blowpipe as that which has just been described is necessary for some operations in glass blowing, and renders the chemist almost independent of a furnace for fusions on a small scale, yet much may be done by the use of the mouth blowpipe supported in a Bunsen's holder (so that both hands may be free) and directed upon a larger gas-flame than usual.

70. A Glass-blower's Lamp: when gas is not available. This, in its simplest form, consists of a flat tin dish, near the centre of which a tin wick-holder is fixed in a slanting direction, sufficiently large to hold a bundle of strands of lamp-cotton about 1 cm. in breadth and 3 cm. in length. The dish is filled with lamp-oil, or melted tallow, and the wick is trimmed, a furrow being formed along the middle of it, in a line with which, and a little above it, the blowpipe-jet is placed.

71. A Clark's Retort and Receiver, Fig. 15; useful for distillations on a small scale.

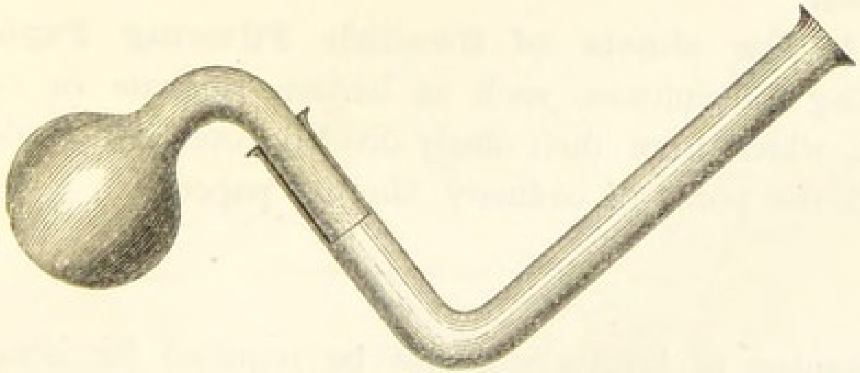

Fig. 15.

72. A Thermometer, with cylindrical bulb, graduated on the stem from $-10°$ to $+150°$ centigrade.

73. A Platinum Spatula, about 9 cm. in length, broader at one end than the other. It need not cost more than 6*s.* or 7*s.*, and will be found most useful. Instead of it, a German-silver spatula of the same size may be obtained at a much lower price, and will answer for most purposes.

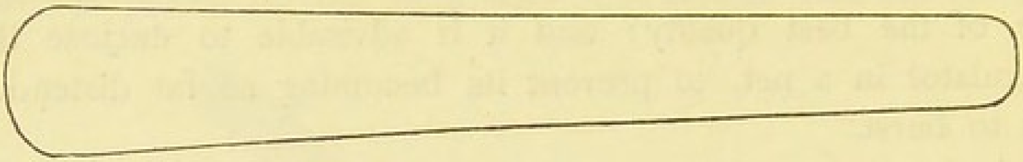

Fig. 16.

74. A Platinum Capsule: hemispherical, about 3 cm. in diameter. This, which would cost about 5*s.*, will answer almost every purpose of a platinum crucible.

75. A Bunsen's Screw Pinch-cock, Fig. 17. This is a contrivance for regulating the flow of a liquid through an india-rubber tube. The tube is placed, as shown in the engraving, between the two parallel rods, and may be compressed by turning the screws, until the passage through it is entirely obstructed.

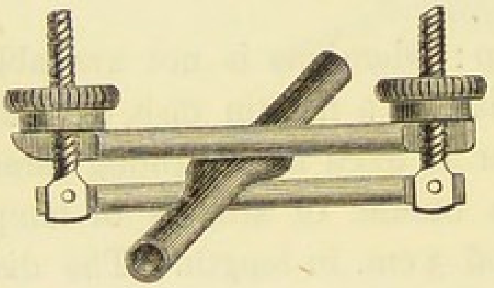

Fig. 17.

76. A small round Wicker Basket, with upright sides, about 14 cm. in diameter and 10 cm. in depth; for holding test-tubes.

77. A few sheets of Swedish Filtering Paper, for separating precipitates, such as barium sulphate or calcium oxalate, which from their finely-divided condition would pass through the pores of ordinary filtering paper.

A number of bottles will also be required for containing substances both solid and in solution. Many substances, such as calcium chloride, from their alterability in the air, will of necessity be purchased in bottles. Inclusive of these, the following stock of bottles will be probably sufficient[1] :—

24	wide-mouthed bottles,	with corks,	300 c.c.	capacity.
24	" "	"	100 c.c.	"
6	" "	with glass stoppers,	300 c.c.	"
12	" "	"	100 c.c.	"
8	narrow-mouthed bottles,	"	300 c.c.	"
40	" "	"	100 c.c.	"
3	" "	"	2000 c.c.	"

The common green or blue glass bottles, costing (when stoppered) from 3*s.* to 6*s.* a dozen, will answer quite as well as the more expensive bottles of white glass.

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[1] It is not absolutely necessary for the beginner to start with so large a stock of bottles. Some substances, e. g. marble, sulphur, manganese dioxide, &c., may be kept in boxes. But bottles are far preferable, on the score both of cleanliness and security.

LIST OF SUBSTANCES[1]

Required for the Course of Practical Work contained in Parts I and II of this Book.

The numbers in the column on the extreme left of the page are intended to give some idea of the relative quantities of the substances which will be necessary. If 1 be interpreted to mean 10 grammes (about one-third of an ounce), 2 = 20 grammes, &c., the quantities will be sufficient for the purposes of most students.

Many of the substances are required for use in solution. In Part I, Sect. 1, Exercise 4, will be found the general method of dissolving a salt in water; and the mode of proceeding in certain special cases will be found in Part I, Sect. 3.

An asterisk is prefixed to the names of those substances which the student should prepare himself.

9	Alcohol, pure, sp. gr. 0.815.
60	" Methylated, for common purposes.
12	Aluminium and Ammonium Sulphate (Ammonia Alum).
3	Ammonium Carbonate, pure.
3	" Chloride, pure crystallised.
12	" " common.
9	" Hydrate, solution of, sp. gr. 0.96 (Caustic Ammonia).
1	" Molybdate.
9	" Nitrate.

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[1] Those who are working in a regular laboratory will probably obtain the necessary chemicals from the common stock. This list is more particularly intended to assist those who are working by themselves, in selecting the substances they will require.

3 Ammonium Oxalate.
2 ,, Phosphate [1].
2 ,, Sodium and Hydrogen Phosphate (Microcosmic salt).
6 ,, Sulphide, solution of.
2 Antimony, Metallic.
3 ,, Trisulphide.
1 Arsenic Trioxide (White Arsenic).
3 Asbestos, in long loose fibres.
3 Barium Chloride, pure.
2 ,, Oxide (Caustic Baryta).
1 Bismuth, Metallic.
1 Cadmium Sulphate.
30 Calcium Carbonate, pieces of white marble.
9 ,, Chloride, thoroughly dried, in lumps.
3 ,, Fluoride, white fluor spar.
10 * ,, Hydrate, solution of (Lime water) [2].
3 ,, Hypochlorite (Bleaching Powder).
20 ,, Oxide, freshly burnt white Quicklime.
20 ,, Sulphate (Plaster of Paris).
* ,, Sulphate, solution of.
6 Carbon, selected pieces of charcoal.
3 ,, Disulphide (Bisulphide of Carbon).
3 Chlorine, solution of (Chlorine water).
2 Chromium and Potassium Sulphate (Chrome Alum).
1 Cobalt Nitrate.
1 Cochineal, solution of.
20 Copper, Metallic; strips of sheet-copper, about 0.5 mm. thick.
,, pieces of wire, No. 16 and 26 wire-gauge.
6 ,, Oxide (Black Oxide of Copper).
12 ,, Sulphate.
2 Cotton-wool.
Distilled Water, see p. 54.

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[1] This salt is preferable, as a test, to sodium and hydrogen phosphate, but is not necessary.

[2] For the method of making this solution, see p 45.

		Gold, Metallic; half a book of gold-leaf.
2		Grape Sugar.
6		Hydrogen Acetate, solution of, sp. gr. 1.04 (Acetic Acid).
6		„ Chloride, pure concentrated (Hydrochloric Acid).
10	*	„ Chloride, pure diluted (20 c.c. concentrated acid to 80 c.c. water).
15		„ Chloride, common concentrated.
6		„ Nitrate, pure concentrated (Nitric Acid).
10	*	„ Nitrate, pure diluted (20 c.c. concentrated acid to 80 c.c. water).
12		„ Nitrate, common concentrated.
6		„ Oxalate (Oxalic Acid).
12		„ Sulphate, pure concentrated (Sulphuric Acid).
10		„ Sulphate, pure diluted (20 c.c. concentrated acid to 80 c.c. water[1]).
24		„ Sulphate, common concentrated.
	*	„ and Silicon Fluoride, solution of (Hydrofluosilicic Acid).
	*	„ Sulphide, solution of, see Part I, Sect. 3 (Sulphuretted Hydrogen).
3		„ Tartrate (Tartaric Acid).
3		Indigo Sulphate, solution of (Sulph-indigotic Acid).
2		Iodine.
12		Iron, Metallic; thin wire and one or two strips of thin sheet-iron.
	*	„ Perchloride.
2		„ Peroxide (Rouge).
9		„ Protosulphate.
9		„ Protosulphide, in lumps.
12		Lead, Metallic; strips of sheet-lead.
3		„ Acetate.
3		„ Nitrate.
3		„ Protoxide (Litharge).

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[1] For the precautions necessary in diluting this acid, see p. 86.

3	Lead, Red Oxide (Red Lead).
3	Litmus, solution of (Archil).
0.5	Magnesium, Metallic; wire or ribbon.
3	,, Sulphate.
30	Manganese Dioxide (Black Oxide of Manganese).
12	Mercury, Metallic.
3	,, Oxide.
3	,, Perchloride (Corrosive Sublimate).
1	,, Protochloride (Calomel).
1	,, Protonitrate.
1	Nickel Sulphate.
2	Phosphorus.
2	,, Amorphous (Red Phosphorus).
	*Platinum Perchloride.
0.5	Potassium, Metallic.
1	,, Bromide.
20	,, Chlorate.
3	,, Chromate (Yellow Chromate of Potassium).
3	,, Cyanide.
2	,, Ferricyanide (Red Prussiate of Potassium).
6	,, Ferrocyanide (Yellow Prussiate of Potassium).
6	,, Hydrate (Caustic Potash).
3	,, Iodide.
12	,, Nitrate (Purified Saltpetre).
2	,, Nitrite.
2	,, Sulphocyanide.
	Silver, Metallic; a book of silver-leaf.
1	* ,, Nitrate.
1	Sodium, Metallic.
3	,, Diborate (Borax).
2	,, Carbonate, pure, anhydrous.
20	,, Carbonate, pure, crystallised.
12	,, Chloride (Common Salt).
3	,, and Hydrogen Phosphate.
3	,, Hyposulphite.
3	,, and Hydrogen Tartrate.
6	,, Sulphate.

3 Starch.
2 Strontium Nitrate.
12 Sulphur; roll Sulphur, and Flowers of Sulphur.
12 Tin, Metallic; in strips and foil.
* ,, Protochloride (Stannous Chloride), solution of.
3 ,, Peroxide (Binoxide of Tin).
2 Turpentine.
12 Zinc, Metallic; pieces of sheet-zinc. The purest form of the metal is the Belgian rolled zinc, scraps of which may be procured from any tinman.

PART I.

EXPERIMENTS ON THE PREPARATION AND PROPERTIES OF SUBSTANCES.

SECTION I.

PRELIMINARY EXERCISES.

EXERCISE 1.

Fusion and Granulation.

Apparatus required—Iron ladle, with bowl about 7 cm. in diameter; iron spoon; pan filled with clean water; pair of pliers; cloth; pieces of metallic zinc.

Put a few small pieces of zinc into the ladle, and place the latter upon a clear fire[1], supporting it on the coals so that the bowl may rest steadily in a horizontal position. Zinc requires a rather high temperature (412°) for its fusion, but when the bottom of the ladle becomes heated to faint redness the fragments of metal will sink down into a fluid mass. When this takes place, add some more pieces of zinc, pressing them down into the fused metal by means of the iron spoon. If any of the pieces of sheet-zinc are too large to go conveniently into the ladle, bend them into a more compact form with the help of the pliers. You will find it much easier to effect this when the metal is made quite hot by being held in front of the fire; since a sheet of zinc which is stiff and unyielding at the ordinary temperature becomes remarkably pliant when moderately heated. Go on adding pieces of zinc until the ladle is about three-fourths filled, and then

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[1] A Bunsen's burner will answer the same purpose, but not so well.

leave it on the fire for a minute or two longer, in order that the metal may become quite fluid. The earthy-looking substance, or dross, which floats upon the melted metal consists of a compound of zinc with one of the constituents of the air, and corresponds in composition to ordinary litharge. This should, at the last moment, be skimmed off with the iron spoon, so as to leave the surface of the melted metal quite bright, like mercury. You will notice, however, that no sooner has the coating of dross been removed than a thin film of it begins again to be formed, owing to the contact of the air with the strongly heated metal. Now take the ladle off the fire at once, and, holding it about a metre or more above the pan of water, pour the liquid zinc in as thin a stream as possible into the water. The steam formed when the hot metal touches the water blows the particles of zinc asunder, and they fall to the bottom of the pan in feathery, tumefied fragments, which from the great surface they expose to the action of a solvent are well adapted for use in the preparation of hydrogen gas (Sect. **2**, Ex. 2). The metal in this form is called granulated zinc.

The water in the pan should be poured away, and the zinc should be collected, dried as far as possible with a cloth, then completely dried by being placed on a plate in front of the fire, and kept for use in a wide-mouthed bottle or jar.

EXERCISE 2.

Glass Working[1].

1. To bend glass tubing.

Apparatus required—Pieces of glass tubing, about 6 mm. in external diameter; pieces of glass rod, rather smaller in diameter; three-square file; fish-tail gas-burner, on iron foot; Herapath's blowpipe.

Select a piece of readily fusible glass tubing about 6 mm.

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[1] In this and the succeeding Exercise only the more elementary operations in glass blowing are treated of, such as must be learnt in order to

in external diameter. Lay it down on the table before you, holding it down with the thumb and forefinger of the left hand, placed about 15 cm. from one extremity. Make a small notch in the tube close to this point with a three-square file, slightly pressing the side of the file against the left thumb, which will thus serve as a guide to prevent the edge of the file slipping along, instead of cutting across, the glass. The notch should be made, not so much by a repeated to-and-fro motion of the file, as by one, or at most two, short forward strokes combined with as much downward pressure as the tube will bear, the hand being raised a little as the file goes forward, so that it may follow the curve of the surface of the glass. By this means the file will cut deeper into the glass with less injury to itself, than if the edge were drawn to and fro from point to handle as is usually done; the effect being then rather a rub than a cut. Now take up the tube, holding it with both hands thus, Fig. 18 (one hand being on each side the notch, and the thumb-nails pressing against the glass on the side opposite the notch), and break it asunder precisely as a stick is broken.

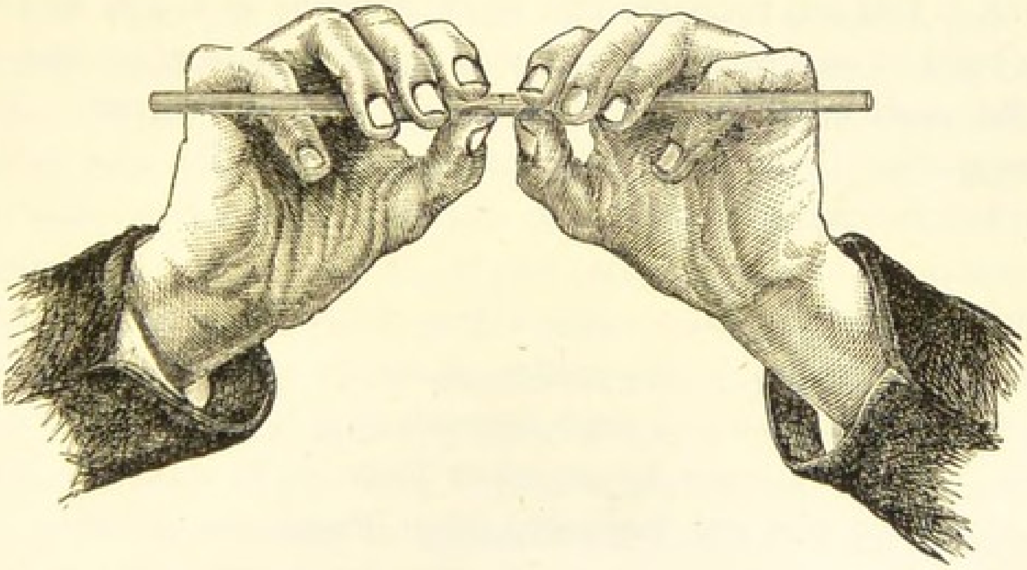

Fig. 18.

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fit up the apparatus required in Part II. Further instructions for glass blowing will be found at the end of the book in Part VII, Sect. 2.

Pass the file lightly with a rasping motion over the sharp edges of the glass, to save the fingers from being cut in holding the tube. For bending the ordinary fusible glass tubing, if its external diameter does not exceed 1 cm., a blowpipe-flame is neither required nor so suitable as the flame of a common fish-tail or bat's-wing gas-burner; but if gas is not at hand, a spirit lamp with a large flame may be used. Light the gas, or spirit lamp; then holding the piece of tube by its extremities, bring it about 7 or 8 cm. above the flame, turning it constantly round and moving it laterally so as to heat about 5 cm. of it equally on all sides. After a few seconds, lower it gradually into the flame, still constantly turning it round. If the gas burner be used, the glass will become covered with soot when immersed in the flame; but this is of no consequence as the heat of such a burner is never high enough to incorporate the carbon with the glass. When the heated portion becomes soft and yielding, which will take place even before it has acquired a visible red-heat, withdraw it from the flame, and gently bend it to a right angle, avoiding the use of much force. You will probably find some difficulty at first in making the bend in one plane, i. e. so that the bent tube when laid on a flat surface may touch it in every part of its length. The best method of accomplishing this is, to support the tube lightly by its extremities, so that the direction of the bend may be determined mainly by the weight of the tube itself; then, holding it before you so that a line drawn from the eye may pass through both its extremities, gradually approach the hands to each other, as if you were endeavouring to snap the tube in two. Do not attempt to use much force, or to make the bend suddenly, or you will inevitably either flatten the glass on the outer side or wrinkle it up on the inner side; either fault being fatal to the strength of the bend. It will usually be found necessary to heat the tube again in order to complete the bend; and it is better, if there are any signs of wrinkling or flattening, not to attempt to bend it further in that part, but to heat another portion of it a little on either side of the partially-made bend, and to complete the curve in that portion.

The correctness of the angle may be judged of by holding the tube close to, but not actually touching, an ordinary square, or even the corner of the table or of a book. When the proper bend is completed, lay the tube on a bit of glass in such a position that the heated portion does not come into contact with any cold surface, and leave it to cool slowly. While it is cooling you may cut off another portion of the same tubing about 18 cm. long, and bend it in a similar way, making the bend, however, not in the middle of the tube, but about 7 cm. from one end. If that end becomes too hot to hold in the fingers it may conveniently be inserted in a hole made in a small cork, which will then answer the purpose of a handle. The tubes thus bent will serve to fit up a washing bottle or generating flask for gases; and several similar ones with the branches varying in length may be made at leisure moments from any waste bits of tubing, and will be found generally useful, saving much time in fitting up apparatus for any particular experiment.

2. To make a stirring rod.

For this purpose the heat of a gas or spirit lamp is scarcely sufficient; it must be intensified by the use of a blowpipe. The best form of this instrument is fully described at p. 14, and it will only be necessary here to explain the method of using it so as to produce the greatest effect. If a Herapath's blowpipe is at hand the process is simple. Turn on the gas full, and light it at the mouth of the blowpipe. Then work the bellows gently and uniformly, and gradually reduce the supply of gas, until you have a steady cone of blue flame about 10 or 12 cm. long. If a spirit or oil lamp must be used, the wick should be trimmed flat and pulled up sufficiently to give a large flame; then with the trimming scissors separate the wick into two parts, and bend each portion sideways so as to leave a clear passage for the air between them. Arrange the lamp and jet so that the latter may lie, pointing slightly upwards, in the line of the passage just formed in the wick, and on a level with the top of it. Light the lamp, and, introducing the jet just within the flame, commence the blast of air. The flame of the lamp will now be almost entirely deflected in the direction of the stream of air; and by pushing the jet a little further into the flame, or drawing it back beyond the margin, any kind of flame may be produced from a quietly-burning, pointed blue cone to a large roaring brush of flame; the former being most suitable for the present purpose. If the flame is ragged or irregular, see whether any filaments of the wick stand in the way of the blast, and if so remove them with the scissors. If this does not cure the defect, the blowpipe-jet is dirty or not truly circular, and must be cleaned out by a large needle, or, better, by a small broach. Having obtained a satisfactory flame, select a piece of glass rod about 5 mm. in diameter, and cut off a piece about 16 or 18 cm. long, as directed in the case of the glass tubing. Hold one end of this piece about 5 cm. in front of the visible flame, turning it constantly round by a twisting motion of the fingers, and gradually bring it just within the apex of the flame, which is its hottest portion. The edges of the glass will soon begin to melt, and the sharp angle will disappear as the glass approaches the liquid condition. The heat should be continued until the end is perfectly round, and then the rod should be gradually withdrawn from the flame and allowed to cool slowly, resting on another fragment of glass or on the table so that the hot end may project over the edge. Make another stirrer, rather longer, in a similar manner; and when the first is cool, round off its other extremity in the same way.

It should be noticed, once for all, that in all kinds of glass working, the process of annealing is of the utmost importance. Glass must never be either heated or cooled suddenly, unless the special object is to produce a crack. The material is such a bad conductor of heat, that the end of a piece of glass may be raised to a red-heat, while at a distance of 3 cm. from this portion it remains for some time sufficiently cool to be held in the fingers. In consequence of this low conductivity, when heat is applied suddenly to a piece of glass, the parts immediately in contact with the source of heat expand before the heat is communicated to the neighbouring parts, and thus tend to tear the latter asunder. Again, when glass is suddenly cooled, the surface contracts at once, and is torn asunder by the still expanded adjacent portions, which have not had time to lose their heat. The useful applications of this property will be alluded to presently; but it should be a rule—1st, Never to bring a piece of glass into the blowpipe-flame suddenly, but to hold it for half a minute, more or less according to the size and thickness of the glass, in the current of hot air which surrounds the flame, constantly turning it round, and heating more of it than is intended ultimately to be brought to a red-heat. 2ndly, After the work is done, to withdraw the glass very gradually from the flame, occupying a minute or so in removing it to a distance of 12 or 13 cm. in front of the flame; then to leave it to cool very slowly in a position protected from currents of air. It will sometimes be found useful to have at hand a dish of strongly heated sand, into which the hot glass may be plunged and left to cool slowly; and in all cases it is better to err on the safe side, than to risk the breaking of a tube owing to its particles being in a state of tension from deficient annealing. 3rdly, Always to keep the glass turning while it is in the flame. The heat of a lamp or blowpipe is mainly applied to one side of an object, viz. that which is turned towards the wick; while almost all operations in glass blowing require that the piece of glass should be uniformly heated on all sides. If this simple rule be not attended to, it will be found impossible to blow a good bulb and even to make a good bend in a tube. Practice alone, however, can give that steadiness of hand, and *adhesiveness*, as it were, of the fingers to the glass, which will enable the student to rotate a piece of tube which is heated to fusion in the middle, without sensibly distorting the softened part. The portions of the tube on either side of the centre may be regarded as two distinct tubes united by a flexible material; and the object should be, to keep these two tubes in the same straight line, and to rotate them continuously at the same rate, without laying any stress on the connecting portion. It is generally best to hold the hands under the glass, the up-turned forefinger and thumb being chiefly employed in rotating the tube, while the other fingers sustain it at such points that the portions of glass on either side of the heated part are pretty evenly balanced, and have no great tendency to tilt in either direction.

3. To make a glass jet.

Cut off a piece of glass tubing (the more infusible the better) about 18 cm. in length and 3 mm. internal diameter. Heat the middle of the tube to the extent of 1 cm. in the flame of the blowpipe, until it becomes quite soft and begins to thicken and contract in diameter: then withdraw it from the flame, and pull the two ends apart by slightly separating the hands, until the drawn-out portion is contracted to an external diameter of 2 mm. Be careful not to use much force in drawing out the glass, or it will be contracted to a fine thread so thin as to be useless. When it is cool, make a fine scratch with a file at the middle of the drawn-out portion, and break the tube at this point. You will then have two jets of the shape represented in Fig. 19, which may be kept for use in future experiments. If the aperture of the jet is too large, hold it for a few moments in the blowpipe-flame, turning it quickly round; the edges of the glass will then sink in, and the size of the aperture may be reduced to any desired extent.

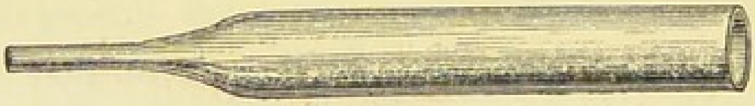

Fig. 19.

EXERCISE 3.

Glass Working (*continued*).

Apparatus required—The same as for Exercise 2, with the following additions: Pieces of less fusible glass tubing; corks; cork-borers; cork-squeezer, or pliers; narrow-mouthed bottle, about 600 c.c. capacity; rat-tail file; piece of charcoal, about 3 cm. in diameter.

1. To fit up a washing bottle similar to that which is included in the List of Apparatus, p. 9.

In doing this you will have to apply the experience in glass working which you have already gained, and in addition to learn the method of boring holes in corks.

Get ready the blowpipe, arranging it to give a brush-flame as described in the last Exercise. Cut off a piece of glass tubing about 5 mm. in external diameter and 45 cm. in length; hold it horizontally in the hands and heat it (observing the precautions given in p. 29) in the blowpipe-flame at a point about 15 cm. from the right-hand extremity. As the tube is somewhat long, you will find an advantage in supporting it near the other extremity on a ring of the retort-stand, or other support, fixed at the same height as the blowpipe. This will render it easier to keep the tube turning between the fingers (p. 29), and to maintain it in the proper position when it becomes soft. Allow the heated portion of the glass to become slightly thickened, then raise the tube out of the flame and draw it out slowly and carefully (waiting until the glass has so far cooled that a little force is required for the purpose), until the contracted portion is about as thick as a large knitting-needle. Allow it to cool slowly, and then cut it in two at the middle of the contracted portion. You have now two tubes, the one 30 cm. and the other 15 cm. in length, each terminating in a jet. Lay aside the shorter tube for the present, and heat the extremities of the longer tube just sufficiently to round off the sharp edges; then (using the fish-tail burner) bend it to an acute angle equal to that of the left-hand tube in Fig. 11, making the bend about 8 or 9 cm. from the jet. While it is cooling take the other tube, cut off the contracted portion at the end, round off the sharp edges of each end in the blowpipe-flame or with a file, and bend it near the middle to an obtuse angle equal to that of the right-hand tube in the figure.

It now only remains to fit these tubes to the bottle by means of a cork perforated with two holes. It will be best however to begin by practising the method of making a single hole in the centre of a cork. Take a good sound cork about 2 cm. in diameter, squeeze it until it becomes soft and elastic (a pair of pliers or nut-crackers will serve the purpose of a regular cork-squeezer), then take it up thus, Fig. 20, between the second finger and the thumb of the left hand, and place the sharpened end of the smallest cork-borer against it, as near the centre of one end as you can judge. Urge the cork-borer into the cork with a twisting motion, as if you were using a cork-screw. Some care will be required to make the hole straight through the cork, so that it may be truly central. Of the proper direction the eye will be the best judge: and when the cork-borer has penetrated some little way, it will be advisable to turn the cork a quarter round, in order that it may be seen whether the axis of the cork-borer and of the cork

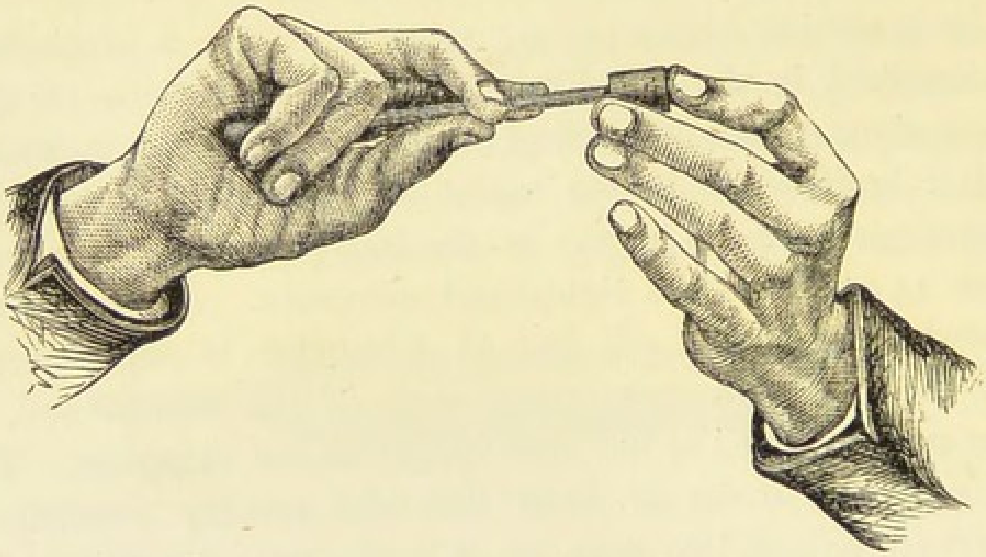

Fig. 20.

are still in the same straight line. If not, a slight pressure on the cork-borer in one direction or the other will set it straight. When the borer has penetrated quite through the cork it may be withdrawn with a twisting motion, and will bring with it a cylindrical plug of cork, leaving a hole, the sides of which should be smoothed with the round or rat-tail file. The plug of cork remaining in the borer may be pushed out by means of a wire which is usually sold with the set of borers for that purpose. It should not be thrown away, as such small corks are often very useful for stopping the ends of drying-tubes, and other purposes.

When you have practised in a similar way on one or two other corks, and have learnt to control the direction of the borer, you may complete the fitting up of the washing bottle. Take an ordinary narrow-mouthed bottle holding about 500 or 600 c.c.; choose a cork slightly too large to fit it (since the cork is reduced in size when it is squeezed), and render it soft and elastic by squeezing it. You have now to make two holes in it in the position of Fig. 21, on opposite sides of the centre, and about midway between the centre and circumference. Take for the purpose a cork-borer rather smaller than the tubing which you have been using, and bore the two holes, with particular care that each hole does not run into the other or pierce the side of the cork. The cork-borer may be slightly oiled, if thought necessary, but this will be seldom requisite if the end is kept properly sharp. The holes should next be smoothed and slightly enlarged by the rat-tail file, until the end of one of the tubes will just enter them when some little pressure is used. Now pass the longer branch of the longest of the two tubes through the cork, with moderate pressure[1] and a twisting motion, until it

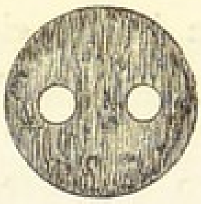

Fig. 21.

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[1] If much pressure is used the tube is not unlikely to break, and the splinters of glass may cause a serious cut. The hole should never be so much smaller than the tube as to make it necessary to use much force in passing the latter through it. It is a good plan, also, to wrap

projects so far as to reach, when the cork is fitted into its place, nearly to the bottom of the bottle. When this is done, pass one of the branches of the other tube through the other hole in the cork, until it projects 3 or 4 mm. on the other side. Fill the bottle with distilled water, fit the cork carrying the tubes tightly into the neck of it, and your washing bottle is ready for use. Blow gently through the up-turned end of the shorter tube, and see whether a fine stream of water issues from the jet of the other tube. If the jet is found to be too large, it may be easily reduced in size by holding the tip of it (constantly turning it round) for a second or two in the flame of a Bunsen's burner[1]; the tube being previously withdrawn from the cork, and thoroughly dried internally by passing it to and fro over the flame of a lamp and sucking, not blowing, air through it. If this precaution is neglected, a drop of water may flow down into the jet, while it is being heated, and will infallibly crack it.

If no water issues from the jet when you blow air into the bottle, either the aperture is closed up, in which case a small portion of the tip of the jet may be cut off with the file, or there is a leakage of air at the cork. Place a drop or two of water on the cork, and observe whether, on blowing as before, bubbles rise through it. If they do, you may remedy the fault either by pushing the cork more tightly into its place or by melting a little sealing-wax over the top of it, or, if these fail, by taking a new cork altogether, and boring the holes with more care. Never be satisfied with an imperfect apparatus.

2. To seal a glass tube.

This is the simplest operation in glass-blowing strictly so called, i. e. in which the assistance of the breath is called in, to mould the glass into shape; and it is one which the student

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the tube in a cloth or handkerchief while it is being passed through the hole in the cork.

[1] The flame of the blowpipe may be used; but without some care and skill in its management you would run the risk of closing up the jet altogether.

is continually called upon to practise for mending broken test-tubes and making arsenic reduction-tubes.

Test-tubes, however, are made of such thin glass that it is by no means so easy as it appears to seal them neatly; and you will find it best to commence work on a piece of soft glass tube about 5 mm. in external diameter. Cut off a piece of this tubing about 15 cm. in length, and having arranged the blowpipe so as to give a large steady flame, hold the tube horizontally and bring it gradually into the hottest part of the flame, so that about 1 cm. near the middle may be thoroughly heated. When the glass becomes quite soft, remove it from the flame and draw it out a little by separating the hands, until it assumes the form of Fig. 22. Now direct the flame against the part which lies a little to the left of the most contracted portion, and draw it out further until nothing but a thread of glass remains to connect the two portions of the tube, Fig. 23. This thread of glass should next be heated just at the point where it joins the tube on the left hand, when it will fuse and separate, running up into a small knob against the thicker portion of the tube. Lay down the right-hand piece of tube (resting it on another bit of glass that it may not burn the table), and proceed to make the end of the other piece smooth and round. This is done by directing the flame upon the small projecting knob, which will soon fuse and partially incorporate itself with the surrounding glass. The whole end of the tube will, however, have become contracted and thickened, and must be expanded a little by removing it from the flame and forcing air very gently into it from the mouth, until it takes the shape

Fig. 22.

Fig. 23.

Fig. 24.

of Fig. 24. Do not blow too hard at first, or heat more of the tube than is necessary, or you will probably expand the sides of the tube into a bulb, which is not your present object: your intention being simply to distribute evenly the thickened glass at the extremity by driving it forwards rather than outwards, and to mould the end of the tube into the regular round form of Fig. 24. If this is not accomplished at the first trial, bring the end of the tube again into the blowpipe-flame until it contracts, and blow it out as before, keeping the attention, while blowing, fixed on the heated glass so as to be ready to moderate or stop altogether the supply of air from the mouth, if the glass shows signs of yielding too much. Now anneal the tube as above directed, and finish the other, or right-hand piece of tube, in a similar way, drawing it out and rounding it exactly as before. If the bit of capillary tube remaining attached to it is too short or too slight to be used as a handle in drawing out the glass, it should be cut off, and while the end of the tube is heated in the flame, a bit of waste glass held in the right hand should be heated and pressed against it, and the flame directed upon the point of junction. The two pieces will then adhere, and by applying the flame a little more to the left the superfluous glass may be melted and drawn off, attached to the bit of waste glass. Moreover, if the remaining knob be too large to be neatly melted into the bottom of the tube, it may, when soft, be touched with a bit of heated glass and drawn off in a similar way.

After a few small tubes have been thus sealed, larger tubes may be operated on in the same way, a larger blowpipe-flame being employed. Test-tubes broken at the bottom will be found good materials for practising on, and may be mended by drawing off the broken portion and sealing them as above. The glass of which these tubes are made is so thin that it is not easy at first to avoid over-heating them in some one spot, thus producing a mis-shapen end, which is quite inadmissible in vessels which, like test-tubes, are exposed to comparatively sudden changes of temperature. The softened portion should not be drawn out much at first, but allowed to sink in and contract of its own accord, in order to thicken it a little : and especial care must be taken that the bottom is of uniform thickness and well annealed. If there is a crack in the tube, the piece must be broken off by a slight blow; or, better, the crack may be led round the tube as described in the next section, otherwise it will extend itself when the tube is heated.

Another very useful form of sealed tube which may be made by the student himself is that known as the 'arsenic tube,' which serves for heating substances either *per se*, or with a flux, in the preliminary examination in qualitative analysis. These tubes are of hard infusible glass, about 5 or 6 cm. long and 5 mm. in external diameter; and the manufacture of them will be a moderate test of proficiency, as it will require some dexterity to get rid entirely of the small knob of glass already mentioned. The end must be very strongly heated, and the breath thrown in with some force, the moment that the glass is removed from the flame. These tubes are shown of the actual size in Fig. 13. It will be seen that the sealed end is slightly expanded so as to form a small bulb. This is done after the knob has been got rid of, by strongly heating a portion of the sides as well as the end of the tube, and then blowing into it with considerable force. The bulb should not, however, be larger than is shown in the figure, or it will be too thin to be of much use.

3. To divide glass by leading a crack along it.

The low conducting power of glass for heat renders it, as has been already noticed, very liable to crack from sudden changes of temperature. From the same cause, however, it is easy to extend a crack, when once begun, in any desired direction by heating the parts of the glass which lie just in front of the crack. The method is extremely simple, and will be found especially useful in cutting off the necks of flasks when they are chipped or uneven, and in making evaporating basins or capsules out of broken flasks or retorts.

Suppose, for instance, a Florence flask has, as is often the case, a neck too uneven to allow of a cork being fitted into it. Arrange the blowpipe to give a rather large flame; take any waste bit of glass rod or tube about 6 mm. in diameter and 7 or 8 cm. in length; draw it out in the flame as directed in the last section, leaving the end somewhat pointed. Now take the body of the flask in the left hand, the neck pointing upwards, and, having heated the pointed end of the rod to full redness, apply it to the outside edge of the neck, and hold it there for a second or two. It is very probable that a small crack will be thus started, but if not, it may with certainty be produced by removing the heated rod, and immediately touching the spot lightly with a moistened finger or splinter of wood. The crack once begun, press the red-hot end of the rod on the glass a little in front of it, Fig. 25, when it will at once extend itself to the heated spot; and by slowly drawing the rod in the direction required, re-heating it from time to time in the blowpipe-flame, the crack may be led at first a short distance downwards, and then, turning at right angles, horizontally round the neck so as to cut off a ring of glass including the uneven portion. It is generally not possible to carry the crack entirely round the glass until it returns into itself; a small portion of the glass will remain undivided, but after laying down the rod the ring may be readily pulled off, a very slight inequality marking the point where the crack was incomplete.

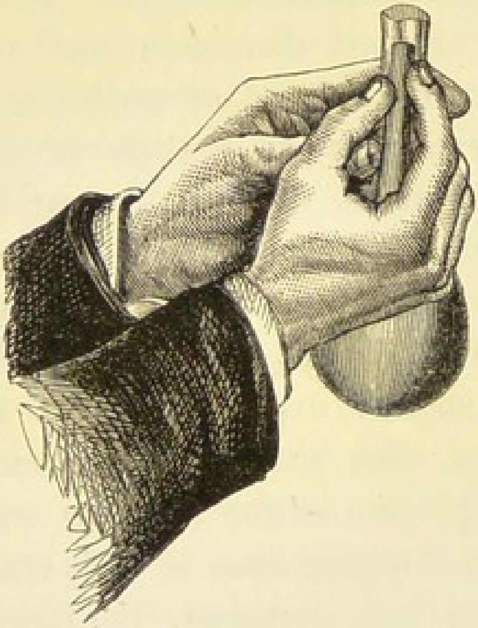

Fig. 25.

A rather better form of termination for the rod, especially when the glass to be cracked is somewhat thick, is the following, Fig. 26. It is made by heating strongly about 5 or 6 mm. of the end of the rod, and then, the rod being

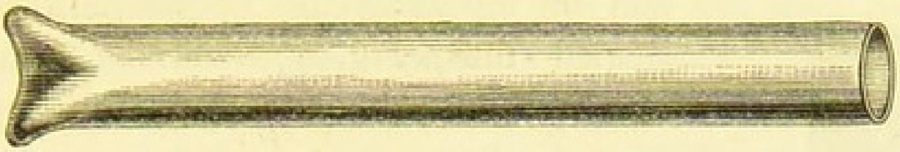

Fig. 26.

held in a slanting direction, pressing it down on any flat metallic surface. The glass will spread out laterally, and by turning the rod half round and pressing it down again in the same way the desired chisel-shape will be obtained. In using it the edge should be kept in the line of the crack required, and moved along the surface with slight pressure. The advantages of this form are: 1st, That more of the glass in the desired line is heated at once, so that the crack extends more rapidly, yet under perfect control; 2ndly, That it is easier to lead the crack in an unswerving straight line, the eye being guided by the line of the edge of the tool, than when a blunt point only is used, precisely as a carpenter finds it easier to cut the side of a mortice by a broad chisel than by a narrow one.

Having thus rendered the neck of the flask even, it will be desirable not only to fuse the edge in the flame, but to turn it slightly outward so as to form a border like that of a bottle, which will give it much greater strength when a cork is to be fitted to it. To do this a very simple tool must be first prepared. Select a piece of sound charcoal, free from fissures, about 3 cm. in diameter, and 8 or 9 cm. long. Cut and rasp the end of this to a point like that of a pencil, but more obtuse, the angle at the apex being nearly a right angle. Take the flask in the left hand, and cautiously heat the extreme edge of the neck in the blowpipe-flame until it softens, turning it constantly round and holding it at right angles to the flame so that the latter may play across the mouth and heat two opposite sides of the glass at once.

When the glass begins to soften and sink inwards, take the pointed charcoal in the right hand and press it gently with a twisting motion into the neck. The edge will spread out, and by repeating the operation a border shaped thus, Fig. 27, will be obtained, which should be annealed with especial care.

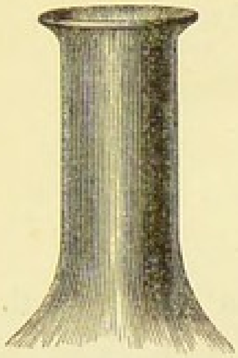
Fig. 27.

Test-tubes, &c. may be bordered in the same way, but it is best to use a spirit lamp or Bunsen's burner, and not the blowpipe, for heating the edges, on account of the thinness of the glass.

EXERCISE 4.

Solution, Evaporation, and Crystallisation.

Apparatus required—Porcelain mortar and pestle; scales and weights; flask, about 300 c.c. capacity; glass measure; retort-stand; sand-bath, with sand; argand, or spirit lamp; funnel, 7 cm. in diameter; filters, 14 cm. in diameter; beaker; porcelain dish, 12 cm. in diameter; watch-glass; glass rod; crucible tongs; washing bottle filled with distilled water; cloth; writing-paper; blotting-paper; alum; sodium sulphate.

Place the mortar on a clean sheet of paper, and put into it two or three lumps of common alum. Reduce these to a coarse powder by first striking them with the pestle until they are broken up into small pieces and then completing the pulverisation by rubbing these fragments with a circular movement of the pestle (not unlike the motion used in stirring a liquid, but combined with downward pressure), occasionally shaking down to the centre portions of the salt which adhere to the sides of the mortar. When this is done, take two pieces of writing-paper about 14 cm. square, turn up two opposite sides of each, so as to form a trough, and place one in each scale of the balance; they should then be found to counterbalance each other, on raising the beam. Into one scale-pan put the 50 grm. weight, and into the other bring some of the powdered alum, using a common bone spoon or spatula for transferring it. Continue to add the salt until the beam turns, then remove any excess of the powder little by little, until the beam remains level, showing that there is the same weight in each scale. Now take out of the scale the paper with the powder in it, and transfer the latter to a flask thus, Fig. 28.

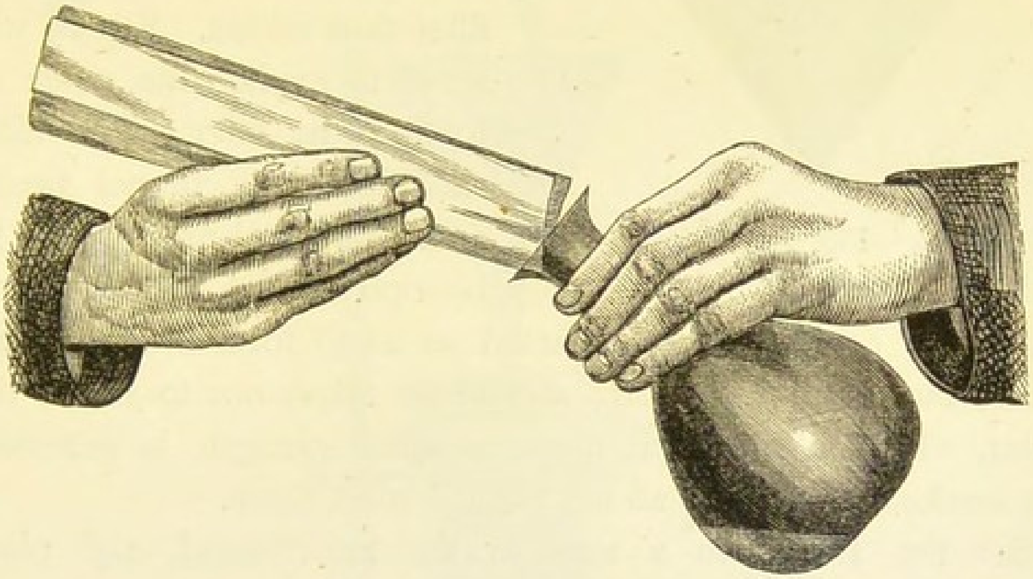

Fig. 28.

Place the measure on a level table, and pour distilled water into it until the lowest part of the curved surface of the water is seen to touch the division which marks 200 c.c. The eye should be placed on a level with this division and neither above nor below it, or it will be impossible to obtain a correct measurement. Pour the measured water into the flask and set the latter on the sand-bath, in which should be placed enough sand to form a stratum about 1 cm. deep, or rather less. Support the sand-bath on the largest ring of the retort-stand, and place under it a gas or spirit lamp with a moderately large flame.

While the solution of the alum is going on, you may get ready a filter, or strainer, to separate any particles of dirt which may be in the liquid. Take a circular piece of filtering paper about 14 cm. in diameter, fold it in half, and then again at right angles to the first fold, so that the circle is reduced to a quarter-circle consisting of four thicknesses of paper. Open this out, so as to form a conical cavity having three folds of the paper on one side and one on the other; and place it in a funnel slightly larger than the filter thus folded. Fig. 29 will serve to explain the mode of folding the filter. The filter should fit the funnel pretty accurately, and may require to be opened out a little more, or, on the other hand, contracted so as to form a more acute cone; but in all cases care should be taken not to injure the point, which, although it requires most strength, is generally the weakest part, since all the creases meet there.

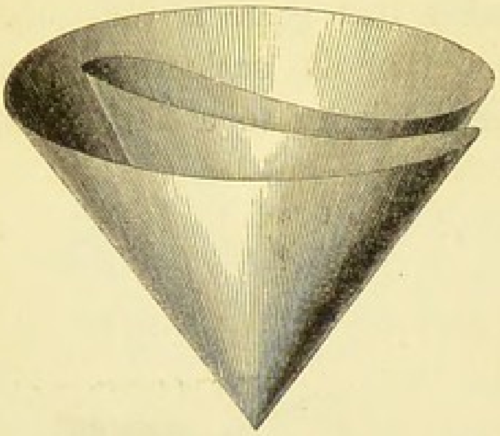

Fig. 29.

Set the funnel in a ring of the retort-stand, and place under it a beaker or other vessel. Pour a little distilled water upon the filter, directing the stream not into the point, but down the thicker part of the side, and allow the water to drain off into the beaker. Meanwhile see if the whole of the alum has by the aid of the heat dissolved in the water, and if not, shake the flask to bring fresh portions of the liquid into contact with the undissolved portion. When the solution is complete, take away the lamp; take the flask out of the hot sand and put in its place a porcelain dish; turn round the ring holding the funnel, raising or lowering it if necessary, until the tube of the funnel just touches the side of the dish near its rim. Now, grasping the neck of the flask with a cloth, pour its contents along a glass rod, Fig. 30, so as to fall on one side of the filter; pouring slowly at first, until the filter becomes fully saturated with the solution, but afterwards keeping the filter nearly full. When all the liquid has run through, remove the funnel and filter, and replace the lamp in order to evaporate the solution, i. e. to drive off the water until only enough is left

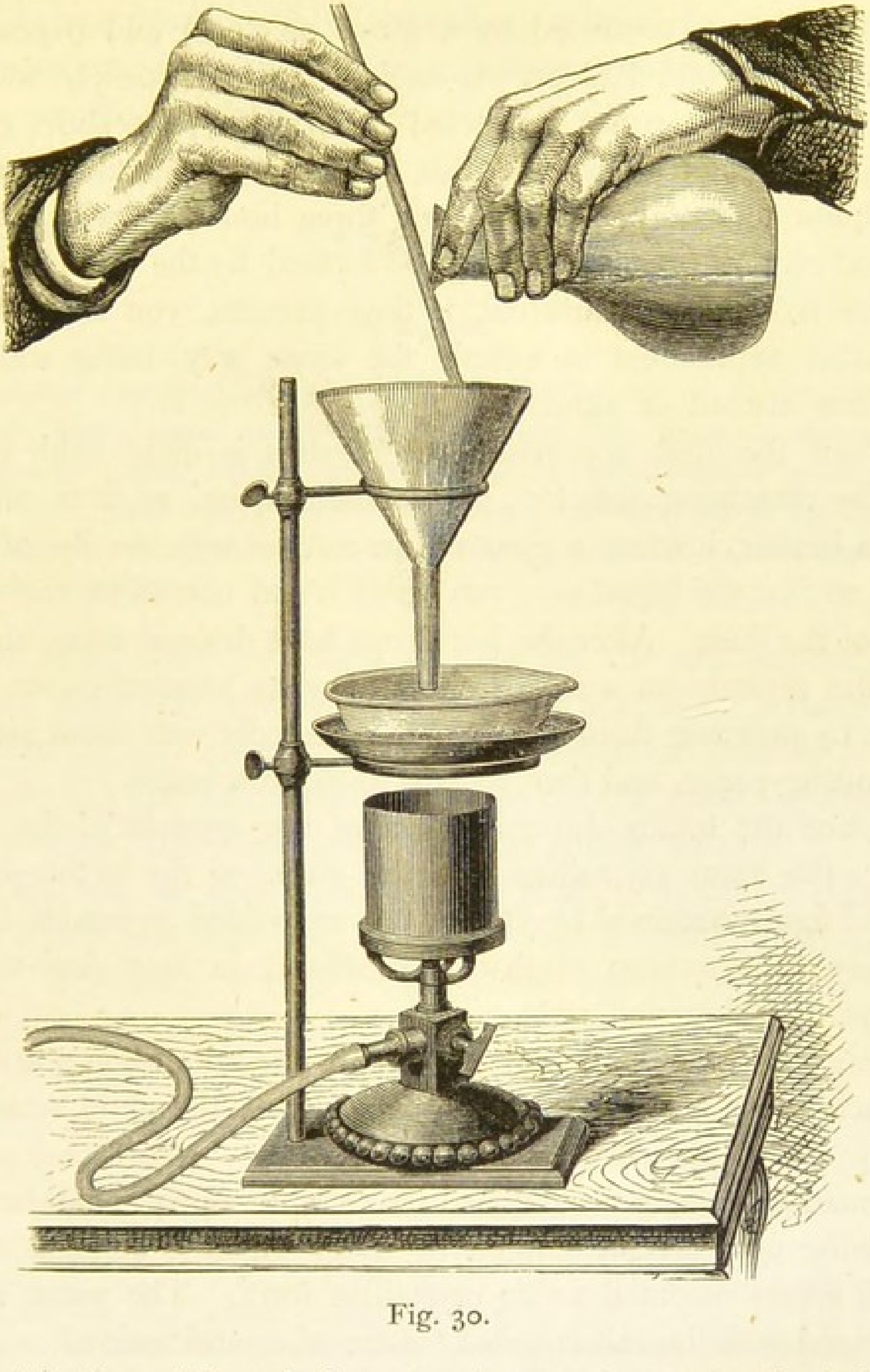

Fig. 30.

to retain the salt in solution at a temperature near the boiling-point. The liquid is then said to be 'saturated' at that temperature. To see when this is the case, dip from time to time a clean glass rod into the solution and place a drop of the liquid on a glass plate or watch-glass. If it deposits minute crystals on cooling, the proper point has been reached. The lamp should then be removed, and the porcelain dish taken carefully from the sand with a pair of crucible tongs (or with the fingers protected by a glove or cloth) and deposited on a folded cloth laid on the table. Cover it loosely with a piece of blotting-paper supported underneath by a short glass rod or tube laid across the dish. The whole should now be left quite undisturbed for two or three hours, in which time a good crop of crystals ought to be formed by the slow cooling of the solution. Meanwhile, if time permits, you may make a similar experiment in exactly the same way, using sodium sulphate instead of alum.

When the dish containing the crystals is quite cold, pour off the remaining solution, or 'mother liquor' as it is called, into a beaker, holding a glass rod in contact with the lip of the dish, so that the liquid may run down it and not down the outside of the dish. After the last drops have drained away, shake out the crystals on a folded sheet of white blotting-paper, dry them by pressing them gently and repeatedly with fresh pieces of blotting-paper, and then transfer them to a bottle.

Notice the totally different form of the crystals of the two salts; the alum crystallising in the form of the octohedron, a solid figure obtained by joining two four-sided pyramids base to base; the sodium sulphate crystallising in long four-sided prisms, like flattened rods.

Notice also the different behaviour of the salts when a crystal of each is exposed to the air for some time: the alum remains nearly unaltered, while the sodium sulphate 'effloresces' as it is termed, or becomes converted into a white opaque substance, by giving up a certain amount of water which it contains, and which seems essential to its crystalline form. The water thus combined with the salt is called 'water of crystallisation.'

Mix in a clean porcelain dish the solutions of alum and sodium sulphate which were poured off the crystals, evaporate the mixture, as before, on the sand-bath, and leave it again to crystallise. You will now obtain a crop of crystals of both salts, and each will be found to have crystallised in its own characteristic form. If, however, the evaporation has not been carried very far, the alum (which is the least soluble of the two salts in cold water) will crystallise alone. This method, of partial crystallisation, is continually employed for the purpose of separating a crystallisable salt from impurities which are more soluble than itself.

EXERCISE 5.

Solution.

Apparatus required—Retort-stand; sand-bath and sand; argand, or spirit lamp; scales and weights; porcelain dish, 12 cm. in diameter; stoppered bottle, holding about 600 c.c. (a wide-mouthed gas bottle will do); glass rod; two watch-glasses; test-tube, about 1.5 cm. in diameter; writing-paper; cloth; washing bottle filled with distilled water; quicklime.

The salts which you have selected for the last experiment are not only very soluble in water, but also more soluble in hot than in cold water[1]. You may take calcium hydrate as an example of a substance which dissolves only in small proportion in cold water, and is still less soluble in boiling water.

Weigh out, as above directed, 10 grms. of good quicklime, place it in a porcelain dish, and pour over it 10 c.c. of distilled water. The lime, if it is freshly burnt, will become very hot and fall to pieces, forming a white impalpable powder. The water disappears entirely, its elements having united with the elements of the quicklime to form a single substance, calcium hydrate[2].

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[1]

	at 0°,	at 33°,	at 100°,	
100 c.c. of water dissolve	5.22 ;	22 ;	421	grms. of alum.
" " "	12.0 ;	322 ;	244	" of sodium sulphate.

Hence we see that, while sodium sulphate is more soluble in hot than in cold water, its solubility does not increase regularly with the temperature, reaching a maximum at 33°, and then slightly decreasing.

[2] The evolution of heat is due to two causes:—(1) The chemical combination which is taking place. It is a good example of the universal law that heat is evolved during chemical combination. (2) The fact that a liquid (water) is entering into combination, while a solid (calcium hydrate) is the sole product. For in all cases in which a liquid becomes a solid, heat is evolved.

Shake out the calcium hydrate upon a half-sheet of writing-paper, and transfer it (Fig. 28) to a stoppered bottle holding about 600 c.c. Fill the bottle nearly to the neck with distilled water, and, after inserting the stopper, shake the liquid for a minute or two, then leave it undisturbed for a quarter of an hour. You will notice that the salt does not dissolve, like the alum, to a clear fluid (although you have added thirty times its weight of water), but that the greater part of it subsides to the bottom of the bottle. Leave the bottle until the next day, occasionally shaking it thoroughly. You will find that even then there is a large quantity of calcium hydrate remaining undissolved; indeed, you will require some positive proof that any of it has been dissolved.

Take out the stopper, wipe the inside of the neck of the bottle with a clean cloth, and pour some of the liquid into a test-tube, inclining the bottle very gently so as to avoid disturbing the sediment. Hold the test-tube up to the light, in order to see if there are any solid particles floating in the liquid. If such is the case, instead of filtering the solution, it will be better to return it to the bottle and to allow the latter to remain undisturbed for a few hours longer, while you proceed to other experiments. When you have obtained a clear liquid, you may try the following experiments with it:—

1. Dip a clean glass rod into the liquid and taste what adheres to the rod. You will find that it is not tasteless, like pure water, but has acquired a sharp caustic taste.

2. Pour a few drops into a watch-glass; place the latter upon the sand-bath supported, as before, on a ring of the retort-stand, and evaporate the liquid to dryness. For the sake of comparison you may evaporate in another watch-glass (placed on the same sand-bath) a little of the distilled water which you have been using. When all the water has been driven off, you will find that in the former case a solid white residue is left on the watch-glass, in the latter case there is no residue whatever, or at all events a mere trace. The occurrence of such a residue is a conclusive proof that something has been dissolved by the water.

3. While the evaporation is proceeding, you may heat over a spirit lamp the test-tube containing the remainder of the solution of calcium hydrate.

The operation of heating a test-tube in the naked flame requires some care, otherwise the tube may crack, or its contents may be thrown out, owing to the sudden formation of large bubbles of vapour at the bottom of the tube. Hold the tube in a slanting position, turning it round between the fingers and moving it to and fro in the flame[1], so as to distribute the heat over as large a surface as possible. Remember, however, that no heat must be applied to the part of the tube above the level of the liquid; otherwise, if the colder liquid should reach this part, the tube will be almost certain to crack. When the liquid has nearly reached the boiling-point, remove the tube from the direct flame and hold it at the side or above the flame, occasionally shaking it to promote the formation of bubbles of vapour.

Observe that the liquid, as the temperature rises, becomes milky, a solid substance being formed in it. This is owing to the fact that calcium hydrate is much less soluble in hot than in cold water[2]. A solution, therefore, of calcium hydrate, which is saturated at the ordinary temperature, will deposit a portion of the substance when it is heated. Cork the tube and cool it by immersing the lower portion of it in cold water, shaking it occasionally. The turbidity will, after some little time, disappear: the calcium hydrate being again dissolved.

Pour off the rest of the solution of calcium hydrate into a clean bottle, inclining the bottle which contains the solution very gently, in order to avoid disturbing the solid substance at the bottom. This operation is called 'Decantation,' and will be again alluded to in the next Exercise. Be careful to cease

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[1] If the tube becomes too hot to be held between the fingers, a simple and convenient holder is obtained by folding a sheet of writing-paper into a band about 2 cm. in breadth. This should be passed round the tube near its mouth, and held (close to the tube) between the forefinger and thumb. The jaws of a Bunsen's holder, Fig. 3, taken out of the socket, also form an excellent holder.

[2] 100 c.c. of water dissolve at 15°, 0.173 grms.; at 100°, 0.083 grms. of calcium hydrate.

pouring before any turbid solution finds its way into the second bottle; close the latter with a good cork or stopper, and, after placing a label on it, put it away for use in future experiments.

EXERCISE 6.

Precipitation and Filtration.

Apparatus required—Retort-stand; sand-bath with sand; argand, or spirit lamp; scales and weights; glass measure; two flasks of about 300 c.c. capacity; two funnels, 7 cm. and 10 cm. in diameter; filters; beaker, 7 cm. in diameter; glass rod; test-tube; washing bottle with distilled water; writing-paper; blotting-paper; sodium carbonate; barium chloride.

Make a solution of sodium carbonate, in the manner described in Exercise 4, by heating 15 grms. of the crystallised salt with 100 c.c. of distilled water in a flask. Make a solution in a similar manner of barium chloride, containing 12 grms. of the salt dissolved in 100 c.c. of distilled water. Filter the solutions if they contain any floating insoluble particles. Pour the solution of barium chloride into a beaker, and add to it the whole of the solution of sodium carbonate. As soon as the two solutions come into contact, a dense white cloud will be formed in the liquid, and a white powder will gradually settle to the bottom of the beaker. This production of a solid by the action of two salts in solution upon each other is called 'Precipitation,' and the solid thus formed is called a 'Precipitate.' In the present instance the precipitate consists of barium carbonate, and the liquid above the precipitate is a solution of sodium chloride, containing also the excess of sodium carbonate. In order to separate the precipitate from the solution, we have recourse to the process of filtration described in Exercise 4.

Place a filter, about 20 cm. in diameter, folded as there directed, in a funnel about 10 cm. in diameter, taking care that the filter fits the funnel closely and does not project beyond its rim. Wet the filter with distilled water and allow it to drain for a few moments; then support the funnel in a ring of the retort-stand, place under it an empty flask, and pour the liquid from the beaker down the side of the filter, using a glass rod to direct the stream, and taking care not to disturb the precipitate more than you can help. When the greater part of the liquid has been poured off, fill up the beaker again with distilled water, stir up the precipitate with a glass rod, and leave it to subside. When the liquid above the precipitate is tolerably clear, pour it off again into the filter, still retaining the bulk of the precipitate in the beaker. Repeat this operation of filling the beaker with water, allowing the precipitate to subside, and then pouring off the clear liquid, two or three times. This process is called 'washing by decantation,' and is especially adapted for cases where we have a powdery, quickly-subsiding precipitate. Finally, transfer the whole of the precipitate to the filter, by stirring it up with a little water, pouring it quickly into the filter and again rinsing the beaker with more water.

It may happen that the first portions of the liquid which run through the filter are turbid. If this is the case, they should be poured back into the filter, another flask or beaker being placed to catch the fluid. If however the filtrate is still turbid, there is reason to suspect that there is a hole in the filter itself. In such a case it will be best to return the precipitate to the flask by making a large hole through the apex of the filter, by means of a glass rod pressed vertically downwards, and washing down the precipitate by a strong stream of water from the washing bottle. Another filter should then be fitted to the funnel and the precipitate transferred to it as before. The washing of the precipitate must now be completed on the filter, in order to free it from all traces of the solutions from which it was formed. This is done by pouring over it a gentle stream of water from the jet of the washing bottle, until the funnel is nearly, but not quite, full, the level of the water being on no account allowed to rise above the edge of the filter. When the washing water has entirely run through the filter, you may pour on a fresh supply, taking care to direct the stream on the sides of the filter so as to wash the precipitate towards the centre. In order to ascertain when the washing has been carried far enough, you must examine the liquid which runs through the filter into the flask, by adding to it a few drops of a solution of barium chloride, made by dissolving in a test-tube one or two crystals of the salt in a little water. If the addition of this produces a turbidity in the filtrate, it is a proof that there still remains some sodium carbonate adhering to the precipitate, and the washing must be continued until the filtrate remains quite clear even on heating with the barium chloride. When this is the case, cover the funnel with a piece of filter-paper turned down at the sides so as to form a cap, and set it aside to dry in a warm place. The barium carbonate will, in drying, contract and separate from the filter: it may be shaken into a mortar, crushed to powder, and kept in a bottle for use in analysis.

EXERCISE 7.

Distillation.

Apparatus required—Plain retort, about 200 c.c. in capacity; retort-stand; sand-bath with sand; argand, or spirit lamp; thistle funnel; Florence flask; porcelain mortar; Bunsen's holder; funnel, 10 cm. in diameter; beaker; test-tube-stand and six test-tubes; watch-glass; washing bottle with distilled water; wooden blocks; blotting-paper; lamp-cotton, or tow; cloth; solutions of barium chloride, silver nitrate, ammonium oxalate, hydrogen nitrate (dilute).

Select a plain retort with a long neck; lay a folded cloth upon the table and place the retort upon it, supporting its neck in a vertical position by passing it through the smallest ring of the retort-stand. Pour common water into the retort until it is rather more than half full, using a thistle funnel with long tube passed down the neck so as to avoid wetting that part as far as possible. When this is done, remove the funnel and place the body of the retort upon the sand-bath supported upon the largest ring of the retort-stand at such a height as to allow the lamp to be placed under it. Pass the beak of the retort into a clean Florence flask, placed in a slanting position in the porcelain mortar, and support the latter on wooden blocks at such a height that there may be a gradual fall from the point at which the neck joins the body of the retort to its outer extremity. Take a piece of blotting-paper about 18 cm. in breadth and about three-fourths as long as the neck of the retort; fold two of its adjacent corners inwards so as to give a wedge-shaped outline to the paper, wet it thoroughly, and place it over the neck of the retort, so that while the narrower part of the paper lies upon the broader part of the neck, the narrower part of the neck may be completely enveloped by the paper, the two unfolded corners hanging down some way below it. Next, take a strip of blotting-paper about 10 cm. in breadth and somewhat shorter than the first strip, fold it, in the direction of its length, into the shape of the

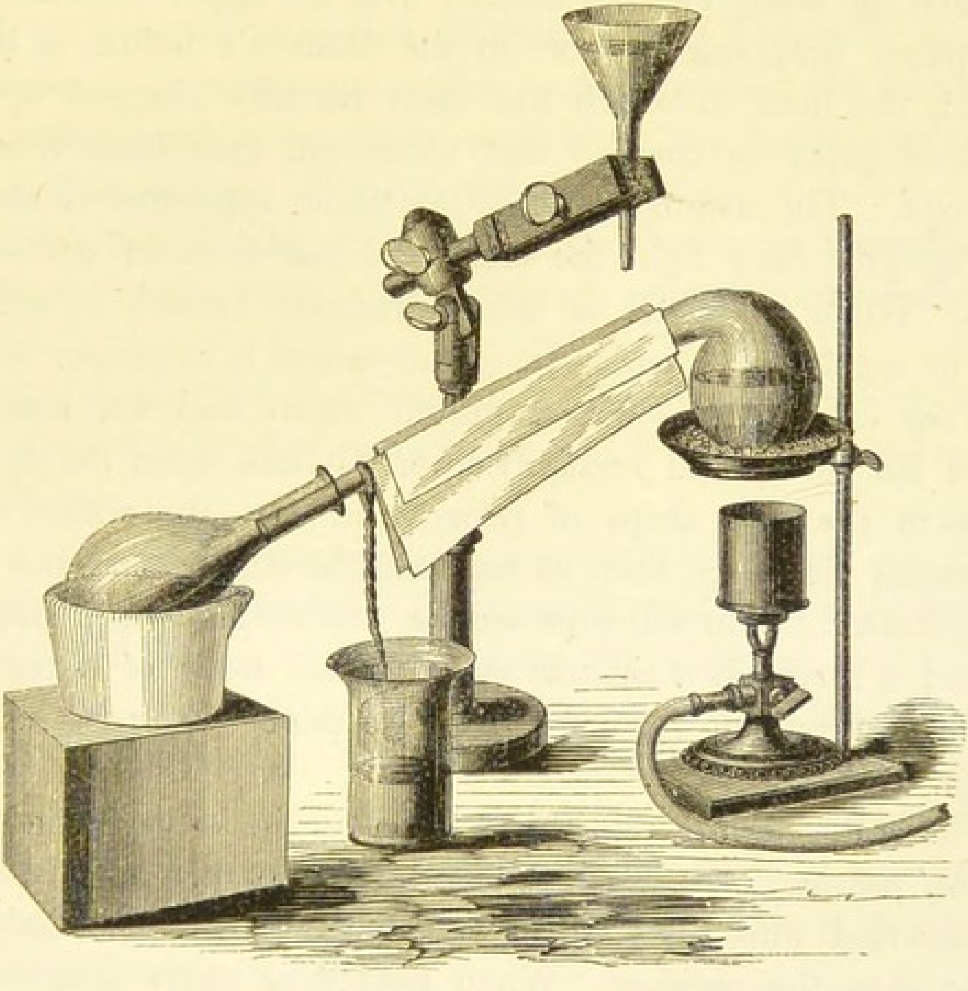

Fig. 31.

letter M, and place it like a saddle upon the first wrapper. Just beyond the lowest end of the folds of paper, twist round the neck of the retort a few strands of lamp-cotton or of tow, thoroughly wetted, the end of which should hang down 10 or 12 cm. The object of this is to prevent the water which trickles down the paper from passing any further down the neck of the retort and entering the receiver. Underneath the end of this piece of cotton is to be placed a beaker or basin to catch the waste water. Directly over the upper extremity of the paper wrapping support in the Bunsen's holder a large funnel, the neck of which has been partially plugged up by a bit of paper or tow, so that water will only issue from it in drops. The arrangement of the whole apparatus is shown in Fig. 31. Now light the lamp and put it under the sand-bath. While the water is being gradually heated, it will be well to see that the condensing arrangement is in proper order. Fill the large funnel with common water, and see that the drops fall slowly and regularly from its beak upon the uppermost of the two strips of paper, and that the water, after saturating the paper, finds its way into the beaker, and not into the receiver. You will now see the use of the upper strip of paper; it serves as a channel to guide the drops of water and to distribute them regularly over the paper wrapping underneath.

The water in the retort will soon begin to boil, and the steam, passing over into the colder parts of the neck, will be reconverted into water which will collect in drops and run down into the receiver. When twenty or thirty drops have thus distilled over, withdraw the receiver (holding the neck of the retort steady with one hand), and pour away the liquid which it contains; since the first portions which come over are liable to contain impurities derived from the neck of the retort. When the last drop has drained away, replace the receiver in its former position and continue the distillation until about three-fourths of the water in the retort has passed over. Pour, from time to time, some cold water into the large funnel so as to keep it nearly full. Be particularly careful to regulate the heat so that the water may not boil so violently as to splash over into the neck of the retort, and thus be carried down into the receiver. If, in spite of care, it boils unsteadily, with 'bumping,' owing to the sudden formation of large bubbles of steam in contact with the more strongly heated portions of the retort, it will be advisable to remove the lamp, to wait until the temperature of the water has sunk a few degrees, and then, having withdrawn the receiver, to push down with a glass rod into the body of the retort a fragment or two of charcoal, or a bit of crumpled platinum foil. This will, in consequence of its numerous angles, materially assist the formation of smaller bubbles of steam, and when the lamp is replaced the boiling will go on more quietly.

While the distillation is going on, you may examine the qualities of the water which you are using, in the following way:—

Place three clean test-tubes in the stand, and fill them about one-third full of the same water which you have taken for distillation.

(*a*) Add to the contents of one test-tube five or six drops of dilute hydrogen nitrate (nitric acid), and then one drop of solution of barium chloride; shake the test-tube, and hold it up to the light. If a white cloudiness or precipitate is produced, it is a proof that the water contains a sulphate.

(*b*) Add to the contents of another test-tube five or six drops of dilute hydrogen nitrate, and then one drop of solution of silver nitrate. If a white cloudiness or precipitate is produced, the water contains a chloride.

(*c*) Add to the contents of the remaining test-tube three or four drops of solution of ammonia, and then one drop of solution of ammonium oxalate. If a white cloudiness or precipitate is produced, the water contains a calcium salt[1].

Before the water in the retort has wholly evaporated, you

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[1] If the water which you have used is fresh spring water, and if, from experiment *c*, you have discovered that a calcium salt is present, you will probably find that, as the distillation proceeds, the water in the retort becomes turbid, and a white deposit is formed. The reason of this will be explained in Sect. 2, Ex. 6.

should stop the distillation by removing the lamp, and withdraw the receiver, resting the beak of the retort temporarily upon the edge of the mortar. You have now to examine the water which has collected in the receiver, in order to see if the process of distillation has freed it from impurities.

Place three clean test-tubes in the stand, pour into each some of the distilled water, and repeat experiments *a*, *b*, *c*. The water should give no cloudiness or precipitate in any of the above experiments, and if a few drops are poured into a clean watch-glass and evaporated to dryness on the sand-bath no solid residue should be left; otherwise it is most likely that the water in the retort has boiled too rapidly, and a portion of it has splashed over, or has been carried over as spray into the receiver.

The retort and the rest of the apparatus should now be cleaned and put away for future use. If there is any deposit in the retort which cannot be removed by water alone, a few drops of dilute hydrogen chloride (hydrochloric acid) will at once dissolve it[1].

EXERCISE 8.

Use of the Pneumatic Trough.

Apparatus required—Pneumatic trough; two cylindrical gas jars, 5 × 18 cm.; one ditto, 3 × 10 cm.; jug of water; cloth.

Fill the trough with clean water up to the level of the overflow-pipe, or about 2 cm. above the shelf, and place it before you on the table, the broad fixed ledge being farthest from you, and the movable shelf being placed near the left-hand

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[1] It will be scarcely worth your while to attempt to distil for yourself all the water you will require. You will in all probability be able to obtain it at the nearest chemist's shop at a cost of 2*d*. or 3*d*. per quart. But the water which you buy should in all cases be examined by the above tests, and rejected at once if any impurity is found in it.

end of the trough. Take one of the larger cylindrical jars, and, holding it nearly horizontally in the right hand, plunge it into the water, the mouth of the jar being held a little higher than the closed end, in order that the air may more readily escape. When the jar is filled with water and wholly immersed in the trough, bring the closed end uppermost and raise the jar vertically until its mouth is on a level with the shelf, and then move it laterally until it rests over the hole in the shelf: taking care to keep the mouth always below the surface of the water. So long as this condition is fulfilled, you will find that the jar remains full of water, the column of water being retained in it by the pressure of the air on the surrounding water. Next, take a similar jar, holding it vertically, mouth downwards, the closed end being grasped by the right hand, and bring its mouth about 3 or 4 cm. below the water-level in the trough. It will now represent a jar full of gas, which is to be transferred or decanted into the other jar. This is effected by a manipulation quite analogous to that by which we pour water from one vessel to another, the only difference being due to the fact that instead of pouring water downwards through air, we have to pour air upwards through water. Hold the jar containing water steady with the left hand, bring the mouth of the other jar under the hole in the shelf, and gradually depress its closed end so that the air contained in it may ascend bubble by bubble through the hole into the jar placed over it. The level of the water in the latter will fall until the jar is completely filled with air. The reason for steadying it with the left hand will now be evident, since it will show a tendency to topple over owing to the upward pressure of the water; and it is best never to fill a jar completely with gas, but only so far that the level of the water inside and outside the jar may be the same. It can easily be filled completely, when required, from another jar.

Repeat the experiment, decanting air from the one jar into the other until you can do so without allowing a single bubble to escape. It is not necessary that the jar should rest on the shelf, but it may be held in the left hand, as in Fig. 35, p. 71, its mouth being retained about 2 cm. below the water-level, and the mouth of the jar of air brought close to its edge, and a very little to the right of it, since the bubbles of gas have a forward as well as an upward direction. You may, in the next place, fill the small jar with water and decant air into it from the larger one. This will not be found so easy, since the bubbles of air from the mouth of the jar are so large that unless the jars are held steady and the decantation is very gradual a waste of gas is likely to occur, and must occur if the disproportion between the mouths of the jars is very great. In such a case, as when gas is to be transferred to a test-tube, it is better to decant it first into a jar of intermediate size, and then into the tube; or, the object may be effected in the following way. Fill the tube in the usual way with water, and insert into its mouth, still held downwards below the water-level, the up-turned beak of an inverted funnel about 7 cm. in diameter. Retain the funnel in its position by placing the little finger under the rim, the tube being held upright between the thumb and the other fingers. If, now, air be decanted from a larger jar, bubble by bubble, into the funnel, the bubbles will rise into the tube without any loss, the displaced water making its escape between the mouth of the tube and the funnel, which latter should not be held too closely in contact with the former.

Having thus learnt the general method of using the pneumatic trough, you will proceed, in the next Exercise, to employ it for collecting and experimenting upon oxygen gas.

Before, however, putting away the trough, you may examine the alteration in volume which gases undergo at different temperatures, in the following way:—

Take a test-tube about 2.5 cm. in diameter and at least 20 cm. in length; place round it, about the middle, a small india-rubber band; fill it with water, and place it inverted upon the shelf of the trough. Decant air into it from a small test-tube until, when your eye is brought on a level with the india-rubber ring, the lowest part of the curve which is formed by the surface of the liquid appears *just* to touch the upper edge of the ring[1]. Heat a little water in a test-tube nearly to boiling, and pour it over the tube containing the measured volume of air. The surface of the water in the tube will rapidly sink below the ring, showing that the air has expanded with the heat; but when a little water at the ordinary temperature is poured over the tube, the air will contract to its original volume. If you can obtain any ice or snow, you may place some in a flask and add some water; when the latter has been cooled down nearly to the freezing-point it should be poured over the tube containing the air. The surface of the liquid will now rise above the ring; the air contracting in volume as the temperature is lowered. The same experiment might be tried with any other gas, and you would find it to be a universal law that *the volume of a gas increases as its temperature is raised, and decreases as the temperature is lowered.*

The relation of the volume of a gas to pressure is not quite so simply demonstrated at the pneumatic trough, since it is impossible to effect much variation in the length of the column of water which gives the pressure; and hence only a small, but still perceptible, difference in volume is obtained. The experiment may, however, be made in the following way. Fit a good cork to the large test-tube used in the last experiment: bore a hole in it (p. 32), and fit into the hole a straight piece of glass tubing about 20 cm. in length, and 6 or 7 mm. in diameter. Take out this tube, when fitted, and insert the cork tightly into the test tube. Slip over the small tube a narrow india-rubber ring (cut from a piece of tubing), and sink it vertically in the trough almost to the bottom; then, holding the test-tube by the rim (lest the warmth of the hand should alter the volume of the enclosed air), fit the small tube into the cork, raise or lower the apparatus until the level of the water in the tube and in the trough is the same, and adjust the ring to this level. The air enclosed in the tube is now at the same pressure

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[1] This is the best mark at which the volume of a gas or liquid can be read, and the accuracy of the reading may be increased by holding just below the surface of the fluid, on the opposite side of the tube or jar, a strip of black paper pasted along the lower half of a card. The surface-curve then appears to be black and bounded by a well-defined line.

as the external air; both pressing on the surface of the water with the same force. Owing to the difference in diameter between the large and the small tube, a slight alteration in the volume of the air in the former will cause a considerable difference in the level of the water in the latter. Now slip under the extremity of the tube a gas jar, wholly immersed in the trough, and raise the whole out of the trough, the gas jar serving as a deep gas tray (p. 64). Set the jar on the table, and raise the tube until its extremity is only just below the water-level: you will observe that the water in the tube falls below its former level, showing that the volume of the air in the apparatus has increased. This enclosed air is obviously under less than the whole pressure of the external air, since the pressure of the latter is partly balanced by the column of water in the tube. Next, sink the tube in the jar until it touches the bottom: the water will rise above its original level in the tube, showing that the enclosed air has contracted in volume. This air is now exposed to more than the pressure of the external air, the latter being aided by the column of water in the jar which is above the level of the water in the tube. We have thus an evidence that *the volume of a gas increases as the pressure to which it is exposed is lessened, and decreases as the pressure is augmented.* By measuring the pressures to which gases may be subjected, and the corresponding volumes, it has been proved that *the volume of a gas varies inversely as the pressure.*

SECTION II.

PREPARATION AND EXAMINATION OF GASES.

EXERCISE 1.

Oxygen.

Apparatus required—Pneumatic trough; argand burner, and fish-tail burner, or spirit lamp; retort-stand; sand-bath; piece of wire-gauze; corks; cork-borers; rat-tail file; three-square file; Florence flask; glass tubing, about 6 or 7 mm. in external diameter; porcelain mortar; two small porcelain dishes; scales and weights; sheets of writing-paper; deflagrating jar; deflagrating spoon; two wide-mouthed stoppered bottles, holding about 700 c.c.; three ditto, holding about 300 c.c.; cylindrical gas jar, 18 x 5 cm.; ground-glass disc, 8 cm. in diameter; taper on wire (Fig. 9); cedar matches; pieces of watch-spring; German tinder, or fusee; crucible tongs; jug of water; blotting-paper; potassium chlorate; manganese dioxide; sulphur.

In the first place the materials for the preparation of the gas should be got ready, as it is important that they should have time to dry thoroughly. If this precaution be neglected, the flask in which they are to be heated is not unlikely to crack during the experiment, owing to the moisture condensing in the neck and dropping down upon the hotter portions of the glass. When potassium chlorate is heated it fuses, and at a higher temperature gives off all the oxygen which it contains[1]. A very pure gas may be obtained by these means, but the temperature at which the decomposition takes place is so high that ordinary glass flasks cannot support it without softening. It is found, however, that if the salt be mixed with manganese dioxide, the decomposition takes place at a much lower temperature, and with greater rapidity. The manganese dioxide does not, apparently, give up any of the oxygen which it contains:

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[1] $2\,KClO_3 = 2\,KCl + 3\,O_2$.

at any rate, it is found at the close of the action to be unaltered in composition, and may be employed repeatedly with fresh portions of potassium chlorate.

Weigh out, in the manner directed in Exercise 1, 25 grms. of crystallised potassium chlorate; crush the crystals in a mortar, but do not reduce them to powder; then place the salt in a porcelain dish on the sand-bath to dry.

Weigh out, similarly, 6 grms. of manganese dioxide, and place it also in a porcelain dish on the sand-bath near the potassium chlorate, stirring them both occasionally with a glass rod.

In the next place, you have to prepare an apparatus for generating and collecting the gas. Since heat is required to decompose the potassium chlorate, a flask supported in a retort-stand will serve to contain the materials; and since oxygen gas is scarcely soluble in water, it may be collected over the pneumatic trough filled with water; a bent glass tube will serve to convey the gas from the flask to the trough.

Take a clean Florence oil-flask, selecting one which has a smooth, even mouth; roughen the sharp edges of the mouth with a file, or, if a Herapath's blowpipe is at hand, turn the edges outwards so as to form a spreading lip, as directed, p. 40. Choose a sound cork very slightly larger than the neck of the flask, squeeze it until it becomes soft and elastic, and bore a hole through it (p. 32) for the delivery tube, using a cork-borer which is rather smaller than the glass tubing which you are going to use.

Cut off a piece of the glass tubing, about 65 cm. in length, and hold each extremity in the flame of a Bunsen's burner until the edges begin to fuse, and have lost their sharpness. Remember to heat the glass gradually, and to turn it continually round while it is in the flame. In the next place, bend the tube to an acute angle, about 10 cm. from one extremity, according to the directions given at p. 26; and lastly, bend it, but only to an obtuse angle, in the opposite direction, as near the other extremity as possible. The object of this second bend is to facilitate the escape of the bubbles of gas by giving them a forward and upward direction. It will be found convenient to use a cork holder (p. 27), in case the end of the tube should become too hot to be held in the fingers. When the glass is quite soft, hold the tube out in front of you, so that the eye may be in the plane of the first bend; it will then be easy to turn up the end which is nearest to you, so that both the bends may lie in the same plane but in opposite directions. The tube, when finished, should have the form of Fig. 32.

Fig. 32.

The end *a* should now be fitted with gentle pressure and twisting motion into the cork, through which it should pass completely and project slightly at the opposite end. Do not attempt to use much force in pushing the tube into the cork, or you may break the tube and be cut with the splinters of glass; but enlarge the hole with the rat-tail file, until the tube will enter it without much difficulty.

The potassium chlorate and manganese dioxide may now be taken from the sand-bath, and set aside to cool for a minute or two, during which time the pneumatic trough may be filled with water up to a level about 2 cm. above the shelf. It is well to have a jug of water at hand, in case more should be wanted. Take the deflagrating jar, slightly grease the stopper and fit it tightly in its place; then immerse the jar sideways in the water, raising the open end a little, so as to allow all the air to escape. When the last bubble is gone depress the open end, and raise the jar by its neck (taking care to keep the lower end below the water-level), until it can be moved laterally to its place on the shelf. Each of the bottles should be filled with water from a jug, the stopper inserted and held loosely in its place with one hand, while with the other the bottle is inverted, and its mouth brought below the level of the water in the trough, when the stopper may be withdrawn, and the bottle moved to the shelf. It is better not to overcrowd the shelf with bottles and jars, which, when filled with water and inverted, are rather unsteady, and hence the other bottles should, after being filled with water, be placed near the trough, to be brought to the shelf when required.

Now mix the potassium chlorate and manganese dioxide in the mortar with a few circular strokes of the pestle, shake the mixture out on a sheet of paper, and transfer it to the flask. After wiping the neck of the latter, fit the cork with the bent delivery tube into its place with careful pressure, and support the flask on a piece of wire-gauze bent into a shallow cup, and resting on the largest ring of the retort-stand. The smallest ring should be passed over the neck of the flask to secure it in an upright position, and the latter should be fixed at such a height that a lamp may be easily placed beneath it, and that the end of the delivery tube may pass under the shelf of the pneumatic trough, and be about 3 or 4 cm. below the surface of the water.

The whole apparatus will then be arranged thus, Fig. 33.

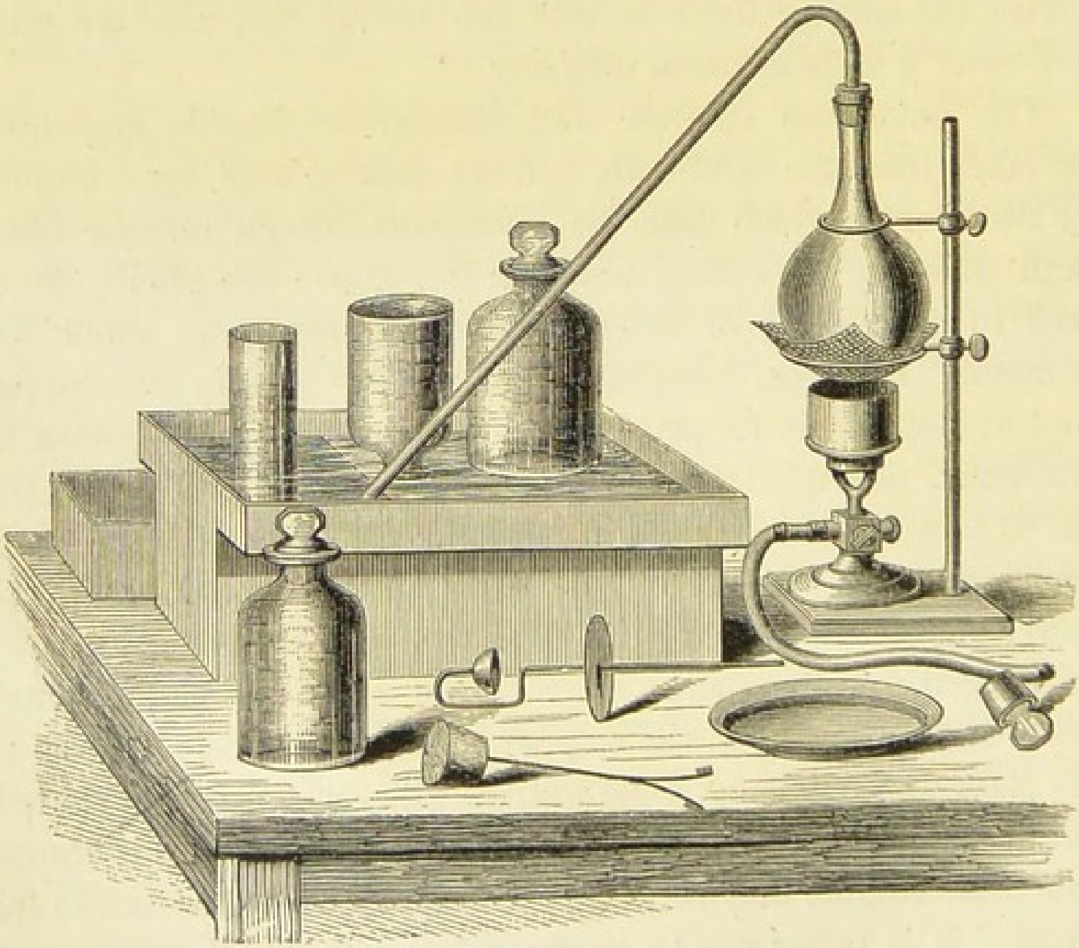

Fig. 33.

Before beginning to heat the mixture in the flask, you should ascertain whether there is any leakage owing to the cork being unsound or badly fitted. Place the warm hand on the flask for a few seconds, and observe whether, owing to the expansion of the air in the flask, the level of the water in the delivery tube is depressed below the level of the water in the trough, and finally a bubble or two of air escapes, and also whether on removal of the hand the water only returns *by degrees*, as the air cools, to its original level in the tube. If no depression of the level takes place, there is a leak in the cork-joint, which must be stopped before anything further is done. If the fault is not cured by pressing the cork farther into the neck of the flask, it will generally be best to take a new cork altogether.

When you have proved that the apparatus is air-tight, you may proceed to heat the flask by a gas or spirit lamp. The best form of gas lamp for the purpose is an argand, or other form of ring-burner, since it distributes the heat over a larger surface, and can be made to give a very small flame if required.

The first effect of the heat (which should be applied cautiously and gradually) will be to expand the air in the flask, which will escape in bubbles through the water in the trough. Fill the cylindrical jar with water and place it mouth downwards on the shelf of the pneumatic trough, in readiness to collect the first portions of gas for the purpose of testing their purity. In a short time the stream of bubbles, which had slackened, will become more rapid, showing that gas is being evolved from the mixture. As soon as this takes place, slide the jar along the shelf until its mouth is over the hole and the bubbles can rise freely into it. The heat must be carefully regulated. A moderately rapid stream of bubbles may be kept up; but on the earliest indication of a more violent evolution of gas, the flame should be lowered, or the lamp withdrawn, until the action subsides. It will be scarcely worth while to test the first jar-full of gas, which will certainly be mixed with air. Fill the jar again with water, and place it as before to receive the gas. When it is full, slide it off the shelf with one hand,

and with the other bring a ground-glass plate upon its mouth, while under water; raise the jar out of the water, still keeping the glass plate pressed against its mouth, and place it mouth upwards on the table. Now light a cedar match at the lamp, blow it out, and while its end is still glowing, plunge it into the jar of gas. If it is re-kindled, bursting sharply into flame, the gas is sufficiently pure for experiments. If this is not the case, return the jar at once to the trough, fill it again with gas, and test it in the same way.

When you have thus ascertained that the gas is pure, you may proceed to fill the bottles with it, bringing each in succession over the hole in the shelf, and when it is full of gas sliding it (without raising its mouth above the level of the water) to one end of the shelf, and bringing another into its place. In order that the pneumatic trough may not be inconveniently crowded with bottles, you may remove them one at a time, when filled with gas, to the table, after inserting the stopper tightly, under water. When the deflagrating jar has been filled, slip under its mouth a shallow tray (a common plate or saucer will answer the purpose); then, keeping the tray horizontal and the mouth of the jar resting in it, raise both out of the water and place them on the table. The water remaining in the plate will thus act as a valve to prevent the gas in the jar from escaping.

It often happens, especially if there is rather more than the due proportion of manganese dioxide in the mixture, that there is a sudden rush of gas at the last, a low incandescence spreading through the half-fused mass; and then the evolution of gas abruptly ceases, thus distinctly marking the termination of the reaction. It is well to be prepared for this, and to take away the lamp at the moment when you perceive any tendency to a rush of gas. Of course the only possible risk would arise from the delivery tube not being sufficiently large to carry off the rapid current of gas, or becoming obstructed by particles of the mixture mechanically carried over with the gas. It is found, however, that all inconvenience and risk from this cause may be obviated by adding to the mixture, before it is placed in the flask, an equal bulk of dry sand. The decomposition then proceeds with perfect regularity from beginning to end, and very little attention to the lamp is required.

As soon as the jar and bottles are filled with oxygen, withdraw the lamp, raise the delivery tube out of the water, and set the flask aside to cool. The porous, half-melted mass which it contains may be readily washed out by a little warm water. It consists of a mixture of potassium chloride and manganese dioxide, and will not be worth preserving. The flask will be found scarcely, if at all, injured, and after being cleaned and dried may be used again for the same purpose.

The properties of the gas should next be examined in the following manner:—

(*a*) Place one of the smaller bottles of gas on the table before you, near the lighted lamp. Loosen the stopper with the left hand; take in the right hand the piece of wax taper attached to a bent wire, and light it at the lamp; then withdrawing the stopper introduce the taper into the bottle of gas. Notice that it is not extinguished but burns with a whiter, more intense flame.

(*b*) Light a splinter of wood (cedar matches are the best for this experiment); when it is thoroughly lighted blow it out, and introduce it, while still smouldering, into another small bottle of the gas. The glowing end will burst quickly into flame, and burn brilliantly.

(*c*) Take two pieces of thin watch-spring (readily obtained from any watch-maker) about 20 cm. in length; soften them by holding them in the lamp-flame until they become red-hot and lose their elasticity: when they are cool, straighten them and lay them together. Push one end of the bundle thus formed into a bung large enough to cover the mouth of the deflagrating jar. Bend up the other end of each bit of spring so as to form a loop, in which place a bit of German tinder or (which will do quite as well) a bit of one of the common cigar-lights made of touch-paper, and sold in strips. Loosen the stopper of the deflagrating jar with the left hand, while with the right hand you take up the cork with watch-spring attached. Light the tinder at the lamp, and immediately take out the stopper, and steadily lower the watch-spring into the gas, until the cork rests on the neck of the jar. The tinder will continue to burn, and the watch-spring, becoming red-hot, will also enter into brilliant combustion, sending out sparks and fusing into globules of iron oxide, which will fall off into the water in the tray. The beauty of the combustion is almost entirely due, not to the iron, but to the carbon present in the steel watch-spring; as may be shown by repeating the experiment, substituting for the spring a piece of pure iron wire ('binding wire') coiled into a spiral by being wound round a test-tube or glass rod about 1 cm. in diameter. In this case the metal will simply burn with a steady glow, emitting few or no sparks according to the purity of the iron.

(*d*) Place one of the bottles of gas on the table before you. Moisten with water a strip of blue litmus-paper, and another of reddened litmus-paper. Lift up the stopper of the bottle for a moment, and introduce the strips of litmus-paper into the gas. Observe that in neither case is the colour altered by contact with the gas; oxygen is therefore said to have a *neutral reaction* on vegetable colours[1].

Take the deflagrating spoon, and adjust its position by sliding the rod through the flange so that it may, when placed in the bottle, hang in the position shown in Fig. 10, about 6 or 8 cm. from the bottom of the bottle[2]. Place in it a piece of sulphur, rather larger than a pea. Loosen the stopper of the bottle of gas with the left hand, hold the deflagrating cup for a few moments in the flame of the lamp, until the sulphur melts and finally catches fire, and then immerse it in the bottle. The sulphur, which was burning in the air with a faint lambent blue flame, will immediately begin to burn much more brilliantly, while white clouds are formed in the bottle. When

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[1] If the evolution of gas has been rapid, chlorine and chlorine oxides may be present in sufficient quantity to be recognised by their odour (pure oxygen being inodorous), and to redden and bleach the litmus-paper in a short time.

[2] It may be adjusted with sufficient accuracy by holding it against the side of the bottle.

the combustion is over, take out the spoon, and observe the pungent, suffocating smell of the gaseous combination of sulphur and oxygen which has been produced; notice also that the colour of the blue litmus-paper in the bottle has been changed to red. A substance which thus reddens blue litmus-paper is said to have an *acid reaction.*

Reserve the remaining bottles of gas for use in future Exercises, placing a written label on each, to show what it contains.

EXERCISE 2.

Hydrogen.

Apparatus required—Pneumatic trough; three gas jars, 18 × 5 cm.; two ditto, 10 × 3 cm.; flask, with flat bottom, holding about 250 c.c.; bent delivery tube, used in the last Exercise; thistle funnel; corks; cork-borers; rat-tail file; retort-stand; piece of wire-gauze; taper on wire; glass disc, 8 cm. in diameter; gas tray, 8 cm. in diameter; Bunsen's burner; fish-tail burner; drying tube; glass tubing, 6 or 7 mm. in diameter; india-rubber tubing, for connectors; glass jet (p. 30); large beaker; glass tube about 30 cm. long and 2 cm. in diameter; jug of water; cloth; granulated zinc; strong hydrogen sulphate; distilled water.

Get ready the pneumatic trough as for the preceding experiment. Instead of bottles it is best to use plain cylindrical jars, ground at the mouth, about 5 cm. in diameter and 18 cm. high. Fill three of these with water as directed in the case of the deflagrating jars, and arrange them on the shelf of the trough. Now take the bent delivery tube, used in the preceding experiment; select a sound cork which will, after being squeezed, fit the neck of the flask; bore two holes in it (Fig. 21, p. 33), one large enough to admit the delivery tube, the other adapted to the tube funnel. If none of your cork-borers will make holes of the precise size required, use the next smaller cork-borer, and enlarge the holes with the rat-tail file. Fit the short arm of the delivery tube into the cork until its end just appears on the opposite side; then with a screwing motion pass the tube of the funnel through the other hole in the cork until the lower extremity, when the cork is fitted into the neck of the flask, would reach nearly to the bottom. Place in the flask about 30 grms. of granulated zinc, fit the cork into its place, and support the flask in the retort-stand on wire-gauze[1], so that the delivery tube may reach under the shelf of the trough, Fig. 34. Pour through the funnel enough distilled water to cover the zinc to the depth of 1 cm., and try whether the joints are tight by placing the warm hands on the flask for a few seconds. This will expand the air within, and

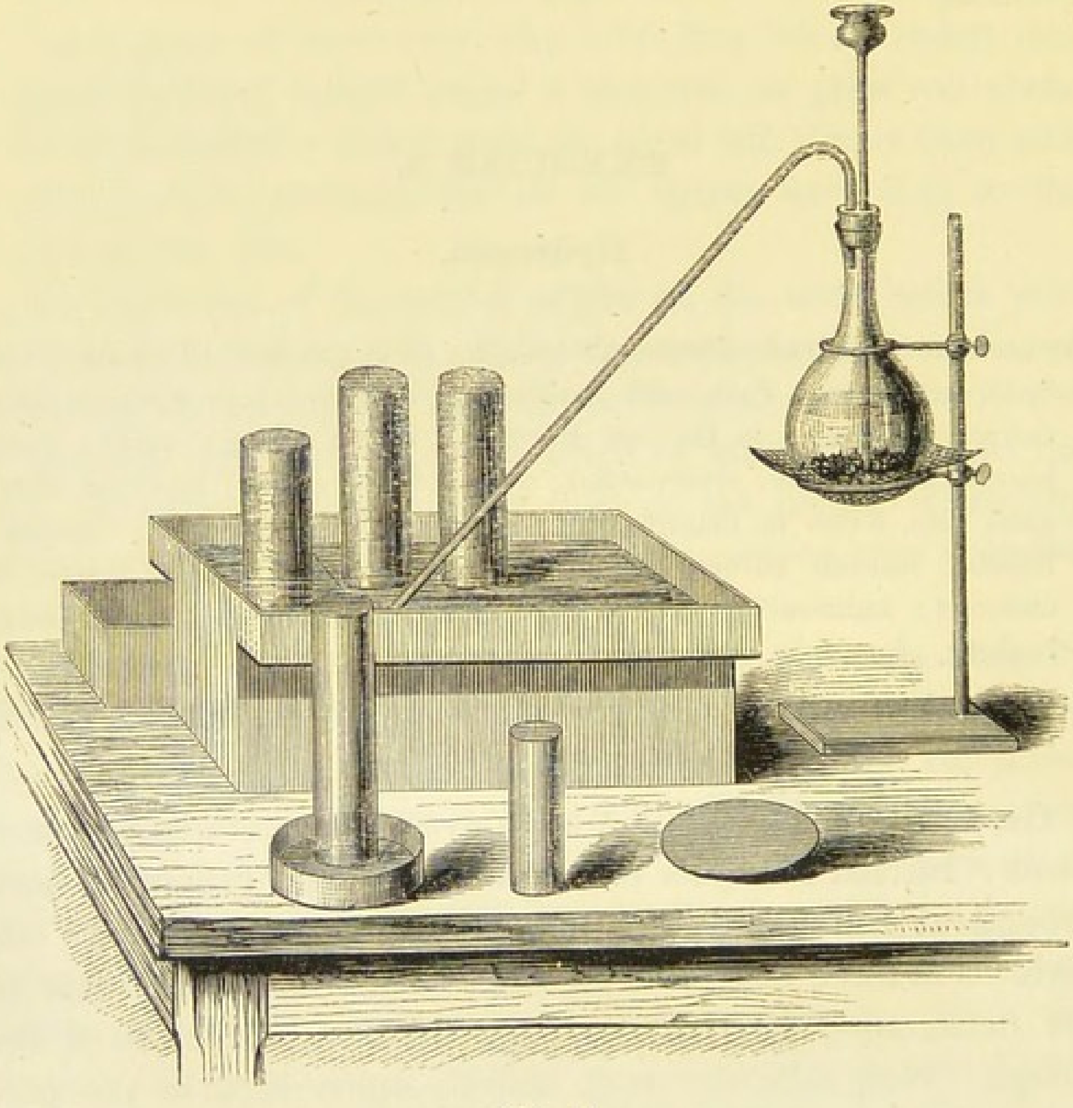

Fig. 34.

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[1] Although the flask does not, in the present case, require to be heated, yet it is advisable to place under it a piece of wire-gauze which serves as an elastic cushion.

raise a column of water in the funnel tube. If on withdrawing the hands this column remains steady for a moment, and only sinks gradually to its former level, the joints may be considered good. If however the column either does not rise at all, or sinks rapidly when the warmth is withdrawn, there is a leak somewhere in the cork. You may try to remedy this by pressing the cork further into the neck of the bottle: if this does not succeed, drop a little sealing-wax on the top of the cork and spread it evenly with a hot wire. This will almost certainly prove a cure; but do not proceed with the experiment until the joints will stand the above test.

Now pour a few drops of strong hydrogen sulphate down the tube funnel, and shake the flask, so that the acid and water may mix. When this reaches the zinc an effervescence will commence, owing to the liberation of hydrogen[1]. After a moment or two a little more acid may be poured through the funnel, so as to keep up a rapid stream of bubbles from the delivery tube; but especial care should be taken not to add too much at a time, lest the action should become too violent, partly from the undue strength of the acid, partly owing to the heat evolved from the mixture of the acid with the water. If the liquid in the bottle should show a tendency to froth over, pour some distilled water down the funnel, to dilute and cool the acid. It is often the case that the action is slow at first, owing to a film of oxide on the zinc which must be dissolved before the gas can be liberated; but it is better to give it a little time than to add acid recklessly. Fill three jars with the mixture of air and hydrogen which first escapes from the flask, and reject their contents before proceeding to collect the gas for your experiments. This precaution is of more importance here than in the case of oxygen, because (as will appear) air forms with hydrogen a mixture which explodes upon contact with a light, and you may have an awkward, if not dangerous, mishap, if you unintentionally experiment with such a mixture instead of pure hydrogen. After sacrificing

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[1] $Zn + H_2SO_4 = ZnSO_4 + H_2$.

this quantity, however, you may safely collect some jars of the gas, precisely as directed in Exercise 1, sliding the jars over the hole in the shelf, and when each is full removing it to the side shelf of the trough. It is best not to fill the jar completely with gas (as it may topple over), but only so far that the water in the tray and in the jar may stand at the same level. Add from time to time a little more strong hydrogen sulphate, to keep up the stream of gas, but manage so that the action may be subsiding while the last jar is being filled, so as to prevent unnecessary waste of gas. When the jars are full, leave the bottle and tube in their position for the present, and examine the following properties of the gas:—

1. Its inflammability.

Light the gas lamp, and place it and one of the jars of gas (still standing in its tray) on the table before you. Take a taper attached to a copper wire (Fig. 8, p. 7), and light it at the lamp: then with the other hand raise the jar of gas steadily from the water, keeping its mouth still downwards, and immediately pass the lighted taper up into it. The gas will take fire where it is in contact with the air, and burn with a pale almost invisible flame; but the taper on being pushed further into the gas will be extinguished. Now withdraw the taper and turn the mouth of the jar upwards; the flame will pass quickly down the jar, and the gas will be found to have disappeared entirely.

2. Its lightness as compared with common air.

(*a*) Take a small empty gas jar about 10 cm. high and 3 cm. in diameter: hold it inverted in the left hand; transfer another of the jars of gas from the trough to the table; raise it out of its tray with the right hand, keeping its mouth downwards, and bring its mouth close to the edge of the first jar; then steadily depress the closed end of the jar of gas, proceeding exactly as if you were pouring a fluid upwards from the one to the other vessel, Fig. 35. Set down the small jar, which is in your left hand (mouth downwards), in the tray of water; light the taper affixed to the wire and bring it into the upturned mouth of the other jar. The gas will be found to have escaped entirely, the taper burning as in the outer air. Now raise the mouth of the small jar above the level of the water in the tray, and immerse the lighted taper. A slight explosion will take place, as the gas catches fire, thus demonstrating that the hydrogen has really ascended and displaced the air in the jar, precisely as it displaces the water in filling a jar at the pneumatic trough.

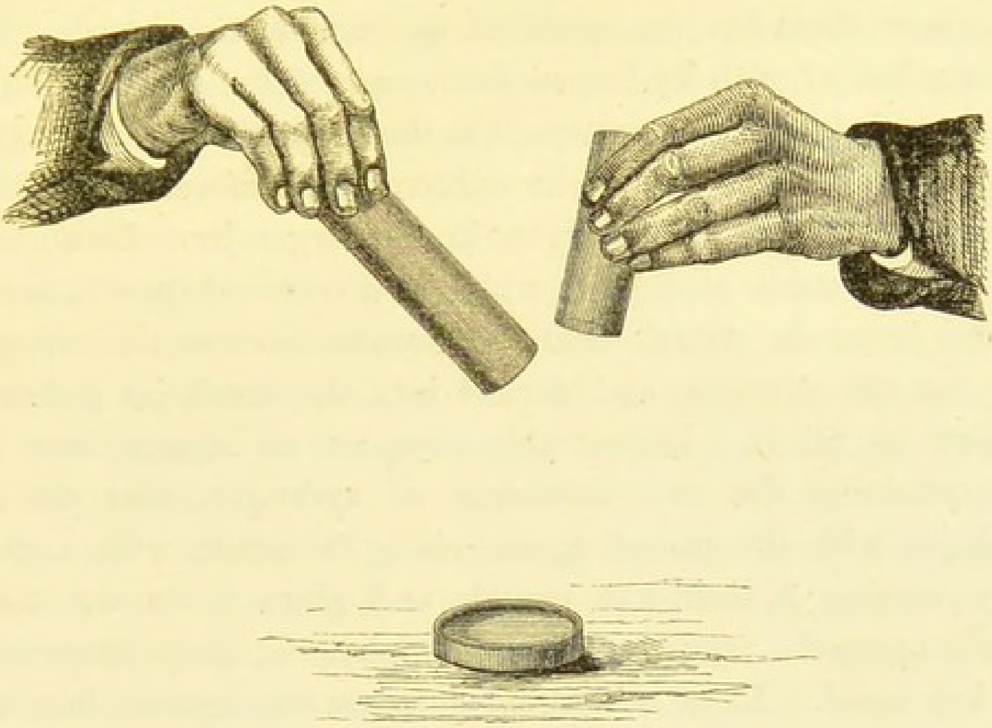

Fig. 35.

(*b*) Place the small jar used in the last experiment, mouth upwards on the table. Remove another jar of hydrogen from the pneumatic trough, closing its mouth with a glass plate. Bring the mouths of the two jars near each other, remove the glass plate from the jar of gas and gradually elevate its other end, proceeding as if you were pouring a liquid downwards into the small jar. Close the mouth of the latter with the glass plate, put the large jar aside, and plunge a lighted match into the small jar. The match will continue to burn as in the air, showing that there is no hydrogen in the jar. This proves that it is not possible to pour hydrogen downwards through air, and confirms the inference drawn from the preceding experiment, that hydrogen is lighter than air: since it is a law of hydrostatics, that the lighter of two fluids rises through the heavier.

3. Its combination with oxygen, to form water.

Fill with water both the jars used in the last experiment, and place them on the shelf of the pneumatic trough. Fill the smaller jar with hydrogen from one of the remaining jars of gas, and decant its contents into the larger jar. Fill it again with gas, and again decant its contents, as before. You have now two measures of hydrogen in the larger jar. Next, take one of the bottles of oxygen which you reserved (p. 67), invert it, and bring its mouth below the water-level in the trough; take out the stopper, and decant into the small jar sufficient oxygen to fill it. Decant this measure of oxygen into the jar containing the two measures of hydrogen, then fill the small jar with the mixed gases, close its mouth with a glass plate, remove it from the trough and place it on the table, mouth upwards, retaining the glass plate in its position with the left hand. Light a match, withdraw the glass plate and apply the lighted match to the mouth of the jar. The explosion which follows, though loud, is quite unattended with danger if a small strong jar is used as above directed. Do not, on any account, apply a light to the mixed gases in the larger and thinner jar, since you may have a serious accident if the jar should break.

You may, if time allows, try two or three similar experiments, varying the proportions of hydrogen and oxygen, and you will find that the explosion, which accompanies the chemical combination, is loudest when one measure of oxygen is mixed with two measures of hydrogen. This is found to be the exact proportion in which the two gases are combined in water[1].

You may, further, try a few similar experiments, using air instead of oxygen. You have noticed, in Experiment 1, that, when a lighted taper is passed into a jar of hydrogen, the gas takes fire at the mouth of the jar, where it is in contact with

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[1] $2H_2 + O_2 = 2H_2O$.

the air. This is due to its combination with the oxygen which is one of the constituents of air. Now, you will find that when air and hydrogen are mixed, and a lighted match applied to the mixture, an explosion takes place less violent than in the case of oxygen and hydrogen, but loudest when five measures of air are mixed with two measures of hydrogen. But we have just seen that *two* measures of hydrogen unite with *one* measure of oxygen to form water. Hence five measures of air contain one measure of oxygen[1]. That water is the product of the union of oxygen with hydrogen will be best shown by causing the combination to take place more slowly; for instance, by allowing a jet of hydrogen to burn in air. For this purpose we must attach to the generating apparatus another form of delivery tube.

Cut off a piece of glass tubing, of the same diameter as the delivery tube which you have been using, about 15 cm. in length; round off the sharp edges of each extremity with a file, or by fusing them in the lamp (p. 28), and bend the tube in the middle to a right angle (p. 26). Make another similar tube in the same way; then, having put out the lamp or removed it to a safe distance, take the bent delivery tube out of the cork of the generating flask and substitute for it one of the elbow tubes which you have just made. Connect the outer extremity of this tube, by means of a bit of india-rubber tubing about 3 cm. in length and 3 mm. in internal diameter, with a drying tube (Fig. 12, *c*, p. 10) which has been filled with fragments of calcium chloride (see p. 11). This drying tube should be supported in a horizontal position by the Bunsen's holder or by wooden blocks, and the second elbow tube should be fitted into the perforated cork at its other extremity. The whole apparatus will then be arranged as shown in Fig. 36.

To the upturned extremity of the elbow tube adapt, by a short bit of india-rubber tubing, a glass jet such as is described at p. 30, stretching the india-rubber connector so far

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[1] The composition of air will be further illustrated in the next Exercise.

over both tubes that their ends may be in contact, and that the tubes may lie in the same straight line.

Pour a little more acid on the zinc in the flask, and allow the gas to escape for half a minute or so. You may then safely light the hydrogen issuing from the jet. When it burns steadily with a flame about 3 cm. high, hold over it a clean dry beaker or bottle, and notice that a dew is at once deposited on the glass, soon collecting into drops of a colourless liquid, which is pure water. It will be hardly worth your while to collect any quantity of the water, but you may at any rate satisfy yourself that the product of the combustion is tasteless, and that when the outside of the glass is gently warmed by

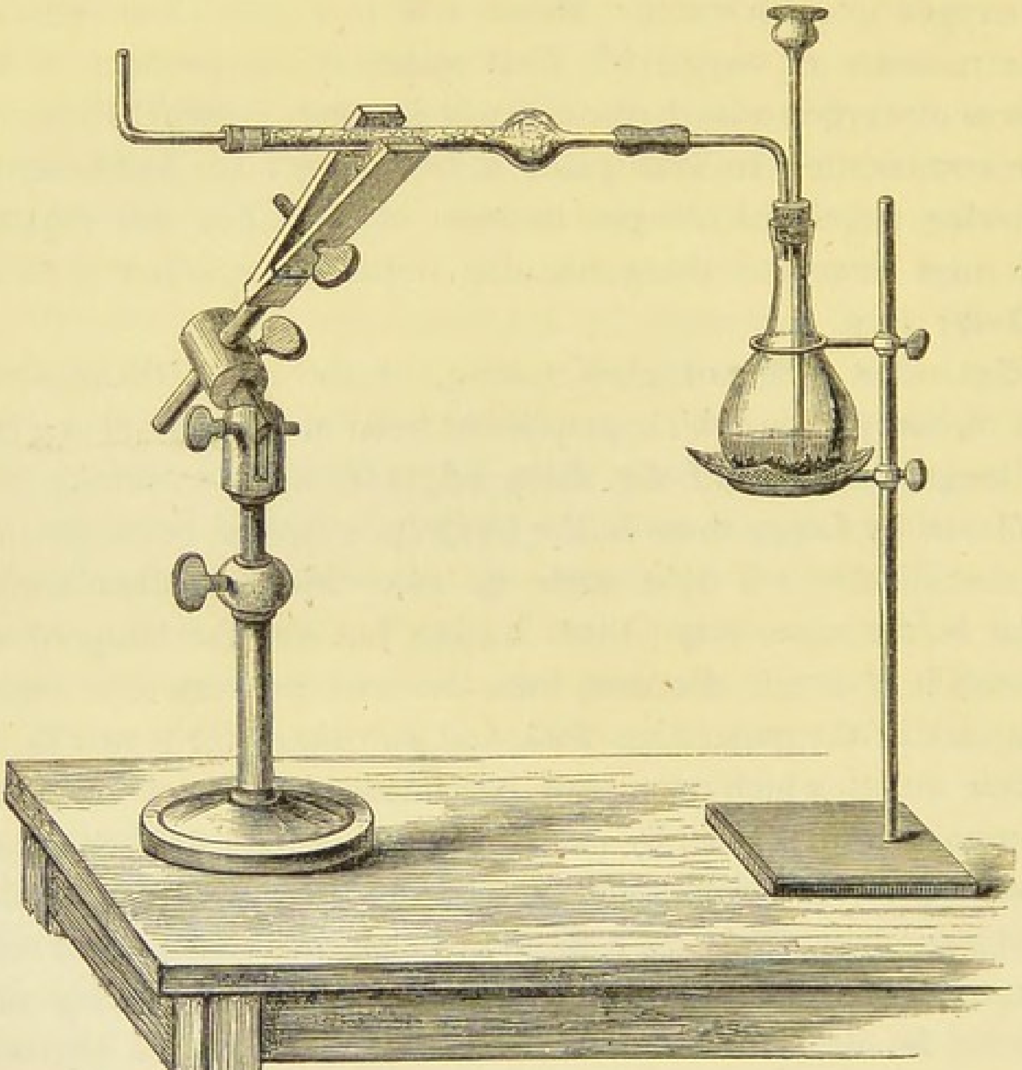

Fig. 36.

moving it to and fro over the lighted jet of gas the deposit volatilises without leaving any residue.

When the current of gas has slackened, but is still burning at the jet, another experiment may be tried which shows in an interesting way that the flame, under certain conditions, although apparently steady, is really expanding and contracting; is, in fact, rather a series of flames quickly succeeding one another, than a continuous flame.

Hold a glass tube, about 30 cm. in length and 1.5 or 2 cm. in diameter, over the jet, and depress it gently until the flame is entirely within the tube. At a certain point the flame will become elongated and a musical note will be produced varying in pitch with the length of the tube, and also, for the same tube, with the size of the flame. This sound is due to the fact that the air in the tube is set in vibration by the extremely rapid succession of explosions caused by the combination of the hydrogen as it issues from the jet with the oxygen of the surrounding air.

4. If you possess one of the small fish-skin balloons sold by opticians, the lightness of hydrogen gas may be further demonstrated in the following way:—

In the first place ascertain that there are no holes in the balloon by expanding it with air and holding it between your eye and the light, turning it round to examine every part of it[1]. If any holes are visible they may be mended by touching the margins with weak gum-water and covering them with small patches of gold-beaters' skin. A short piece of quill should be inserted in the neck of the balloon, and secured in its place by folds of thread. Take off the glass jet from the apparatus used in the preceding experiment, and, having carefully squeezed and sucked out *all* the air from the balloon, stretch the india-rubber connector over its neck, and proceed to fill it with gas, pouring a little more acid on the zinc whenever the current of gas becomes slow. When the balloon is full, you may allow it to ascend in the room, after inserting a small plug of cork

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[1] These balloons are very liable to be attacked by insects. They should be kept in a wide-mouthed bottle with a small bit of camphor.

into the neck, and attaching a long piece of thread to the quill, in order to have the movements of the balloon under control.

After trying the above experiments, you may take the apparatus to pieces, wash the tubes, and set them aside to dry. The liquid which is in the flask is a solution of zinc sulphate, and may be filtered into an evaporating dish, and evaporated as directed in Sect. **1**, Ex. 4, until it deposits crystals on cooling. The zinc sulphate thus obtained is very pure, and may be reserved for use in the laboratory.

EXERCISE 3.

Nitrogen.

Apparatus required—Pneumatic trough; porcelain dish, 5 cm. in diameter; ditto, 12 cm. in diameter; deflagrating jar; two gas jars, 18 × 5 cm.; glass disc, 8 cm. in diameter; taper on wire; large gas bottle; jug of water; cloth; blotting-paper; crucible tongs; knife; Bunsen's burner; phosphorus; tow; turpentine.

Float the small porcelain dish on the water in the pneumatic trough, retaining it over the movable shelf. Take a stick of phosphorus from the bottle with the crucible tongs, place it in a dish of water, and cut off with a knife (still holding it under water) a piece about as large as a pea[1]. Dry the piece of phosphorus thoroughly, by gently pressing, not rubbing, it between folds of blotting-paper; then place it at once, using the (dried) crucible tongs in the floating dish. Take the stopper out of the deflagrating jar, and, holding the jar in one hand over the phosphorus, light the latter by touching it with

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[1] In all experiments where phosphorus is made use of, great care must be taken to prevent it catching fire. It must never be allowed to remain for more than a few seconds out of water, and should always, if possible, be held with the forceps, not with the fingers. A jug of water should always be within reach to extinguish it in case it should accidentally inflame, as may happen in a hot room.

a piece of wire slightly heated in the lamp, and immediately lower the jar over the burning phosphorus until it rests upon the shelf of the trough, and insert the stopper, previously greased. The level of the water in the jar may at first be depressed, owing to the expansion of the enclosed air by the heat of combustion, but it will soon rise above the level of the water in the trough, showing that one of the constituents of the air is being withdrawn. When the phosphorus ceases to burn, allow the jar to remain undisturbed for half a minute, until the white fumes (which consist of a compound of phosphorus with oxygen, phosphorus pentoxide) have been for the most part absorbed, and meanwhile fill two gas jars with water and place them inverted on the shelf of the trough. Decant (p. 56) some of the contents of the deflagrating jar into the smaller jars, and examine the properties of the residual gas, as follows :—

1. Slide one of the jars of gas off the shelf of the trough, cover its mouth with a glass plate, lift the jar out of the water, retaining the plate in its position with one hand, and place it on the table mouth upwards. Light the piece of taper affixed to the wire, remove the glass plate from the mouth of the jar, and plunge the lighted taper into the gas. Observe that the taper is extinguished while the gas itself does not catch fire; thus showing it to differ from oxygen, in which the taper continued to burn, and from hydrogen, which extinguished the taper, but was itself inflamed.

2. Twist a piece of tow round the end of the copper wire in place of the taper, so as to form a ball about 1 cm. in diameter. Remove another jar of nitrogen from the pneumatic trough, and place it mouth upwards on the table, as in the last experiment. Pour a few drops of turpentine on the ball of tow, set it on fire, and immerse it in the jar of gas. The flame will be as instantly and completely extinguished as the small flame of the taper in the last experiment.

3. We have, in the above process for making nitrogen, divided atmospheric air into two parts : one, which supports the combustion of a taper, and which has combined with the phosphorus; and another, the residual gas, which has been

shown to be incapable of supporting the combustion of a taper, and which constitutes four-fifths by volume of the air. It will be interesting to try whether, by simply mixing nitrogen and oxygen in proper proportions, we can obtain a gas having the same properties as common air. We have ascertained by analysis that air consists of at least two substances; we may now proceed to obtain, by synthesis, an additional proof of the constitution of air.

Fill a large gas bottle with water, and place it, inverted, on the shelf of the trough. Fill one of the cylindrical gas jars with nitrogen from the deflagrating jar, and decant the gas into the bottle. Repeat this three times; you will then have four measures of nitrogen in the bottle. Now fill the cylindrical jar with oxygen from one of the bottles which you reserved in Exercise 1, and decant it into the gas bottle, which will now contain a mixture of nitrogen and oxygen in the proportion of four measures of the former to one of the latter gas. Insert the stopper of the bottle (under water), then place it mouth upwards on the table and immerse in it a lighted taper. The latter will not burn brilliantly as in oxygen (p. 65) or be extinguished as in nitrogen, but will continue to burn as it did in the external air[1].

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[1] The brilliancy may be slightly increased, since air is not *entirely* freed from oxygen by the action of phosphorus under the above conditions, and hence the mixture would contain rather more than the proper amount of oxygen.

EXERCISE 4.

Nitrogen Protoxide.

Apparatus required—Pneumatic trough, filled with warm water; stoppered retort, with long beak; retort-stand; piece of wire-gauze; argand burner; scales and weights; deflagrating spoon; three gas jars, 18 × 5 cm.; one ditto, 10 × 3 cm.; one gas bottle, holding 700 c.c.; one ditto, holding 300 c.c.; taper on wire; glass disc; cedar matches; sheet of writing-paper; corks; ammonium nitrate, in crystals; sulphur.

Weigh out 20 grms. of ammonium nitrate, and transfer it to the retort thus, Fig. 37. Support the latter on a piece of wire-gauze bent into the form of a basin, and placed on the largest ring of the retort-stand, in such a position that the beak may dip under the shelf of the pneumatic trough, and that there may be room for the argand burner under the wire-gauze. In order to keep the retort steady, the smallest ring may be brought down over the tubulure, a wedge or two of cork being inserted between the two. Instead of the stopper, a soft cork should be fitted into the tubulure; you will thus lessen the

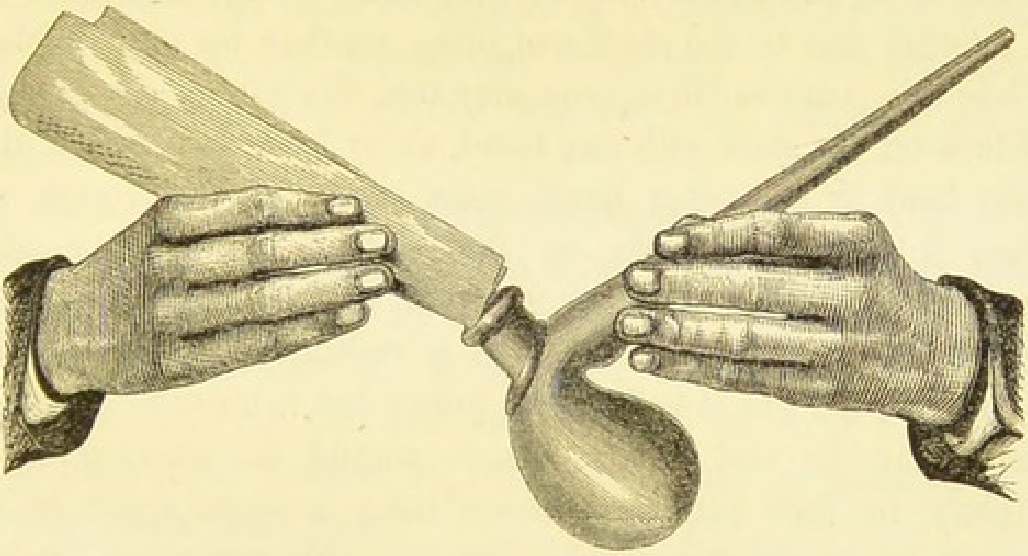

Fig. 37.

risk of the latter being cracked[1]. Heat the retort, slowly at first, by a lamp, and while it is gradually becoming hot, fill the gas jars and bottles with water, and place them mouths downwards on the shelf of the trough. The salt will soon fuse into a clear liquid, and by a slight increase of the heat will begin to decompose with effervescence owing to the escape of nitrogen protoxide[2]. Be careful not to use more heat than is necessary to keep the fluid steadily effervescing, and do not, on the contrary, withdraw the lamp entirely while the beak of the retort is under water; or the sudden condensation of the steam, which is one of the products of the decomposition, will be likely to draw up water from the trough into the hot retort, and crack it.

When a steady stream of bubbles has escaped for about a minute, you may place one of the smaller jars over the hole in the shelf, and allow it to fill with gas. When full, remove it to another part of the shelf and bring another jar into its place. While this latter is filling, you may test the gas in the first jar. Slide it off the shelf with one hand, cover its mouth with a glass plate held in the other hand, raise it out of the trough and place it mouth upwards on the table. Remove the glass plate, and immediately introduce a match which has been lighted and *just* blown out. If it is re-kindled, as in the case of oxygen, the gas is sufficiently pure; but it must be borne in mind that the end of the match should be glowing rather brightly, in fact only just blown out; a mere spark is not enough to begin the combustion, as with oxygen. Proceed with the filling of the other jars, carefully watching the decomposition of the salt. If the temperature is rather high, white fumes will be seen in the retort. So long as these

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[1] It is a decided advantage to use a retort instead of a flask and delivery tube, in the preparation of this gas. If the latter is used, the water which is formed in the reaction collects in the neck, and drops down on the melted salt, producing a burst of vapour. The retort can be more uniformly heated, and the water which condenses in the neck flows down into the trough: moreover the beak of the retort is wider than the usual delivery tube, and hence the condensation of the water is more regular.

[2] $(H_4N)NO_3 = 2H_2O + N_2O$.

appear only in moderate quantity, no harm need be apprehended, but if they increase so as to fill the retort, the heat should be diminished (not withdrawn) at once. Fill, besides the jars, one of the small gas bottles, and when it is completely full, insert the stopper (greased) under water, and reserve it for use in the next Exercise. Now lift the beak of the retort out of the water, and set the apparatus aside to cool. Meanwhile examine the properties of the gas, as follows:—

1. Cover the mouth of one of the jars with a glass plate, and place it mouth upwards on the table. Light the taper affixed to the end of a wire, and plunge it into the gas. The flame will become larger and more brilliant, but the gas will not itself ignite: thus proving it to belong to the same class as oxygen; viz. to be one of those gases which are not inflammable, but supporters of the combustion of a taper.

2. Burn a small piece of sulphur in a deflagrating jar of the gas, performing the experiment according to the directions given at p. 66. The sulphur will burn as brilliantly as in oxygen[1].

3. Remove another jar from the trough, pour into it 8 or 10 c.c. of cold water from a test-tube, replace the glass plate and shake the jar briskly for half a minute. Pour out the water into a glass and taste it. It will have acquired a sweet taste and smell, showing that the gas differs from oxygen both in being perceptibly soluble in cold water, and in possessing a peculiar odour and taste.

4. Re-fill with water the jar used in Experiment 3, and place it, inverted, on the shelf of the trough. Fill the small thick gas jar with nitrogen protoxide, and decant its contents into the larger jar. Fill the small jar with hydrogen (see note at the end of this Exercise), and decant the gas into the jar containing nitrogen protoxide. You have now a mixture of equal measures of nitrogen protoxide and hydrogen. Decant some of this mixture into the small jar, close its mouth with a glass plate,

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[1] The sulphur must be heated considerably above its melting-point, and must be in full combustion in the air; otherwise it is extinguished when plunged into the gas.

place it on the table (mouth upwards), and apply a light to the mixed gases[1]. The detonation which takes place is as violent as when the combination of oxygen with hydrogen was effected in a similar way[2].

5. The most remarkable property of this gas, its property of producing a peculiar intoxication when respired, is one which the student is advised not to try by himself; since particular precautions are necessary to obtain a perfectly pure gas for the purpose, and the effects of inhaling it vary with different individuals, and to some are decidedly injurious.

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[1] $N_2O + H_2 = H_2O + N_2$.

[2] A sufficient quantity of hydrogen for the above, and other similar experiments, may be made in the following way. Take a test-tube, about 1.5 to 2 cm. in diameter; place in it one or two bits of granulated zinc, and fill it completely with dilute hydrogen sulphate. When a brisk action has commenced, place an inverted porcelain dish, 9 cm. in diameter, over the test-tube like a cap; then, keeping the dish pressed close to the mouth of the tube, invert the apparatus quickly. The zinc will fall to the lowest part of the tube, and the evolved hydrogen will gradually fill the tube, displacing the acid which collects in the dish, and prevents the entrance of air. A minute bubble of air may possibly enter at the moment that the tube is inverted, if the operation is not skilfully performed, but so small a quantity will not materially vitiate the purity of the gas. When the tube is full of gas, fill up the dish with water, raise the tube a little so that the remainder of the zinc may fall into the dish; then close the mouth of the tube with the thumb, and transfer it to the pneumatic trough. A small gas jar may, of course, be substituted for the test-tube; its mouth being closed with a ground-glass disc, and the jar inverted quickly and placed in a basin containing a little water. The glass plate should be retained in contact with the mouth of the jar until the latter is full of gas, in order that the acid may not be diluted by mixing with the water in the dish.

This was probably the earliest method devised for the collection of a gas in order to examine its properties.

EXERCISE 5.

Nitrogen Dioxide.

Apparatus required—Pneumatic trough; flask, fitted with the delivery tube and funnel used in Ex. 2; glass measure; beaker; two 300 c.c. gas bottles; three gas jars, 18 × 5 cm.; two ditto, 10 × 3 cm.; taper on wire; glass disc; test-tubes and stand; blue litmus-paper; india-rubber rings; copper clippings; hydrogen nitrate; solution of iron proto-sulphate.

Place in the flask about 30 grms. of clippings of sheet copper, such as strips about half a centimetre broad and 6 or 8 cm. long. Support the flask, fitted with the delivery tube, in the retort-stand, arranging it so that the delivery tube dips under the shelf of the pneumatic trough, Fig. 34. Measure out 60 c.c. of water and place it in a beaker large enough to hold more than twice the quantity; then measure out the same volume of strong hydrogen nitrate and add it to the water in the beaker. While the mixture is cooling you may pour a little water into the flask, sufficient to cover the end of the funnel, and try the tightness of the cork as directed in p. 69. Fill the gas bottles and jars with water, and place them, inverted, on the shelf of the trough. Pour about 90 c.c. of the diluted acid upon the copper clippings. An effervescence will almost immediately commence, and the flask will be filled with red vapours[1]. These will, however, soon disappear, and a permanent gas will come over, with which, after a sufficient volume has been allowed to escape, the jars and bottle should be filled. No heat is required to accelerate the action: indeed, it may become too violent, and should then be moderated by surrounding the flask with a wet cloth or moistened blotting-paper. On the other hand, if the evolution of gas becomes slow before the receivers are all filled, more of the diluted acid

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[1] $3\,Cu + 8\,HNO_3 = 3\,Cu(NO_3)_2 + 4\,H_2O + 2\,NO$.

should be added through the funnel. When sufficient gas has been collected, the retort-stand and flask should be taken away at once, and placed in the open air.

The following experiments may now be tried with the gas you have collected:—

1. Insert the stopper into one of the bottles of gas, and place the latter mouth upwards on the table. Plunge into the gas the taper, lighted; it will be immediately extinguished, and the gas will not take fire.

2. Close the mouth of one of the jars with a glass plate, and place it mouth upwards on the table. Remove the glass plate for a moment, so as to allow air to enter the jar. Red vapours will be formed, owing to the union of the nitrogen dioxide with the oxygen of the air, to form higher nitrogen oxides[1]. Pour a little water into the jar and shake it up with the gas: the red fumes will be absorbed by the water, and a piece of blue litmus-paper dipped into the solution will be reddened, owing to the formation of hydrogen nitrite and nitrate[2].

3. Pour into a jar of the gas some solution of iron protosulphate: close its mouth with a glass plate, and agitate the liquid. A dark-brown solution will be formed, which, when poured into a test-tube and boiled over a spirit lamp, will give off the gas with effervescence, becoming nearly colourless again.

4. Fill one of the larger gas jars with water and invert it on the shelf of the trough. Decant into it the contents of one of the smaller jars of nitrogen dioxide, and then pass up into it, bubble by bubble, some oxygen gas from one of the reserved bottles (p. 67). Brownish-red fumes will be formed as each bubble rises into the gas: and the bulk of the mixed gases, which will at first be slightly increased owing to the heat produced by their union, will soon diminish; and finally, when

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[1] $N_2O_2 + O_2 = N_2O_4$. It should be observed that the formulæ N_2O_2 and N_2O_4 represent weights of the gases which occupy twice the space of the usual unit of volume, viz. the volume occupied by the weight of hydrogen expressed by the formula H_2.

[2] $N_2O_4 + H_2O = HNO_2 + HNO_3$.

sufficient oxygen has been added, both gases will entirely disappear, if the nitrogen dioxide is pure.

5. It was by the above method that Cavendish made his analyses of air, and the following experiment will serve to illustrate his mode of proceeding:—

Take a cylindrical jar, about 2 or 3 cm. in diameter and 20 cm. long (a large stout test-tube will answer perfectly). Select another test-tube, about 1 cm. in diameter and 7 cm. long, to serve as a unit of measurement. Fill this measure completely with water, pour it carefully into the larger tube, and mark the level at which the water stands by a small india-rubber ring (p. 56) slipped over the tube. Add another measure of water, and mark the level similarly with another ring. Proceed thus until five measures of water have been poured into the larger tube. You have now a tube graduated, with fair accuracy, into five parts of equal capacity, which should be filled to the fifth division with air. This is most conveniently done by filling the jar completely with air, and then allowing bubble after bubble to escape, until the water stands at the required level. Remember that in reading the volume of a gas, the jar should be lowered until the water inside the tube is on a level with the water in the trough. The enclosed gas will then be always measured under a nearly constant pressure, viz. that of the atmosphere[1]. Fill the small measuring tube with nitrogen dioxide, and decant the gas gradually into the air inclosed in the larger tube. Repeat this once more: you will then have added two volumes of nitrogen dioxide to five volumes of air. Now agitate the graduated tube (taking care that its mouth is not raised above the surface of the water in the trough), in order that the higher nitrogen oxides may be entirely absorbed; and, finally, depress the tube in the trough until the water inside and outside the tube stands at the same level, and note the volume of the residual gas. It will be approximately four-fifths of the original volume of air, and if no further red fumes are formed on addition of a little more nitrogen dioxide, we may

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[1] In Sect. 1, Ex. 8, we have seen that the volume of a gas varies with the pressure to which the gas is exposed.

assume that the oxygen of the air is entirely removed, and consequently that five volumes of air contain approximately one volume of oxygen. The residual gas may be tested with a lighted taper and shown to be a non-supporter of combustion.

6. Repeat Experiment 4, using the jar of nitrogen protoxide reserved in the last Exercise, instead of oxygen. No red fumes will be formed in this case; and this experiment furnishes a ready method of distinguishing between nitrogen protoxide and oxygen; gases which, as we have seen, greatly resemble each other in many of their properties.

7. Pour into a small test-tube 10 or 12 drops of carbon disulphide; cork the tube, and place it in the stand, within reach[1]. Place one of the larger jars of nitrogen dioxide on the table, previously decanting into it, if necessary, a few bubbles of gas, so as to fill it completely. Pour the carbon disulphide quickly into the jar (taking care to admit as little air as possible), shake it up once, to diffuse the vapour through the gas, replace the jar on the table, and apply a lighted match to its mouth. No explosion will follow, but a brilliant flash of light will pass down the jar[2].

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[1] Be particularly careful not to bring the carbon disulphide near a light, lest it should take fire. It is nearly as volatile as ether, and its vapour is highly combustible.

[2] Besides the process above given, the following is an excellent method of obtaining pure nitrogen dioxide. Measure 80 c.c. of water and place it in a beaker large enough to hold twice the quantity. Measure 30 c.c. of strong (common) hydrogen sulphate, and add it, a few drops at a time, to the water in the beaker, stirring the mixture with a glass rod. Much heat is evolved by the union of the acid with the water (compare the action of water on quicklime, p. 45), and, unless the operation is carefully performed, the beaker may be broken. When all the acid has been added, place the beaker aside to cool. Meanwhile, mix in a mortar 6 grms. of potassium nitrate and 30 grms. of iron protosulphate. Arrange an apparatus as above directed, and place the mixture in the flask. Pour on it the diluted acid through the tube funnel, and apply a gentle heat to the flask. The mixture will become brown, and pure nitrogen dioxide will be evolved.

$$6\,FeSO_4 + 4\,H_2SO_4 + 2\,KNO_3 = 3\,Fe_2(SO_4)_3 + 4\,H_2O + K_2SO_4 + 2\,N_2O_2.$$

COLLECTION OF GASES BY DISPLACEMENT.

We have hitherto only had to deal with gases which are comparatively insoluble in water, and which therefore may be collected without much loss by means of the ordinary water pneumatic trough. But many of the gases which we have yet to prepare are so freely dissolved by water that it would be impracticable to collect them over that fluid. In such cases we must either fill our trough with some fluid in which the gas is insoluble, such as mercury, or have recourse to an entirely distinct method, which depends on the difference between the density of the gas and that of common air. When we pour water into a vessel, the water, being heavier than the air contained in the vessel, descends to the bottom and displaces the air, which is driven out at the top. Precisely the same thing occurs when we lead a gas heavier than air into an open jar. The gas does not mix with the air at once, but collects at the bottom of the jar and gradually rises, driving the air before it, and finally overflowing the edge of the jar like other fluids. It is obvious that a gas lighter than air may be similarly collected by simply reversing the position of the receiving vessel, placing it mouth downwards, and leading the gas into its upper part.

It is, however, necessary, in order to obtain by this method a gas even approximately pure, to pass into the jar at least twice the volume of gas required to fill it; since diffusion must take place between the fluids with greater rapidity as the difference in their densities is less, and hence the boundary-line between the gas and air is never so sharply marked as in the case of water and air. The method, however, of collection by displacement is so convenient and simple that it will be used in the following Exercises in preference to the employment of a mercury trough, especially as the great expense of mercury would place it beyond the reach of most of those for whom these Exercises are intended.

It should be remembered that, when a gas is to be collected by displacement, all currents of air in the room must be avoided as much as possible: the tendency of such currents being to stir up the gas, and produce a more rapid mixture with air than diffusion alone would cause.

EXERCISE 6.

Carbon Dioxide.

Apparatus required—Flask, fitted with cork and tube funnel as in Ex. 2; elbow tube, with branches 9 cm. in length; ditto, with branches respectively 9 cm. and 20 cm. in length; corks; cork-borers; rat-tail file; retort-stand; wire-gauze; wooden blocks; card; two gas bottles, holding 700 c.c.; four ditto, holding 300 c.c.; gas jar, 18 × 5 cm.; glass disc; test-tubes; porcelain dish; Bunsen's burner; taper on wire; deflagrating cup; marble; strong hydrogen chloride; distilled water; litmus-paper; magnesium ribbon.

Take the flask which was used in Ex. 2, and substitute for the bent delivery tube another of the form shown in Fig. 38. It is made up of two elbow tubes united by a cork joint: one of the elbow tubes having two equal branches about 8 or 9 cm. in length, the other having one branch about 8 cm., the other about 20 cm. in length. The cork joint is made by boring a central hole through a small cork (as free from fissures as possible), and pushing one branch of each tube into the hole until they meet in the middle of the cork. This forms an excellent joint, and has one special advantage over an india-rubber connector, viz. that it is stiff, and yet allows the tube to be turned round in any direction, retaining it in whatever position it is placed[1].

Place in the flask 30 grms. of marble, previously broken with a hammer into lumps rather larger than peas. Secure the

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[1] A similar but neater joint may be made by cutting off a piece of glass tubing about 3 cm. in length and 8 or 9 mm. in diameter internally, and (after fusing the sharp edges in the lamp) fitting into it the two elbow tubes by means of rings of india-rubber tubing.

flask in the retort-stand at such a height that the open extremity of the delivery tube may be raised about 14 cm. above the table. Pass the end of the delivery tube into one of the bottles, supporting the latter on blocks, so that the tube may reach almost to the bottom. Over the mouth of the bottle lay a card in which a slit has been cut, half way across it, wide enough to admit the delivery tube. Pour through the funnel sufficient water to cover the marble, and then add, a few drops at a time, some strong hydrogen chloride, until a brisk effervescence is set up, due to the evolution of carbon dioxide[1]. After the lapse of half a minute, light the taper, remove the card from the mouth of the bottle, and pass the taper steadily down into it. It will probably be extinguished before it reaches

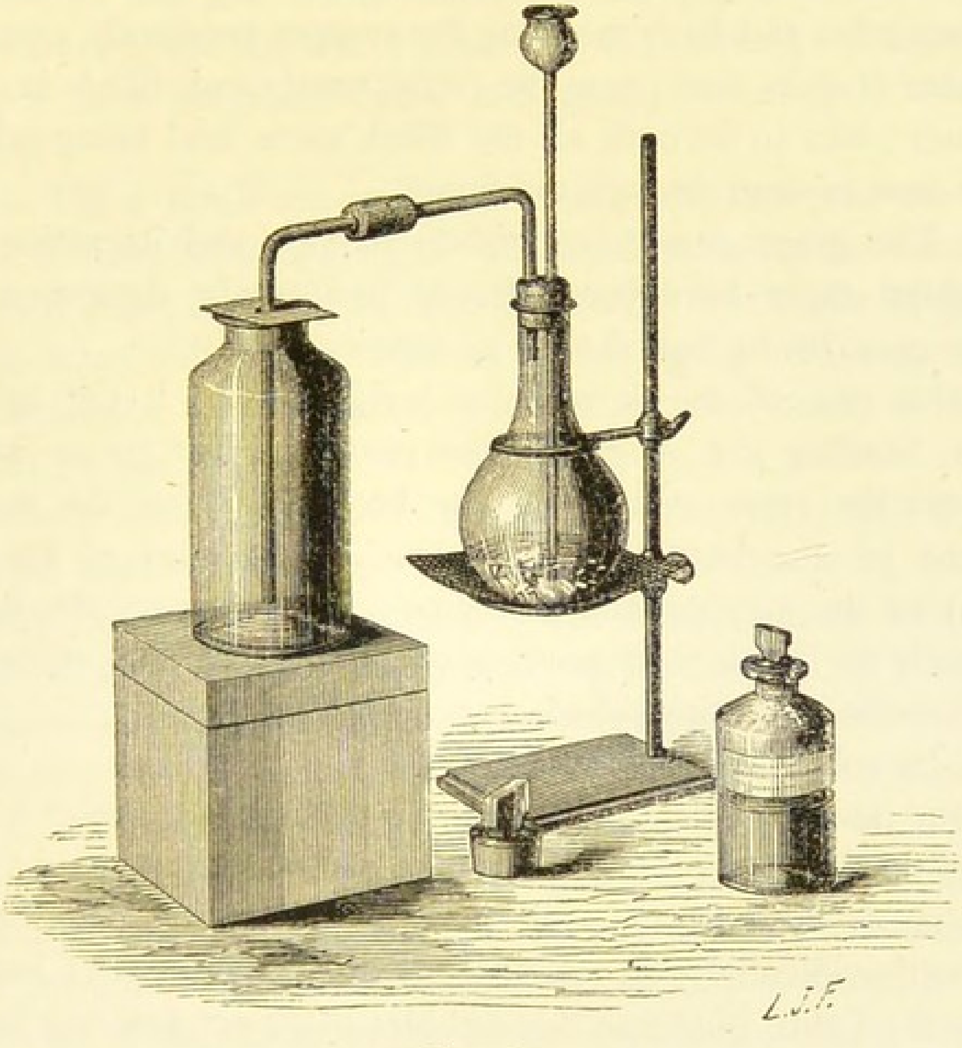

Fig. 38.

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[1] $CaCO_3 + 2HCl = CaCl_2 + H_2O + CO_2$.

the bottom, showing that the gas has partially filled the bottle. It may be withdrawn, re-lighted, and after a few seconds plunged again into the bottle, to ascertain the point to which the gas has now risen. When the taper is quickly and decidedly extinguished when held at, but not within, the mouth of the bottle, the latter may be considered to be full of gas. It should then be withdrawn, by first taking away the wooden blocks, then lowering the bottle slowly until clear of the delivery tube, and lastly inserting the stopper previously greased. Another bottle may now be substituted and filled in like manner; and so on until all are filled, more acid being added from time to time through the funnel.

1. The great density of carbon dioxide and its action on a lighted taper have been already incidentally demonstrated. They may be further shown as follows:—

Take one of the larger gas jars, place in it the lighted taper, bending the wire over the mouth of the jar so as to support the taper steadily at the bottom. Bring the mouth of one of the bottles of gas close to the edge of the jar, withdraw the stopper, and gently pour the gas over the taper, precisely as if you were pouring out a liquid[1]. The taper will be immediately extinguished.

2. Its solubility in water.

Pour into a bottle of the gas sufficient distilled water to fill it one-third, and replace the stopper, inserting a small strip of thin paper between it and the neck of the bottle. Shake the bottle violently for a few seconds, then invert it and hold it up to the light. Observe that bubbles of air are entering at the side of the stopper, thus showing that there has been an absorption of the gas. If the piece of paper had not been inserted at the side of the stopper, the latter would have become so firmly fixed in its place by the pressure of the external air, that it would have been difficult to loosen it. Shake the bottle again for a few seconds, then pour out some of the

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[1] Do not pour the gas directly over the centre of the jar, but at its edge, since the gas receives a forward as well as a downward impetus while the bottle is being inverted.

solution into a beaker and taste it. The water will have acquired a distinct acid taste. Pour some more of the solution into a test-tube, and heat it over a lamp. Bubbles of gas will be given off as the temperature rises, and if the liquid is boiled for a minute or two, the whole of the gas will escape[1].

3. Its action on vegetable colours.

Introduce a piece of moistened blue litmus-paper into a bottle of the gas. The colour of the paper will change to a wine-red, but its blue colour will be restored on laying the paper in a porcelain basin and gently warming it over the lamp.

4. Fill a small test-tube with lime water, and pour the liquid into another bottle of the gas. Close the mouth of the bottle with a glass plate, and shake it slightly at first, afterwards vigorously. The liquid will at first become milky, owing to the formation of insoluble calcium carbonate, but will afterwards, if the gas is in excess, become clear again, because a soluble calcium and hydrogen carbonate is formed. If the liquid still remains turbid after agitation, pour it into another bottle of gas, and agitate again. If, now, some of the clear solution be poured into a test-tube and heated to boiling over the lamp, the calcium and hydrogen salt will be decomposed; carbon dioxide will be given off, and the insoluble carbonate re-precipitated.

5. Affix a piece of magnesium ribbon, about 16 cm. in length, to the flange of the deflagrating cup, or to a cork, in the manner described in p. 65, so that its extremity may reach nearly to the bottom of one of the large bottles of carbon dioxide. Hold the end of the ribbon in the lamp-flame until it begins to burn with a brilliant white light, then plunge it into the bottle of gas. It will continue to burn brilliantly, forming white flakes of magnesium oxide interspersed with black particles which consist of carbon. In order to separate the latter, pour into the bottle, when the combustion is over, a little distilled water, shake it up and pour it, with the suspended particles, into an evaporating dish. Add 8 or 10 drops of strong hydrogen

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[1] Carbon dioxide may, in fact, be collected over warm water, like nitrogen protoxide; but the displacement method is preferable.

chloride, and heat the liquid over a lamp. The magnesium oxide will disappear, while the black flakes of carbon will remain floating undissolved in the clear liquid. This experiment proves that the gas which you are examining contains carbon, and also that some substances will burn in a gas in which a taper will not burn. Combustion is purely a relative term, and may be defined as the chemical action of one substance upon another accompanied with the evolution of light and heat.

EXERCISE 7.

Carbon Protoxide.

Apparatus required—Pneumatic trough; flask, with elbow tubes and tube funnel, used in the preceding Exercise; bottle holding about 200 c.c.; corks; cork-borers; rat-tail file; bent delivery tube used in Ex. 1; wooden blocks; retort-stand; wire-gauze; argand burner; india-rubber connector; three jars, 18 × 5 cm.; one ditto, 10 × 3 cm.; glass disc; crystallised hydrogen oxalate; strong hydrogen sulphate; solution of potassium hydrate (caustic potash); solution of calcium hydrate (lime water); potassium ferrocyanide; small bottle of oxygen.

The usual method of preparing this gas is to heat hydrogen oxalate with an excess of strong hydrogen sulphate. A mixture of carbon dioxide and protoxide is given off[1], and by absorbing the former gas we obtain pure carbon protoxide. The carbon dioxide is withdrawn by allowing the mixed gases to bubble through a solution of an alkali or through lime water, which has no action on carbon protoxide, but, as we have seen, unites with carbon dioxide to form a metallic carbonate. We cannot, therefore, as hitherto, allow the gas to pass directly from the generating to the receiving vessel, but we must interpose between the former and the latter a bottle containing the solution of alkali or lime water, a solution of caustic potash being preferable, as lime is comparatively insoluble in water.

[1] $H_2C_2O_4 = H_2O + CO_2 + CO$.

Adapt a cork to a bottle with a rather wide mouth, about 200 c.c. capacity. Bore two holes in the cork: into the one fit the bent delivery tube used in the preparation of hydrogen; into the other fit the longer branch of the elbow tube used in the last Exercise, passing the tube through the cork to such a distance that its extremity may reach almost to the bottom of the bottle. Before the cork is finally fitted into its place, fill the bottle to about three-fourths of its height with a solution of caustic potash or soda[1]. Support it on blocks at such a height that the delivery tube may dip under the shelf of the pneumatic trough. In the next place, take the flask, fitted with the short elbow tube and funnel, used in the preceding Exercise, place in it about 20 grms. of crystallised hydrogen oxalate, and fix it in the retort-stand, with a piece of wire-gauze under its bottom, at such a height as to admit of the argand burner being placed under it, and to allow the elbow tube to be connected with the tube of the wash-bottle by a short piece of india-rubber tubing.

The whole apparatus will then appear thus, Fig. 39.

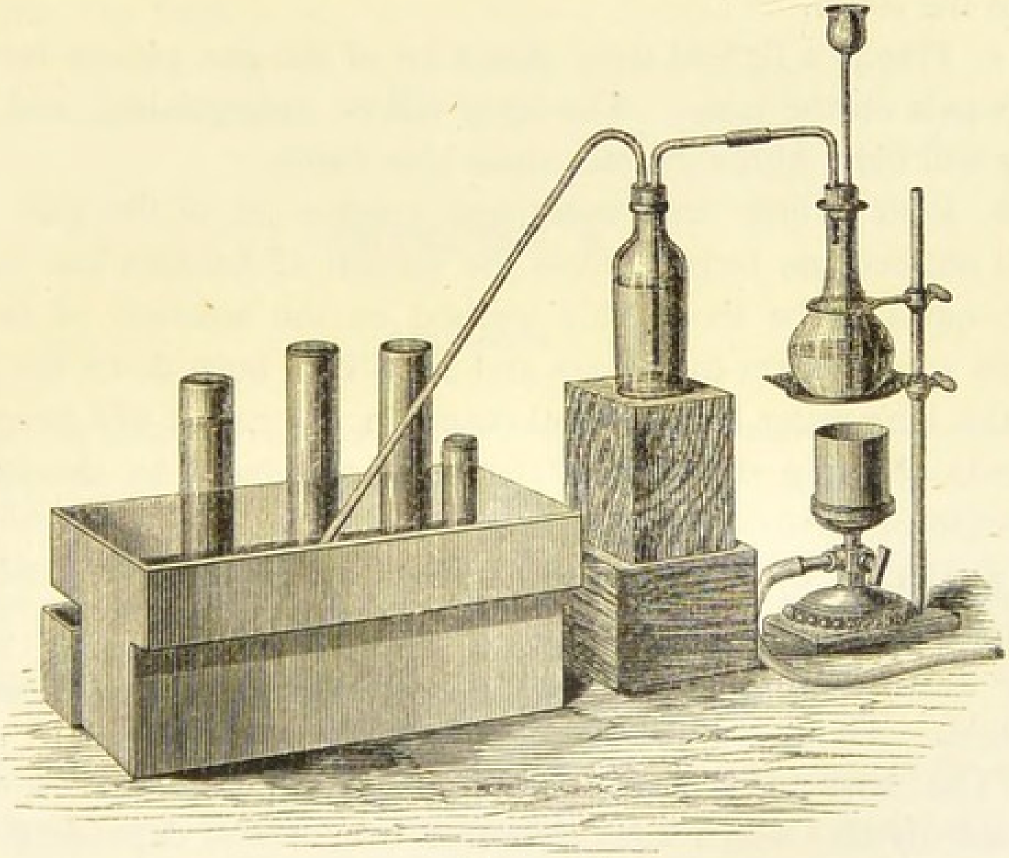

Fig. 39.

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[1] For directions for preparing this, see Sect. 3.

Pour into the flask about 60 c.c. of strong hydrogen sulphate. As there are several chances of leakage, it will be necessary to test the joints as directed in p. 69, and not to proceed with the experiment until they are all made tight. The flask may then be gradually heated with the lamp, especial care being taken to avoid any chance of cracking it, and thus spilling the strong acid to the damage of table, clothes, and hands. While the flask is being heated, the four jars may be filled with water and inverted on the shelf of the trough.

When the mixture in the flask begins to effervesce, the heat should be moderated, so that the stream of gas may pass through the solution in the wash-bottle in bubbles not succeeding each other too quickly to be counted.

After a sufficient quantity of the gas has escaped, the jars may be filled. As soon as this is done, the india-rubber connector between the flask and the wash-bottle should be taken off, and the flask removed at once into the open air, as the gas is poisonous and should not unnecessarily be allowed to escape into the room.

1. Plunge a lighted taper into a jar of the gas, placed mouth upwards on the table. The taper will be extinguished, and the gas will burn with a characteristic blue flame.

2. Pour a little lime water into another jar of the gas. It will not become turbid, unless the stream of bubbles has been too quick to be thoroughly washed by the solution of soda. Now apply a light to the gas and allow it to burn down the jar. If the lime water be now shaken up in the gas, it will become turbid, showing that carbon dioxide is produced by the combustion of carbon protoxide in air.

3. Decant into one of the gas jars two measures of carbon protoxide (the measure being the small gas jar, p. 72). Add one measure of oxygen, transfer the jar to the table, and apply a light. The gases will unite with a slight explosion.

[The best method of procuring carbon protoxide is undoubtedly that which was discovered by Fownes, depending on the decomposition of potassium ferrocyanide when heated with hydrogen sulphate.

The apparatus may be the same as that described above. The wash-bottle, although not absolutely necessary, is useful to absorb traces of hydrogen carbonate, cyanide, and sulphite, which come over.

Place in the flask about 60 c.c. of strong hydrogen sulphate. Weigh out 15 grms. of potassium ferrocyanide, reduce the salt to powder, and add it little by little to the acid in the flask, shaking between each addition, in order to prevent the salt caking into lumps. Now fit in the cork and proceed to heat the mixture, watching carefully for the first evolution of gas[1]. No gas comes off until the temperature is pretty high, and then there is a sudden rush, which must be moderated by withdrawing the lamp at once. The temperature should not be allowed to rise higher than is necessary to obtain a slow stream of the gas. It is better not to pour water into the flask for the purpose of washing out the residue, until as much of the latter as possible has been shaken out; otherwise the sudden heat produced by the addition of water to the strong acid is likely to crack the flask.]

EXERCISE 8.

Ammonia.

Apparatus required—Florence flask; corks; cork-borers; elbow tubes used in the last Exercise; drying tube, Fig. 12; india-rubber connector; sand-bath; argand burner; retort-stand; Bunsen's holder; wooden blocks; card; taper on wire, Fig. 8; glass disc; pan of water; one large and three small gas bottles; mortar; scales and weights; porcelain dish; plates; sheets of paper; ammonium chloride (sal-ammoniac); quicklime; turmeric and litmus-paper; distilled water.

Weigh out 30 grms. of sal-ammoniac, and reduce it to powder in the mortar. The salt in its sublimed state is so

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[1] $K_4Fe(CN)_6 + 6\,H_2SO_4 + 6\,H_2O = 3\,(H_4N)_2SO_4 + 2\,K_2SO_4 + FeSO_4 + 6\,CO$.

tough as to render this rather difficult, and more may be effected by direct blows of the pestle, especially if aided by a thrust and slight twist of the arm at the moment of impact, than by the usual rubbing motion. Place the powdered salt in a basin and set it to dry on the sand-bath over the lamp. Place about an equal weight of quicklime on a plate, pour over it just sufficient water to slake it, and leave it to cool, covering the plate with another to protect the lime from the air[1].

Adapt a cork to a clean dry Florence flask, and fit into the cork an elbow tube, each branch of which is about 8 cm. long. Fill the drying tube used in Ex. 2, with fragments of quicklime about as large as split peas, in the manner described at p. 11. Into the perforated cork at the other end of the drying tube fit another elbow tube, the branches of which are respectively 9 cm. and 20 cm. long. The sal-ammoniac should now be taken from the sand-bath and set aside to cool. Meanwhile the bottles in which the gas is to be collected may be dried and their stoppers greased. When the sal-ammoniac and lime are both cool, but not before, mix them quickly by rubbing in a mortar, and transfer the mixture at once to the flask, using a paper gutter for the purpose, p. 41.

Replace the cork with the short elbow tube in the neck of the flask, and support the latter in the Bunsen's holder at such a height that the argand burner or spirit lamp will pass easily under it. Connect the drying tube with the flask by an india-rubber connector, so that, the drying tube being horizontal, the long branch of the elbow tube may point vertically upwards. Support this latter at the bend by the smallest ring of the retort-stand[2]; and bring over it the second retort ring, clamping it at such a height that when one of the bottles is inverted over the tube, its neck may rest on the retort-ring when the extremity of the vertical tube nearly reaches to its bottom, Fig. 40.

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[1] The chief reason for slaking the lime is, that most specimens of quicklime are difficult to powder in a small mortar.

[2] It will be advisable to fit into the ring a cork having a notch cut across it in which the tube may lie. This is not represented in the engraving.

The flask may now be gently heated by the lamp. If the spirit lamp must be used, it should be moved to and fro under the flask, the flame being never allowed to rest in one place, otherwise the flask is apt to crack; an accident which is not unlikely to happen in any case, as the substance to be heated is a solid of low conducting power, and not a liquid, which would distribute the heat by convection.

The gas comes off at a comparatively low temperature, and from its low density collects in the highest part of the bottle, driving the air downwards before it[1]. It will be well to cover the mouth of the bottle with a card similar to that used in Exercise 6, in preparing carbon dioxide, retained in its place by turning up its opposite edges and slipping it over the border of the neck of the bottle. To ascertain when the bottle

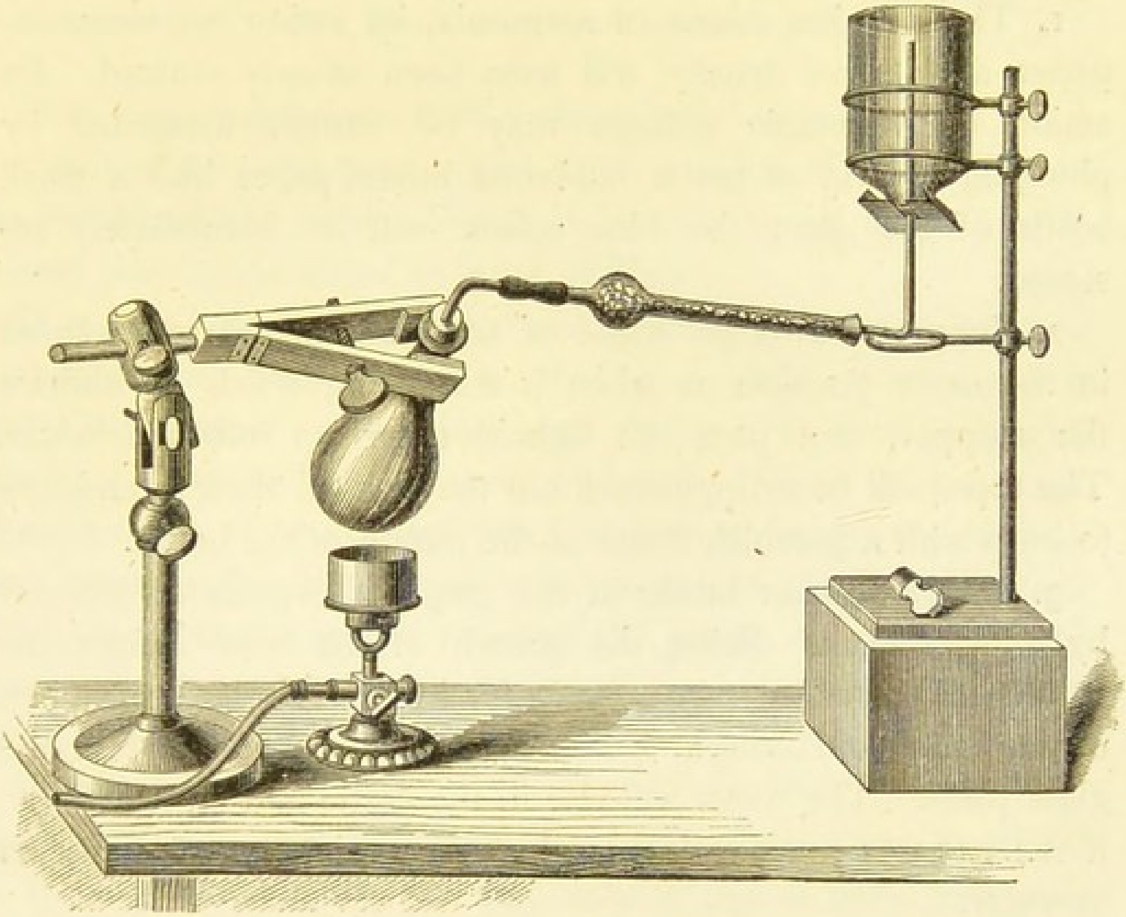

Fig. 40.

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[1] $Ca H_2 O_2 + 2 (H_4 N) Cl = Ca Cl_2 + 2 H_2 O + 2 H_3 N$.

is full of gas, hold a piece of moist turmeric-paper close to and slightly above its mouth. If the gas is overflowing, it will quickly and decidedly redden the test-paper. The bottle should then be slowly raised until clear of the delivery tube, when the card should be withdrawn, and the stopper inserted. Another bottle may then be placed in the same position and filled in like manner. When all the bottles have been filled with the gas, withdraw the lamp, take the cork out of the flask, and remove the latter into the open air.

1. The peculiar odour of ammonia, its action on turmeric-paper, and its low density, will have been already noticed. Its action on vegetable colours may be further illustrated by plunging a strip of moist reddened litmus-paper into a small bottle of the gas; the blue colour will be immediately restored.

2. Support the large bottle of ammonia in the retort-stand in the same position as when it was being filled. Withdraw the stopper, and pass the lighted taper up into the bottle. The taper will be extinguished, but the gas will show a tendency to burn with a greenish flame at the mouth of the bottle.

3. Invert another bottle of the gas, and replace its stopper by a glass plate. Bring the mouth of the bottle under the surface of some water in a basin (or in the supplementary pan of the pneumatic trough, *b*, Fig. 1), and gradually withdraw the glass plate. The water will rise in the bottle and fill it entirely, if the gas was pure, owing to the great solubility of ammonia in water.

4. The last bottle of the gas should be reserved for use in the next Exercise.

EXERCISE 9.

Hydrogen Chloride.

Apparatus required—Iron ladle; mortar; apparatus similar to Fig. 38; argand burner; three small gas bottles; taper on wire; two glass discs; porcelain dish; sand-bath; test-tube; sodium chloride; hydrogen sulphate; distilled water; blue litmus-paper; turmeric-paper.

Fuse some common salt in a clean ladle or iron spoon over a common fire, and pour it out upon a piece of iron (such as the sand-bath) to cool. When it is cold break it up in the mortar into lumps about as large as peas.

Fit up an apparatus similar to that which was used for preparing carbon dioxide, p. 89, placing a piece of wire-gauze under the bottom of the flask, and fixing it at such a height that the lamp will pass easily under it. Place in the flask about a dozen lumps of the fused sodium chloride, and having set a bottle under the delivery tube, pour into the flask enough strong hydrogen sulphate to cover the lumps of salt[1]. Apply a very gentle heat if the gas does not come over with sufficient rapidity. Fill four bottles with the gas by displacement, using a lighted taper to ascertain when they are full, precisely as directed in the case of carbon dioxide. Take away the flask as soon as the bottles are full, as the gas is very irritating to the lungs.

1. The incombustibility of the gas, its inability to support the combustion of a taper, its high density, and the fumes which it produces in moist air, will have been already noticed.

2. Plunge a piece of blue litmus-paper into a bottle of the gas. It will be at once strongly reddened.

3. Repeat Experiment 3 of the last Exercise with another bottle of hydrogen chloride. It will be seen to be as soluble in water as ammonia.

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[1] $H_2SO_4 + NaCl = NaHSO_4 + HCl$.

4. Remove the stopper from a third bottle and replace it by a glass plate, placing the bottle mouth upwards on the table. Bring over it the bottle of ammonia which was reserved in the last Exercise, its mouth being downwards and covered by a glass plate. Place the mouths of the bottles close together, and remove both plates, thus allowing the gases to mix. Dense white clouds will be produced, and still more if the position of the bottles is reversed, their mouths being still held together, so that the bottle of hydrogen chloride is uppermost, causing the heavier gas to descend into the lighter. Enough heat is evolved by the union of the gases to make the bottles sensibly warm; and a snow-white powder is deposited in flakes on the sides of the bottles [1]. When the action is over examine the properties of this powder as follows. Pour 20 or 30 c.c. of distilled water into one of the bottles, shake it up until the white powder is dissolved, then pour the liquid into the other bottle and dissolve the deposit in it also. Place the liquid in a porcelain dish and evaporate it to dryness, using a gentle heat. Scrape together some of the white residue with a spatula, place it in a test-tube, and heat it over the lamp. It will volatilise entirely, forming a white ring in the cooler part of the tube. This distillation of a solid substance is called 'sublimation.' Observe that the substance does not fuse before volatilising, but passes at once from the solid to the gaseous state. This is an exception to the general rule that solids become liquids before they volatilise.

Dissolve the remainder of the residue in a little water and test the solution with blue litmus and turmeric-paper. No alteration of colour will be produced in either case, while we have seen that hydrogen chloride reddened litmus-paper, and that ammonia reddened turmeric-paper. By the combination of the two gases a neutral substance, or salt, has been formed.

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[1] $H_3N + HCl = (H_4N)Cl$.

EXERCISE 10.

Chlorine.

Apparatus required—Flask, tubes, and wash-bottle, used in Ex. 7; elbow tubes and cork-joint used in Ex. 9; argand burner; wooden blocks; card; two large and four small gas bottles; taper on wire; deflagrating spoon; crucible tongs; porcelain dish; one large and one small cylindrical gas jar; glass disc; supplementary pan of the pneumatic trough, filled with warm water; manganese dioxide; strong (common) hydrogen chloride; blue litmus-paper; solution of cochineal; phosphorus; brass filings, or metallic antimony; distilled water.

We have in this case to deal with a gas which is soluble to a considerable extent in water, though much less so than ammonia or hydrogen chloride. Its solubility rapidly diminishes as the temperature of the water is raised, and hence it may be collected without much loss over warm water, the ordinary pneumatic trough being employed. But its density is so high that it can be most readily collected by downward displacement, like carbon dioxide.

Its action on the lungs is so extremely irritating and injurious, that it should never be prepared in a room which cannot be thoroughly ventilated. Every laboratory has, or should have, a cupboard communicating with a flue, for dealing with noxious gases; and, in default of this, a shed out of doors is the best place for such experiments. If, however, the chlorine must be prepared in a room, observe, (1) to collect it over warm water, not by displacement; (2) to allow *none* to escape into the air unnecessarily[1]; (3) as soon as the experiments are performed, to throw away the water in the trough,

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[1] The first portions of the gas, which come over mixed with air, should be collected in a large gas bottle; into this, when full, some solution of caustic soda should be poured, and shaken up in the gas until the smell of chlorine has disappeared.

and wash it and the bottles at once with clean water[1]. The displacement method will, from its simplicity and convenience, be employed in the Exercise.

Arrange an apparatus similar to that which was used for preparing carbon protoxide in Ex. 7, substituting for the bent delivery tube the elbow tubes united by a cork-joint used in the last Exercise. A smaller wash-bottle may be used, and should be one-third filled with plain water, its object being only to retain any hydrogen chloride which may pass over.

Place in the flask about 20 grms. of manganese dioxide; and after fitting the apparatus together, pour through the tube-funnel about 60 c.c. of strong hydrogen chloride, and heat the mixture very gently[2]. Collect two large and four small bottles of the gas, applying a little grease to the stopper of each bottle. The yellowish-green colour of the gas will sufficiently show when the bottles are full, especially if a sheet of white paper is held behind each bottle while the gas is passing into it.

1. Immerse a lighted taper in a small bottle of chlorine. It will not be extinguished, but will burn with a dull smoky flame, depositing abundance of soot; and the gas itself will not catch fire.

If the taper is withdrawn quickly when the above result has been observed, the same bottle of gas will serve for the next experiment.

2. Place a piece of moist blue litmus-paper in a bottle of the gas. It will, at the first moment, be reddened, but will speedily lose its colour entirely.

3. Shake up a little water in another bottle of chlorine, preventing the stopper from being fixed immovably by inserting a little slip of paper between it and the neck of the bottle. The water will absorb the gas, as will be proved by its acquiring the colour and smell of chlorine. If a few drops of

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[1] A little ammonia sprinkled on a warm plate is the best means of getting rid of the gas. If the lungs are affected, pour a little alcohol into a test-tube, warm it, and inhale the vapour.

[2] $MnO_2 + 4HCl = MnCl_2 + 2H_2O + Cl_2$.

the solution thus obtained are added to some solution of cochineal or of indigo sulphate in a test-tube, the colour in both cases will be at once discharged.

4. Cut from a stick of phosphorus a small piece about as large as a split pea, observing the precautions given at p. 76. Dry it thoroughly on blotting-paper, pressing, but not rubbing it; place it in a deflagrating spoon, and immerse it in one of the large bottles of chlorine. It will catch fire spontaneously, burning with a greenish flame, and a yellowish crystalline deposit of phosphorus pentachloride will be formed in the bottle.

5. Place some fine brass filings, or, better, some metallic antimony reduced to powder in a mortar, in a paper gutter, and shake the substance little by little into a bottle of chlorine. The metal will catch fire and burn brilliantly when it comes into contact with the gas. Dutch gold-leaf may be burnt in a similar way, by wrapping a few leaves of it round a glass rod and immersing them in the gas.

6. Fill the supplementary pan of the pneumatic trough with warm water. Fill the small stout gas jar with hydrogen gas (by the method given at p. 82), and decant its contents into the larger gas jar, placed in the pan of warm water. Next, fill the small jar with chlorine from one of the small bottles of the gas, and add it to the hydrogen in the larger jar[1]. You have now a mixture of equal volumes of hydrogen and chlorine. Agitate the jar for a second or two, in order to mix the gases intimately, keeping its mouth below the water-level[2]; then transfer some of the mixture to the small jar, remove it to the table and apply a light to its mouth. The gases will unite with explosion[3].

7. Fill the small jar with the remainder of the mixed gases, close its mouth with a glass plate (greased), remove it from the trough and allow it to remain for four or five minutes in a good

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[1] If this is done neatly and rapidly, the loss of chlorine by solution in the water will be very small.

[2] The gases differ so much in density that their mixture by diffusion alone would be comparatively slow.

[3] $H_2 + Cl_2 = 2\,HCl$.

diffused light, not in direct sunshine, for fear of an explosion. If you then examine the contents of the jar, you will find that the colour and characteristic odour of chlorine have disappeared, and that the gas fumes in the air, reddens and does not bleach litmus-paper, and possesses the pungent odour of hydrogen chloride.

EXERCISE 11.

Ethylene.

Apparatus required—Pneumatic trough; flask, tubes, wash-bottle, &c., which were used in Ex. 7; beaker; glass measure; basin of water; three large and two small gas jars; two small gas bottles; taper on wire; glass disc; alcohol; strong hydrogen sulphate; solution of caustic potash; bottle of chlorine; bottle of oxygen.

Measure 30 c.c. of alcohol (rectified spirits) into a beaker or flask capable of holding at least 100 c.c. Place the vessel in a basin of cold water, or hold it in the pneumatic trough, and add to it by degrees three times its volume of strong hydrogen sulphate. The mixture will grow dark, and probably boil at first, from the heat produced by the addition of the acid, and the temperature should be kept down as much as possible by agitating the flask in the surrounding water and adding the acid slowly. While the liquid is cooling you may arrange an apparatus similar to that used for preparing carbon protoxide, placing a dilute solution of caustic soda in the wash-bottle to absorb any sulphur dioxide which may come over, and putting some sand in the flask in order to prevent the mixture frothing up towards the end of the reaction. Pour the mixture of acid and alcohol into the flask, and heat it carefully[1]. When a permanent gas comes over, allow a sufficient quantity to escape, and then collect the four jars full. Be careful so to regulate the heat that the contents of the flask may not froth over into the wash-bottle.

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[1] $C_2H_6O + H_2SO_4 = C_2H_4 + H_2O, H_2SO_4$.

1. The peculiar fragrant odour of the gas will have been already noticed.

2. Place a jar of the gas mouth upwards on the table, and plunge a lighted taper into it. The taper will be extinguished, but the gas will burn with a bright white flame. By pouring some water from a jug into the jar, the gas will be forced out, and the flame seen to better effect.

3. Re-fill the jar used in the last experiment with water, and decant into it one measure of ethylene and three measures of oxygen gas, using the small strong jar as the measure. Fill this last jar with the mixture, and apply a light to it. The gases will unite with an explosion which is even more violent than that produced by the union of oxygen and hydrogen under the same conditions; and the experiment should only be made with small quantities of the gases and in a thick jar. After the experiment, allow the remainder of the mixed gases to escape into the air, to avoid any chance of an accident.

4. Decant into a small gas bottle one measure of ethylene and two measures of chlorine[1] (from the bottle which you reserved in the last Exercise). Insert the stopper, and invert the bottle once or twice, to mix the gases; then place the bottle on the table mouth upwards, and apply a light. The gases will burn quickly, but not explosively, with a reddish flame, and a dense cloud of soot will be formed[2]. This deposition of carbon is due to the stronger affinity of chlorine for hydrogen than for carbon; on which account, when a compound of carbon and hydrogen is decomposed by chlorine at a high temperature, the whole of the chlorine unites with the hydrogen, while the carbon remains uncombined. The luminosity of the flame of ethylene when burning in air (Experiment 2) proceeds from an analogous cause; oxygen, like chlorine, has a stronger affinity for hydrogen than for carbon, and the liberated carbon is raised to a white-heat by the intensity of the chemical action. If a cold porcelain dish is held

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[1] This should be done quickly, to avoid loss of chlorine owing to its solubility in water.

[2] $C_2H_4 + 2\,Cl_2 = C_2 + 4\,HCl$.

for a moment in the flame of ethylene or of ordinary coal gas (which consists chiefly of ethylene and other hydrocarbons mixed with hydrogen) the carbon is cooled and deposited as soot[1]. The next experiment will, however, prove that the nature of the action of chlorine on ethylene at a lower temperature is entirely different.

5. Decant into another gas bottle one measure of ethylene and add one measure of chlorine. Insert the stopper loosely, and place the bottle for a short time in a good diffused light, or in sunshine if possible. The gases will gradually combine, forming an oily liquid which collects in drops on the sides of the bottle: it is ethylene dichloride, or Dutch liquid, as it was formerly called[2].

EXERCISE 12.

Sulphur Dioxide.

Apparatus required—Flask, elbow tubes, &c., used in Ex. 9; one large and three small gas bottles; taper on wire; glass disc; basin of water; copper clippings; strong hydrogen sulphate; blue litmus-paper; solution of logwood; a few flowers, such as violets or pansies.

This gas, which was formed in Ex. 1, when sulphur was burnt in oxygen, is best prepared by the action of strong hydrogen sulphate on mercury, but as that metal is somewhat expensive, copper may be substituted. It is very soluble in water, and should therefore be collected by displacement; which, from the great density of the gas, may be readily accomplished.

Arrange an apparatus similar to that used for preparing hydrogen chloride. Place in the flask 20 grms. of mercury, or

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[1] This difference of affinities is less marked in the case of oxygen; and when, as in Bunsen's burner, gas is burnt with a full supply of air, the whole of both the hydrogen and the carbon is consumed, and the flame possesses little or no luminosity and deposits no soot.

[2] $C_2H_4 + Cl_2 = C_2H_4Cl_2$.

of copper clippings, and add about 60 c.c. of strong hydrogen sulphate[1]. Heat the mixture, and when effervescence begins, collect four bottles of the gas, using a lighted taper, as in the case of hydrogen chloride, to ascertain when the bottles are full. Take the flask away into the open air as soon as enough gas has been collected.

1. The suffocating odour of the gas, and its action on a lighted taper will have been already noticed. From its great density a taper may be extinguished by pouring the gas upon it as directed in the case of carbonic acid, p. 90. Its action on test-paper may be tried by placing a piece of blue litmus-paper in a bottle of the gas, when it will be immediately reddened.

2. Its solubility in water may be shown, as in the case of hydrogen chloride, by replacing the stopper of a bottle of the gas by a glass plate, inverting it, and withdrawing the glass plate under water.

3. Its bleaching action on vegetable colours will be best demonstrated by placing a few violets or pansies in a bottle of the gas. It does not, however, discharge the colour so completely as chlorine, and if the flowers are dipped in a very dilute solution of caustic soda, their colour will be restored. Nor is its action the same on all vegetable colours. Logwood is bleached slowly, while indigo is not at all affected; and if a variegated pansy or hyacinth is placed in the gas, some of the colours, e.g. the reds, are speedily bleached, while the greens resist a prolonged action of the gas.

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[1] $2\,H_2SO_4 + Hg = HgSO_4 + 2\,H_2O + SO_2$.

SECTION III.

(A) PREPARATION OF REAGENTS USED IN THE LABORATORY.

A 'reagent' may be defined as a substance which the chemist employs in order to detect other substances. For example, in examining the properties of carbon dioxide (p. 91) you poured some lime water into the gas, and found that a white precipitate was produced which possessed certain definite properties. In examining other gases you would find that lime water produces no such precipitate when brought in contact with them. Lime water is called a 'reagent,' and is employed for the detection of carbon dioxide.

EXERCISE 1.

Solution of Hydrogen Sulphide.

Apparatus required—Flask with tube-funnel and elbow tube, used in Sect. 2, Ex. 9; retort-stand; wire-gauze; glass disc; two narrow-mouthed bottles, holding 300 c.c.; one ditto, holding 200 c.c.; two small gas bottles; corks; cork-borers; glass tubing, 5 mm. in diameter; fish-tail burner; three square file; india-rubber tubing for connectors; two porcelain dishes; glass jet; blotting-paper; iron protosulphide; hydrogen sulphate; lead acetate; bleaching powder; distilled water.

Adapt sound corks to two narrow-mouthed bottles, holding 300 c.c. Prepare four right-angled elbow tubes of glass tubing about 5 mm. in diameter; two of them with branches of equal length, about 6 cm.; the other two with unequal branches, the shorter about 6 cm., the longer about 20 cm. in length, or of sufficient length to reach, when fitted into the cork, nearly to the bottom of the bottle. Bore two holes in each cork and fit into it one long and one short elbow tube.

Fit up a flask with funnel and elbow tube, similar to that used in Sect. **2**, Ex. 9. Place in it about 40 grms. of iron sulphide, previously broken up with a hammer into lumps about as large as peas. Pour into the flask enough water to cover the iron sulphide, and fill the bottles about three-fourths full of distilled water; replace the corks and connect the tubes, as shown in Fig. 41, with short pieces of india-rubber tubing, so that the gas may bubble through the water in each bottle successively.

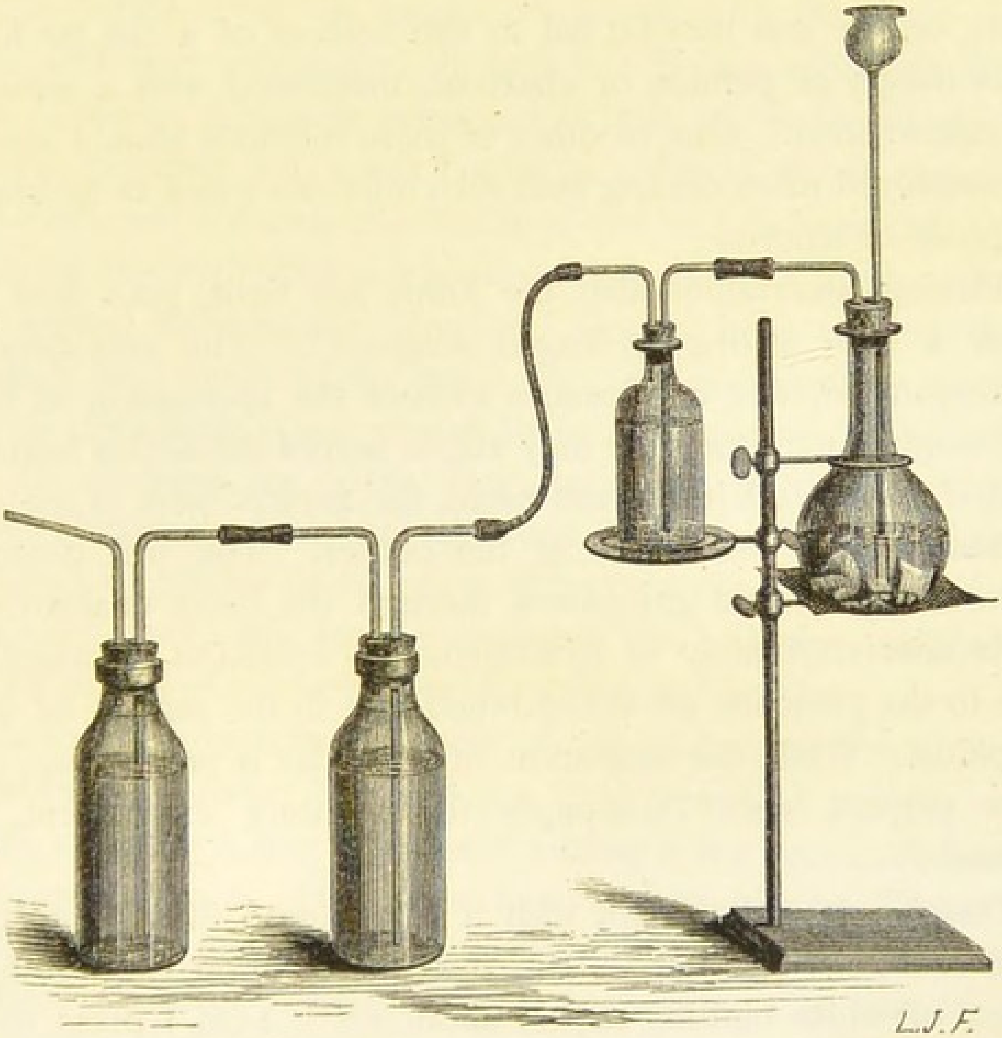

Fig. 41.

The above apparatus will serve for general use when a liquid is to be saturated with a gas. It will be necessary in some cases, and indeed not inexpedient in all, to interpose a small wash-bottle containing a little water between the generating flask and the first bottle, for the purpose of retaining any acid or other impurities which may come over with the gas. This bottle may be supported upon a glass disc placed on one of the rings of the retort-stand, as shown in the figure.

It will also be usually necessary to provide some means of getting rid of any excess of gas which may escape absorption by the water. This may be done most simply by attaching to the elbow tube proceeding from the last bottle a long tube passing through the window or, better, into a chimney or stove-flue; or, the gas may be led to the bottom of a tall jar filled with lumps of pumice or charcoal, moistened with a solution of caustic soda. One or other of these methods should always be employed when dealing with such injurious gases as hydrogen sulphide or chlorine.

Having ascertained that the joints are tight, pour into the flask a little strong hydrogen sulphate[1]. The evolution of hydrogen sulphide commences without the application of heat (although some little time may elapse before the action begins), and, if the stream is not too rapid, the greater part of the gas is absorbed by the water in the bottles. You will observe, however, that some gas passes through the water unabsorbed. This consists mainly of hydrogen, the evolution of which is due to the presence of uncombined iron in the sample of iron sulphide. While the saturation of the water is proceeding, you may prepare some test-paper for a future experiment, as follows:—

Dissolve in a porcelain dish a small lump of lead acetate in 8 or 10 c.c. of water[2]. Dip into the solution two or three strips of white blotting-paper, about 10 × 2 cm.; press them between folds of blotting-paper to remove surface-moisture, and place them in a wide-mouthed, closely stopped bottle.

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[1] $FeS + H_2SO_4 = FeSO_4 + H_2S$.

[2] This should be done, if possible, in another room, lest the substance should be acted on by the gas which may unavoidably escape.

When a steady stream of hydrogen sulphide has passed through the bottles for five or six minutes, disconnect the first bottle, and connect the other directly with the flask. Ascertain whether the water is saturated with the gas, in the following way. Take out the cork and tubes, close the bottle tightly with the thumb or palm of the hand, and shake it briskly for a few seconds in order to bring the water thoroughly in contact with the gas. Hold the bottle in a slanting position with its mouth downwards, and relax the pressure of the hand, noticing whether any bubbles of air enter the bottle, or whether on the contrary the liquid is forced outwards. In the latter case the water is fully saturated with the gas; in the former case the bottle must be again connected with the flask as at first, and more gas passed through it. Before replacing the bottle, however, you may pour out a little of the solution of hydrogen sulphide into a gas bottle, in order to try an experiment which will illustrate the action of certain 'disinfectants' in destroying the hydrogen sulphide which is evolved from putrefying animal substances. You will already have noticed the extremely fetid smell of the gas. Put into the bottle a small lump of 'bleaching powder,' and shake the mixture. You will find, on opening the bottle, that the smell of the gas has entirely disappeared, a faint chlorous odour being alone perceptible. Bleaching powder is a substance containing calcium hypochlorite, and this has decomposed the hydrogen sulphide with formation of calcium chloride, water, and sulphur.

You will thus be able to get rid of any of the gas which may escape into the room by placing a little bleaching powder on a plate, covering it with water, and adding a few drops of dilute hydrogen sulphate. Hydrogen hypochlorite will be liberated and diffuse into the air, decomposing the hydrogen sulphide in the same manner as the calcium hypochlorite. Do not, however, add too much acid to the bleaching powder, lest the remedy should prove less endurable than the evil it is intended to cure.

When no more gas is absorbed by the water in the first bottle, it may be withdrawn, and a little more gas passed through the water in the second bottle, which is already partially saturated by the excess of gas which has passed through the first bottle. Finally, the whole of the solution should be poured into a larger bottle, which should be kept, well corked, in an inverted position, resting in the angle at the corner of a shelf, or in a tumbler or test-glass.

Immediately after disconnecting the bottles, before putting away the apparatus, you may examine one or two more of the properties of the gas.

1. Connect a glass jet with the end of the elbow tube of the generating flask; pour a little more hydrogen sulphate into the flask, if necessary, to obtain a sufficient stream of gas, and apply a lighted match to the gas issuing from the jet[1]. It will take fire and burn with a blue flame. Bring a small gas bottle over the flame, so that the gas may burn in it for a few seconds, in order to retain the products of combustion. Moisture will be deposited, and the presence of sulphur dioxide in the bottle will be proved by its characteristic odour[2].

2. Take out of the bottle one of the strips of paper soaked in lead acetate which you prepared just now; extinguish the burning gas, and hold the strip of paper in front of the jet. It will be instantly blackened, owing to the formation of lead sulphide. This is the most delicate test for the presence of hydrogen sulphide, and will serve, for instance, to detect the presence of traces of it in coal gas.

Finally, take the apparatus to pieces, at once, in the open air, and fill up the flask with water two or three times, to wash away all traces of hydrogen sulphate from the iron sulphide. The latter may then be dried and put away for future use.

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[1] A gentle heat may now be applied with advantage, if the action is slow.

[2] $2\,H_2S + 3\,O_2 = 2\,H_2O + 2\,SO_2$.

EXERCISE 2.

Solution of Chlorine.

For this purpose an apparatus similar to that which was used in the last Exercise is requisite. The small wash-bottle is necessary in this case, to retain hydrogen chloride which may distil over: the other bottles need not be so large as those used for hydrogen sulphide, as less of the chlorine water is likely to be wanted, and it does not keep well.

Prepare the gas as already directed (Sect. **2**, Ex. 10), and pass it into the water until the latter assumes a perceptible yellow colour. It will be better to stop short of the point of saturation, as a comparatively weak solution of chlorine is all that is required for analytical purposes. It should be kept in a stoppered bottle, protected carefully from the light, which causes its decomposition[1]. A good method of protecting it is, to paste over the bottle two or three thicknesses of brown paper, through which, when it is dry, two narrow slits should be cut on opposite sides of the bottle, in order that the quantity of solution contained in the bottle may be seen.

EXERCISE 3.

Tin Chlorides.

Apparatus required—Iron ladle, or crucible; pan of water; flask, holding 300 c c.; retort-stand; sand-bath; argand burner; funnel, 7 cm. in diameter; filters; flask, with tubes, &c., used in the preceding Exercise; metallic tin; hydrogen chloride; distilled water.

Granulate some tin in the same way in which zinc was granulated in Part I, Ex. 1, taking care to use a perfectly clean

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[1] The bottles of deep yellow glass, used in photography, answer very well for this purpose.

ladle or crucible. Place about 20 grms. of it in a 300 c.c. flask, and pour on it 50 c.c. of strong hydrogen chloride. Digest the acid on the metal for some time at a moderate heat, taking care that some of the tin may always remain undissolved[1]. When the action has ceased, dilute with 50 c.c. of water, and filter off the fluid into a bottle in which a few pieces of granulated tin have been placed. Divide the filtrate into two portions; keep the one in the bottle, closely stopped, as tin protochloride (stannous chloride); transfer the rest of the solution to one of the bottles used for preparing solution of chlorine, and pass chlorine gas through it (Ex. 2) until it becomes slightly yellow[2]. Finally, boil it in a flask to get rid of the excess of chlorine (previously fitting a tube to the flask to carry off the chlorine into the chimney or through the window); filter once more if the solution is not clear, and keep it in a bottle labelled 'Tin Perchloride.'

Iron perchloride is easily prepared in a similar way, using coils of clean iron-wire, or iron tacks in place of granulated tin.

EXERCISE 4.

Hydrogen and Silicon Fluoride.

Apparatus required—Florence flask; glass tubing, 7 or 8 mm. in diameter; corks; cork-borers; fish-tail burner; argand burner; retort-stand; wire-gauze; gas jar, 18 × 5 cm.; tube-funnel; funnel, 10 cm. in diameter; filter, or circular piece of linen, about 20 cm. in diameter; bottle, holding 200 c.c.; test-tube; fluor spar; sand; hydrogen sulphate; mercury; strontium nitrate; distilled water.

Take a piece of glass tubing about 60 cm. long, and of rather larger internal diameter than the ordinary gas delivery tube. Having slightly fused the ends in the gas-flame, bend it twice to a right angle in the same direction, making the first bend about 10 cm. from one end, and the second about 30 cm.

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[1] $Sn + 2 HCl = SnCl_2 + H_2$.

[2] $SnCl_2 + Cl_2 = SnCl_4$.

from the other end. Select a Florence flask of stout glass (since it is liable to be corroded by the hydrogen fluoride, and will soon give way if the bottom is thin), fit the shorter branch of the delivery tube to it, by a perforated cork, and support it in the retort-stand with a piece of wire-gauze under it. Place under the outer extremity of the delivery tube a cylindrical gas jar containing a stratum of mercury about 12 mm. deep, into which the end of the delivery tube must dip. This stratum of mercury is quite essential, since it acts as a valve and prevents the entry of water into the tube, which would then soon be obstructed by the silica which is one of the products of the decomposition of the silicon fluoride.

Reduce 30 grms. of fluor spar to a coarse powder in the mortar, mix it with twice its weight of fine sand (that which is called 'silver-sand' is the best), and transfer the mixture to the flask. Pour on it through an acid funnel 60 c.c. of strong hydrogen sulphate, and stir the whole with a glass rod until it is thoroughly incorporated. Replace the cork and delivery tube, and after seeing that the end of the latter is completely covered by the mercury, fill up the jar or test-glass with distilled water, pouring it gently down the sides of the vessel so that none may enter the delivery tube through a disturbance of the surface of the mercury. Heat the flask very slowly with an argand burner or spirit lamp. The reaction between the fluor spar and the acid requires only a moderate temperature, and if too much heat be applied, the mixture froths up inconveniently, and may pass over into the water[1]. When the gas begins to come over, each bubble as it emerges from the mercury becomes coated with a film of silica, which is left behind when the bubble reaches the surface of the water, as a shrunken, collapsed bag[2]. If a strong solution of hydrogen and silicon fluoride be wanted, it is a good plan to set up another similar apparatus, with a delivery tube leading into the same jar of water. The time necessary to obtain the required solution is thus diminished by one half.

[1] $2\,CaF_2 + SiO_2 + 2\,H_2SO_4 = 2\,CaSO_4 + 2\,H_2O + SiF_4$.

[2] $3\,SiF_4 + 4\,H_2O = 2\,H_2SiF_6 + SiH_4O_4$.

Increase the heat slightly if the stream of silicon fluoride becomes slow, and continue it until no more gas comes over[1]: then take the delivery tube quickly out of the solution by raising the retort-stand, and filter the liquid from the gelatinous silica through a paper, or more conveniently, a fine linen filter; care being taken that none of the mercury falls on the filter. The silica thus obtained is very pure, and is hence worth the trouble of washing, an operation which takes some time, and must be done very thoroughly. After a final rinse with warm water, the filter containing the silica may be left to dry, either spontaneously or in a hot-air cupboard.

It is best to keep the solution of the acid, not in a glass, but in a gutta-percha bottle, since the former is always acted upon to a certain extent. Before using it as a test for barium, see that it contains no hydrogen sulphate, which may have been carried over mechanically, if the action has proceeded too rapidly.

Dissolve a few small crystals of strontium nitrate in 10 c.c. of water, and add to the clear solution about 5 c.c. of the acid which you have prepared. If a precipitate or turbidity is produced immediately, or within a few minutes, hydrogen sulphate is present.

If the result of the above test is satisfactory, dissolve a crystal or two (about 0.5 grm.) of barium chloride in 10 c.c. of water, and add to the solution a few drops of the acid. An immediate crystalline precipitate should be produced; otherwise the solution of the acid is not sufficiently strong, and should have more silicon fluoride passed through it, in the manner above described.

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[1] Do not, however, apply a strong heat, or hydrogen sulphate may distil over, and render the solution useless for analytical purposes.

(B) PREPARATION OF NORMAL SOLUTIONS OF REAGENTS.

It is intended that these solutions shall be made of such a strength that volumes which bear a simple relation to each other may contain weights of the different substances which correspond to the weights indicated by their chemical formulæ. It will thus be possible by a little care in measurement to avoid the risk of adding too much or too little of the reagent required to effect a decomposition.

The formulæ printed in italics represent absolute weights, the centigramme being taken as the unit. Thus, H_2 standing for two parts by weight of hydrogen, $\mathit{H_2}$ represents 2 centigrammes of hydrogen. Again, '$(\mathit{H_4 N})_2 \mathit{C\, O_3}$ in 5 c.c.' signifies that 5 c.c. of the solution contain 96 centigrammes of ammonium carbonate; and '$\mathit{Ba\, Cl_2}$ in 20 c.c.' signifies that 20 c.c. of the solution contain 208 centigrammes of barium chloride. If, then, it is desired to precipitate all the barium from 20 cc. of the latter solution, it will only be necessary to measure out 5 c.c. of the solution of ammonium carbonate for the purpose[1].

Reagents, even though procured, as they ought to be, from a trustworthy chemist, are liable to contain impurities; and before use they should be invariably tested with care, and rejected at once (at least for analytical purposes) if found impure[2].

The more usual impurities are mentioned in connection with each substance, together with simple tests for detecting them.

1. Ammonium Carbonate ($(\mathit{H_4 N})_2 \mathit{C\, O_3}$ in 5 c.c.). Dissolve 17 grms. of the pure white sublimed salt in 75 c.c. of distilled

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[1] $Ba\,Cl_2 + (H_4 N)_2 C\, O_3 = Ba\, C\, O_3 + 2\,(H_4 N)\, Cl$.

[2] In most cases, where the quantity purchased is small, it will probably be best to exchange it for a pure sample, and not to attempt to purify it yourself.

water, add 15 c.c. of solution of ammonium hydrate (sp. gr. 0.96), place the solution in a measure, and add more water until the whole measures 100 c.c.

The salt often contains ammonium chloride and sulphate, and also iron chloride. If either of the two latter are present the salt is unfit for use, and another sample should be obtained.

(*a*) Heat a small piece of it on perfectly clean platinum foil over a Bunsen's burner. It should volatilise entirely.

(*b*) Add to a portion of the solution in a test-tube a drop of ammonium sulphide. If the solution becomes dark, or if a black precipitate is formed, iron is probably present.

(*c*) Add to another portion of the solution pure dilute hydrogen nitrate, drop by drop, until no further effervescence occurs and a drop of the liquid placed on blue litmus-paper turns it decidedly red : then add a drop of solution of barium chloride. If a cloudiness is produced, a sulphate is probably present.

(*d*) Acidify another portion with hydrogen nitrate, as in *c*, and add a drop of solution of silver nitrate. If a cloudiness is produced, a chloride is probably present.

2. Ammonium Chloride ($(H_4N)Cl$ in 5 c.c.). Dissolve 10.7 grms. of the pure salt in 80 c.c. of water, and dilute with water to 100 c.c.

The salt may contain ammonium sulphate and iron chloride. It should be tested in the same manner as the carbonate (with the omission of Experiment *d*), and rejected, except for making ammonia, if found impure. Both these impurities may be removed by recrystallisation.

3. Ammonium Hydrate ($(H_4N)HO$ in 5 c.c.). Dilute 50 c.c. of solution of ammonia (sp. gr. 0.96, containing 7 grms. of ammonium hydrate in 100 c.c.) with 50 c.c. of water.

The solution should leave no residue on evaporation at a temperature not much above 100°, and should give little or no precipitate on addition of an equal volume of lime water.

4. Ammonium Molybdate ($(H_4N)HMoO_4$ in 30 c.c.). Dissolve 6 grms. of the salt in 80 c.c. of dilute hydrogen nitrate, warming the mixture very gently; dilute it to 100 c.c., and allow it to stand for a day before filtering it, in order that any traces of ammonium phospho-molybdate may separate.

5. **Ammonium Oxalate** ($(H_4N)_2C_2O_4$ in 30 c.c.). Dissolve 4.1 grms. of the salt in 60 c.c. of water, and dilute to 100 c.c.

6. **Ammonium Sulphide** ($(H_4N)_2S$ in 5 c.c.). The solution usually sold varies in strength, but may generally be diluted with three times its bulk of water for use[1].

It may be easily prepared by saturating 50 c.c. of solution of ammonium hydrate (sp. gr. 0.96) with hydrogen sulphide (p. 108), and then adding 50 c.c. of the same solution of ammonium hydrate.

The solution is colourless at first, but soon becomes yellow, owing to the formation of ammonium polysulphides. It still, however, remains fit for use in analysis for some time.

The solution is liable to contain a sulphate, and possibly a calcium salt. Test one portion with a drop of solution of barium chloride, and another portion with solution of ammonium oxalate. No precipitate should be produced in either case.

7. **Barium Chloride** ($BaCl_2$ in 20 c.c.). Dissolve 12.2 grms. in 80 c.c. of water, and dilute to 100 c.c.

The purity of the salt may be tested as follows. Place about 5 c.c. of the solution in a test-tube, heat it nearly to boiling, and add a slight excess of dilute hydrogen sulphate. While the precipitate of barium sulphate is subsiding, get ready a filter (of Swedish paper), wash it two or three times with warm water, then filter off the barium sulphate and evaporate the filtrate to dryness on a clean watch-glass. No solid residue should be left.

8. **Calcium Chloride** ($CaCl_2$ in 10 c.c.). Dissolve 11.1 grms. of the dry salt (or 22 grms. of the crystallised salt) in 80 c.c. of water, and dilute to 100 c.c.

This reagent is, however, required but seldom, and may be dispensed with.

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[1] For a method of determining its strength, see Part IV, Sect. 2.

The salt often contains iron perchloride, from which it may be purified by crystallisation from a small quantity of water. The solution should be quite neutral to test-paper, and should not become dark coloured, or give a precipitate when a drop of solution of ammonium sulphide is added.

9. Calcium Hydrate ($Ca\,H_2\,O_2$ in 540 c.c.). For the method of making this solution, see Sect. **1**, Ex. 5.

10. Calcium Sulphate ($Ca\,S\,O_4$ in 500 c.c.). This solution should be made in the same manner as that of calcium hydrate.

11. Cobalt Nitrate ($Co\,(N\,O_3)_2$ in 20 c.c.). Dissolve 7.25 grms. of the crystallised salt in 40 c.c. of water, and dilute to 50 c.c.

12. Hydrogen Acetate ($H\,C_2\,H_3\,O_2$ in 5 c.c.). The acid of which the sp. gr. is 1.04 is nearly of the right strength. It often contains a chloride and a sulphate, but traces of these will not interfere with its use in analysis.

(*a*) Evaporate a portion of the acid on a watch-glass. It should leave no residue.

(*b*) To another portion add three or four drops of dilute hydrogen nitrate, and then a drop of solution of silver nitrate. If a cloudiness is produced, a chloride is present.

(*c*) To another portion add a drop of solution of barium chloride. If a cloudiness is produced, a sulphate is present.

13. Hydrogen Chloride ($H\,Cl$ in 1 c.c.). The acid of which the sp. gr. is 1.158 is very nearly of the right strength. It is liable to contain a sulphate, and also iron and arsenic, and must, for analytical purposes, be obtained free from these impurities.

(*a*) Dilute a portion with three or four times its volume of water, and test for a sulphate by solution of barium chloride.

(*b*) The presence of iron is generally indicated by the yellow colour of the acid. Add to a portion a drop of hydrogen nitrate, and boil the mixture for a few seconds; then add an equal volume of water, and allow it to cool. If the liquid turns red on addition of a drop of potassium sulphocyanide, iron is present.

(*c*) Arsenic must be tested for by Marsh's method (see under Arsenic, Sect. **5**).

14. Hydrogen Chloride, dilute (HCl in 5 c.c.). Place 20 c.c. of the strong acid in a measure, and dilute with water to 100 c.c.

15. Hydrogen Nitrate (HNO_3 in 1 c.c.). The acid of which the sp. gr. is 1.3 is very nearly of the right strength. It is liable to contain lower nitrogen oxides, and also a sulphate and a chloride.

(*a*) It should leave no residue when evaporated on a watch-glass.

(*b*) It should be colourless. If it is yellow, lower nitrogen oxides are present. These, however, are usually found in the acid when it has been exposed to light, and do not in general interfere with its use in analysis.

(*c*) When separate portions are diluted with water and tested with solution of barium chloride and silver nitrate, no cloudiness should be produced in either case.

16. Hydrogen Nitrate, dilute (HNO_3 in 5 c.c.). Place 20 c.c. of the strong acid in a measure, and dilute with water to 100 c.c.

17. Hydrogen Sulphate (H_2SO_4 in 0.55 c.c.). The acid of which the sp. gr. is 1.845 is very nearly of the right strength. It may contain lead and arsenic, and also a nitrate, and must, for analysis, be obtained free from these impurities.

(*a*) Evaporate a few drops on a porcelain dish, taking care not to inhale the dense white fumes of the acid. No solid residue should be left.

(*b*) Dilute a few drops of the acid with 2 or 3 c.c. of water. If the mixture becomes turbid, lead is present.

(*c*) Place a little of the acid in a test-tube and pour, very slowly, down the side of the tube some solution of iron protosulphate, so that the lighter fluid may rest upon, not mix with, the heavy acid. If, after a time, a brown stratum appears at the junction of the two fluids, a nitrate is present.

(*d*) Arsenic should be tested for by Marsh's method (Sect **5**).

18. Hydrogen Sulphate, dilute (H_2SO_4 in 5 c.c.). Measure out 60 c.c. of water into a beaker and add by degrees (see p. 86, note) 20 c.c. of the strong acid, stirring the mixture continually with a glass rod. When the liquid is cool, pour it into the measure, and dilute to 100 c.c.

19. Iron Perchloride (Fe_2Cl_6 in 50 c.c.). Dissolve 2.25 grms. of fine iron-wire in about 30 c.c. of dilute hydrogen chloride, heating the mixture in a small flask. When all the iron is dissolved pass chlorine through the solution until it smells strongly of the gas, then pour it into a porcelain dish and evaporate it to a small bulk. Finally, dilute it with water to 100 c.c. and filter it.

20. Iron Protosulphate ($Fe\,S\,O_4$ in 50 c.c.). Dissolve 5.5 grms. of the pure green crystals in 80 c.c. of cold water, and dilute to 100 c.c. Owing to the great tendency of iron protosalts to absorb oxygen and pass into persalts, the solution cannot be preserved unaltered for any length of time, and will contain an increasing quantity of iron persulphate the longer it is kept.

21. Lead Acetate ($Pb\,(C_2H_3O_2)_2$ in 50 c.c.). Dissolve 6.5 grms. of the salt in 80 c.c. of water, with the addition of one or two drops of hydrogen acetate, and dilute to 100 c.c.

22. Magnesium Sulphate ($Mg\,S\,O_4$ in 50 c.c.). Dissolve 5 grms. of the salt in 80 c.c. of water, and dilute to 100 c.c.

23. Mercury Perchloride ($Hg\,Cl_2$ in 50 c.c.). Dissolve 6.4 grms. of the crystallised salt in 80 c.c. of water, and dilute to 100 c.c.

24. Mercury Protonitrate ($Hg_2(NO_3)_2$ in 50 c.c.). Dissolve 11.2 grms. of the crystallised salt in 80 c.c. of water, add a few drops of dilute hydrogen nitrate, and dilute to 100 c.c. Keep the solution in a bottle containing 2 or 3 grms. of metallic mercury.

25. Platinum Perchloride ($Pt\,Cl_4$ in 50 c.c.). A comparatively small quantity of this substance will be required, and it will on the whole be best to purchase it in the state of solution (1.4 grms. of the salt dissolved in 25 c.c. of water). The method of obtaining it from platinum scraps and residues is given in Part VI.

26. Potassium Chromate ($K_2 Cr O_4$ in 50 c.c.). Dissolve 4 grms. of the salt in 80 c.c. of water, and dilute to 100 c.c.

27. Potassium Ferricyanide ($K_3 Fe (C N)_6$ in 50 c.c.). Dissolve 6.6 grms. of the salt in 80 c.c. of water, and dilute to 100 c.c. The solution, especially if it has been kept for a month or two, is liable to contain traces of potassium ferrocyanide. It should give no blue precipitate, but only a brown colouration when tested with a drop of a solution of pure iron perchloride.

28. Potassium Ferrocyanide ($K_4 Fe (C N)_6$ in 50 c.c.). Dissolve 8.4 grms. of the salt in 80 c.c. of water, and dilute to 100 c.c. Potassium ferricyanide is sometimes present as an impurity: it may be detected by adding to a portion of the solution a drop of solution of silver nitrate. If a reddish-brown and not a white precipitate is found, potassium ferricyanide is present. The salt may easily be purified by recrystallisation.

29. Potassium Hydrate ($K H O$ in 5 c.c.). Dissolve 11 grms. of the substance (which is sold in sticks) in 80 c.c. of water, closing the mouth of the flask with a cork, to prevent absorption of carbon dioxide from the air. When the solution is cool, pour it into the measure, and dilute to 100 c.c.; then transfer it without loss of time to a stoppered bottle, which should not be made of flint glass. It is difficult to obtain pure potassium hydrate, and its solution attacks the glass of the bottles in which it is kept, and takes up carbon dioxide from the air. A fresh solution should therefore be made from time to time; and for some experiments it will be best to dissolve a small lump of the substance in water and use it at once.

The usual impurities are—a carbonate, an aluminate, a chloride, and a sulphate, of which the last two are not important.

(*a*) Add to a portion of the solution pure dilute hydrogen nitrate, until a drop of the liquid placed on blue litmus-paper colours it decidedly red. No effervescence indicative of the presence of a carbonate should be produced. Traces, at any rate, of the two first-mentioned impurities will be found in a solution which has been kept some time: the carbonate derived from the carbon dioxide in the air, the aluminate from the glass of the bottles.

(*b*) Test one portion of the solution which you have acidified with hydrogen nitrate, for a sulphate by adding a drop of solution of barium chloride.

(*c*) Test another portion of the same solution for a chloride, by adding a drop of solution of silver nitrate.

(*d*) Acidify a portion of the solution of potassium hydrate with dilute hydrogen chloride, then add solution of ammonia until the liquid is alkaline to test-paper, and boil the solution. If a flocculent, semi-transparent precipitate is formed after a little time, an aluminate is present.

30. Potassium Iodide (KI in 50 c.c.). Dissolve 3.3 grms. of the salt in 80 c.c. of water, and dilute to 100 c.c.

The salt usually contains a little potassium iodate. It should be bought in the form of colourless, well-defined cubic crystals. The solution should not turn yellow at once when one or two drops of pure dilute hydrogen sulphate are added.

31. Potassium Sulphocyanate ($KCNS$ in 50 c.c.) Dissolve 2 grms. of the salt in 80 c.c. of water, and dilute to 100 c.c.

32. Silver Nitrate ($AgNO_3$ in 50 c.c.). Dissolve 3.4 grms. of the salt in 80 c.c. of water, and dilute to 100 c.c.

33. Sodium Carbonate (Na_2CO_3 in 10 c.c.). Dissolve 28.6 grms. of the crystallised salt in 80 c.c. of water, and dilute to 100 c.c.

The salt is liable to contain sodium sulphate and chloride, and should, for analytical purposes, be free from these impurities.

Add to a portion of the solution dilute hydrogen nitrate as long as an effervescence occurs, warm the liquid until the carbon dioxide is completely expelled, and divide it into two portions. Test one portion for a sulphate by adding a drop of solution of barium chloride, and the other portion for a chloride by adding a drop of solution of silver nitrate. No precipitate should be produced in either case.

34. Sodium and Hydrogen Phosphate (Na_2HPO_4 in 50 c.c.). Dissolve 7.2 grms. of the salt in 80 c.c. of water, and dilute to 100 c.c.

The salt is liable to contain a chloride and a carbonate. These impurities should be tested for in the manner described under the head of Potassium Hydrate, p. 123[1].

35. Sodium and Hydrogen Tartrate ($NaHC_4H_4O_6$ in 20 c.c.). Dissolve 9.5 grms. of the salt in 80 c.c., and dilute to 100 c.c.

36. Starch. Place 0.5 grm. of pure white starch in a flask, and add 80 c.c. of water. Shake the flask until the starch is thoroughly mixed with the water, and then heat the mixture gradually to boiling, occasionally shaking it to prevent any lumps from collecting at the bottom of the flask. When the boiling-point is nearly reached, the liquid will lose its milky appearance and become semi-transparent; the starch-granules have swelled and broken up, and their contents have dissolved in the water. Dilute the solution to 100 c.c., filter it, and transfer it to a bottle. It does not keep in good condition beyond a week or two.

37. Tin Protochloride ($SnCl_2$ in 50 c.c.). For directions for making this solution, see p. 113. A method for determining its strength will be found in Part IV, Sect. **2**.

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[1] A green fungus is almost invariably found at the bottom of bottles in which a solution of this salt has been kept for some time.

SECTION IV.

EXPERIMENTS WITH THE MOUTH BLOWPIPE.

[It is difficult to attach too much importance to practice in the use of the blowpipe. The apparatus required is extremely portable and cheap, and a great many minerals may be analysed with certainty by its employment alone, even when only a minute portion of the substance is available. No branch of chemical study is better adapted to teach the beginner habits of close, accurate observation and economy of material.]

Apparatus required—Blowpipe; blowpipe lamp; piece of platinum wire; piece of platinum foil; two or three pieces of charcoal; small glass mortar; watch-glasses; crucible tongs; test-tube; knife; sodium diborate; sodium carbonate; potassium dichromate; potassium cyanide; strontium nitrate; barium chloride; solution of cobalt nitrate; manganese dioxide; iron peroxide; tin peroxide; silica; bismuth; cadmium; calcium sulphate; dilute hydrogen chloride.

The mouth blowpipe consists, in its simplest form, of a tapering tube, usually bent near its smaller end to a right angle, and termining in a fine jet.

But as the moisture of the breath soon collects in the tube and interrupts the flow of air, a much superior form of blowpipe is that which was introduced by Dr. Black, and which is represented in Fig. 42.

Fig. 42.

All moisture is condensed in the wider part of the tube, while the movable nozzle can be readily unscrewed and cleaned out, if it should become stopped up by soot or oxide. Before the instrument is used, the nozzle should be carefully examined to see that the aperture is clear, round, and not very large, otherwise the cone of flame will be ragged, irregular, and brush-like.

The best fuel to be used with the blowpipe is undoubtedly ordinary coal gas, where it can be obtained. The most efficient form of burner is that represented by Fig. 5 *a*. It consists merely of a short piece of brass tube about 1 cm. in diameter, flattened out at one extremity until its edges form a narrow rectangular aperture about 0.5 mm. broad, inclined at an angle of 70° to the axis of the tube. This tube, which need not be more than 3 cm. long, may be screwed into the same iron foot which serves as the base of the argand burner. The orifice of the burner should not be more than 10 cm. above the table, in order that the greatest steadiness may be secured by resting the arms on the table, while the right hand holds the blowpipe and the left hand holds the support containing the substance to be examined. The blast of air should be directed obliquely downwards, parallel, in fact, with the orifice of the burner. The following figure (Fig. 43, next page) will serve to make the position clear.

If gas is not available, an oil lamp, a tallow lamp, or a spirit lamp fed with a mixture of ten parts spirit of wine and one part turpentine, may be used, or finally, but not preferably, a wax candle. The lamp should have a flat wick, which just before use must be carefully trimmed smooth and divided along the middle with the trimming scissors, so as to leave a furrow along which, and about 2 mm. above it, the blast of air from the blowpipe must be directed.

The first thing to be learnt in the use of the blowpipe is the method of keeping up a regular, continuous blast of air for several minutes without interrupting the ordinary process of respiration. This is by no means difficult. In the ordinary double organ bellows, we can distinguish two essential parts;

(*a*) the lower compartment or 'feeder,' which by its alternate expansion and contraction supplies air intermittently; and (*b*) the upper compartment or 'reservoir,' which receives and stores up the air thrown in by the feeder, and sends it out in a continuous stream at a uniform pressure to the pipes. When the blowpipe is properly used, the lungs and the mouth are acting respectively as the feeder and the reservoir of the bellows; the muscles of the cheeks acting as the weights placed on the reservoir to secure a constant pressure on the gas; the tongue, slightly drawn back and applied to the roof of the mouth, representing the valve between the feeder and the reservoir; and the nostrils representing the aperture through which air enters the feeder.

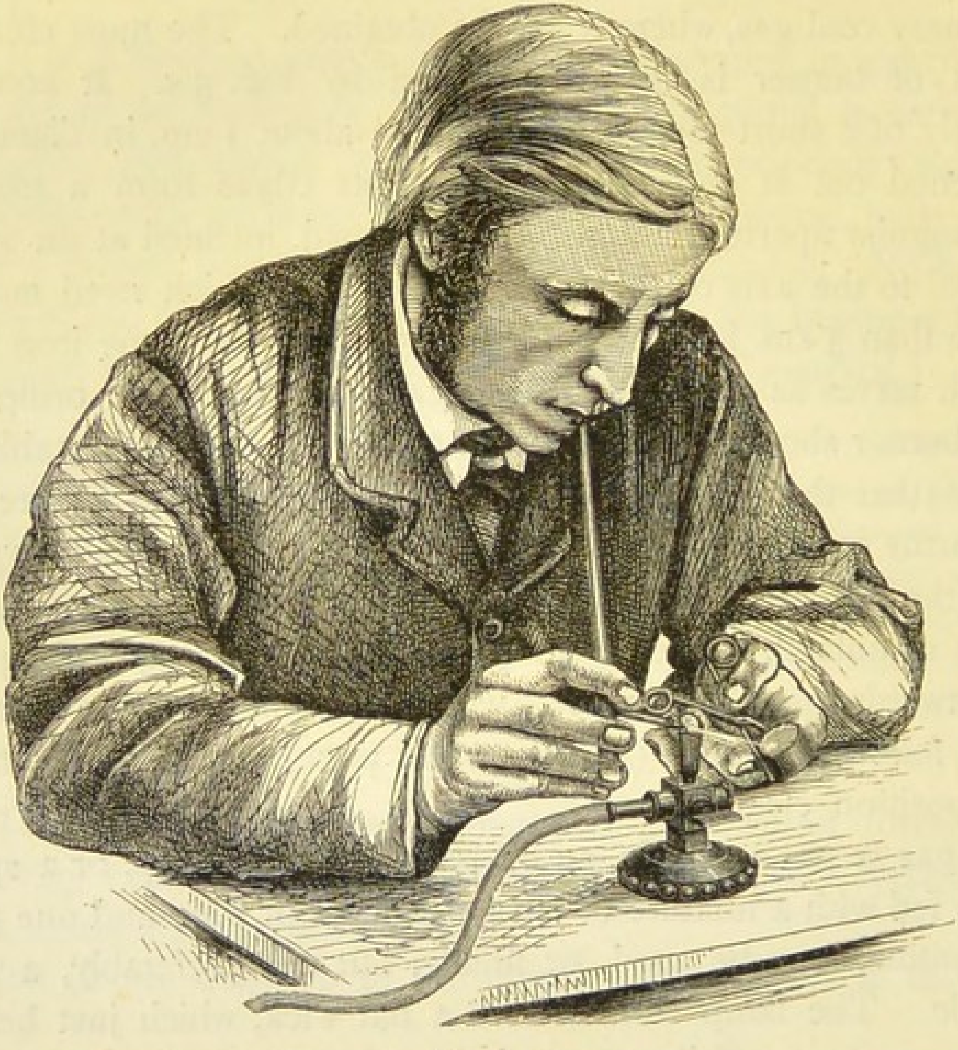

Fig. 43.

These analogies being borne in mind, the following general directions will, it is thought, be sufficient to guide the student in learning the use of the blowpipe; a few minutes' practice being of more value than a prolonged description.

Begin by distending the cheeks with air from the lungs; keep them thus distended while you breathe freely through the nose. In doing this you will have unconsciously placed the tongue in the position it should occupy as a valve between the mouth and the lungs. Now insert the blowpipe between the lips, and expel the air from the mouth through the jet by compressing the muscles of the cheeks. When the stock of air in the mouth is nearly, but not quite, exhausted, introduce a fresh supply of air direct from the lungs, interrupting the ordinary respiration for a moment only, and slightly relaxing the muscles of the cheeks, so that they again become distended with air.

A difficulty will probably be found at first in keeping the pressure constant just at the moment at which air is thus thrown into the mouth from the lungs. A very little practice, however, will enable you almost unconsciously so to balance the action of the muscles of the chest and cheeks, that the latter yield exactly in proportion as the former impel air into the mouth, and thus no variation is perceptible in the blast of air from the jet. When, after one or two trials, you find that you can produce a fairly uniform stream of air for a minute or so, you may bring the jet into the lamp-flame[1] in the position above described, and use the blast of air to deflect the flame. The appearance of the latter will sufficiently indicate the regularity and pressure of the stream of air. It should appear as a well-defined cone of blue light, burning noiselessly, surrounded by a faint nebulous yellowish envelope, which reaches for some distance beyond the apex of the cone. If it flickers, and burns with a roaring noise, either the jet is not introduced sufficiently far into the flame, or the aperture is too large, or the pressure of air too great. If it is irregular in outline, the aperture of the jet is not round

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[1] This should be about as large as the flame of an ordinary candle.

and must be cleaned out and rounded by introducing a large needle; or, if you are using a lamp with a wick, the latter may not be evenly trimmed, the projecting filaments breaking the current of air. If the flame appears as a luminous tongue, either the aperture of the jet is too small, or the stream of air is not propelled with sufficient force; or, finally, the flame itself is too large.

The blowpipe-flame is used for three distinct objects.

1st, and always, to subject substances to a higher temperature than the lamp alone would give.

2nd. To promote the union of oxygen with substances capable of combining with it; in other words, to oxidise them.

3rd. To abstract oxygen from substances which readily part with it; in other words, to reduce them.

The possibility of employing the blowpipe for the last two purposes will be evident if we examine the structure of the lamp-flame before it is deflected by the blast of air. It will be readily seen to consist of at least two distinct parts.

1. A dark central portion, composed of gaseous compounds of hydrogen and carbon, unburnt as yet, since no oxygen can reach them, but raised to a high temperature by the surrounding portions of the flame.

2. A luminous zone, depositing soot on cold bodies placed in it, consisting of the gaseous hydrocarbons in the act of combining with the oxygen of the air; their hydrogen being wholly burnt to water, their carbon only partially burnt to carbon dioxide; the remainder of the carbon being set free and raised to a white heat by the temperature of combustion, thus imparting to the flame all its luminosity. This carbon is itself consumed as it reaches the exterior of the zone.

3. A scarcely visible, ill-defined external envelope, consisting of the gaseous or vaporised products of combustion, viz. carbon dioxide and water, mixed with a great excess of air strongly heated owing to its proximity to the zone of combustion.

It is obvious that a substance placed in the centre of the flame and gradually brought to the exterior will be successively subjected to reduction, simple ignition, and oxidation. The heated hydrocarbons which compose the central portion will, if the substance contain oxygen loosely combined, abstract that oxygen and be converted into carbon dioxide and water. As the substance approaches the exterior of the flame it will arrive at an area of perfect combustion, where the hydrocarbons meet with just enough oxygen to burn them completely, and where, consequently, the temperature is highest.

Finally, when it arrives at the extreme border of the flame, it is in contact with excess of highly heated air, or diluted oxygen, which will combine with it if combination is possible under such conditions.

The cone of flame produced by driving a blast of air from the blowpipe through the lamp-flame is precisely similar in its constitution; its powers being intensified, not changed in kind, by a judicious adjustment of the relative proportions of gas and air.

1. To effect reduction;—more gas and less air will be needed, the object being to burn the gas only partially, and thus to compel it to obtain the oxygen necessary for combustion from the substance submitted to its action. Admit more gas, therefore, until the flame is about 8 cm. high, and, holding the blowpipe-jet at the *border* of the visible flame, blow a gentle stream of air through it, so as to deflect the flame in the form of a long luminous tongue, within the tip of which the substance to be reduced must be held, wholly immersed in the flame, and thus exposed to the same conditions as the ore in the body of a smelting furnace, surrounded by ignited fuel and combustible gases.

2. To effect oxidation;—more air and less gas is needed, the object being not only to burn the latter completely, but also to provide, over and above, a supply of highly heated oxygen, and thus to expose the substance to conditions similar to those which exist on the hearth of a cupelling furnace. Diminish the supply of gas until the flame is only 5 or 6 cm. in height; introduce the blowpipe-jet about 1 mm. within the border of the flame, and blow more strongly so as to produce a well-defined blue cone, surounded by a faintly luminous envelope,

and hold the substance to be oxidised about 1 cm. in front of the tip of the blue cone.

3. To expose a substance to the highest attainable temperature, it must be held just at the tip of the blue cone mentioned above. Here the combustion is complete, and hence we have the maximum temperature which can be produced by the union of the gases.

It must be understood that, where an oil or spirit lamp is used, the raising and lowering the wick will have the same effect as increasing or diminishing the supply of gas to the burner.

The supports for substances to be exposed to the blowpipe-flame are principally of two kinds.

1. Platinum foil or wire: the former in strips about 2 cm. broad and 5 cm. long, held in a pair of crucible tongs or forceps; the latter, No. 26 brass wire-gauge, in pieces about 6 cm. long, terminating in a small ring about 2 mm. in diameter, formed by bending the end of the wire round any small cylindrical body; the tip of the blowpipe-jet answers very well.

Platinum foil is used as a support when substances are to be heated *per se*, to test their fusibility or volatility. The loop of platinum wire serves to hold a bead of melted borax when we wish to examine the colour imparted to it by certain metals.

2. Charcoal, in the form of flat pieces of beech or elm charcoal, which should be carefully selected free from cracks and of close even texture. Good pieces may always be found among ordinary charcoal, sold for burning, and should be cut across the grain with a fine saw into pieces about 1.5 cm. thick. A cross section is always to be preferred to a longitudinal one, since the former absorbs the flux more readily, and does not split when heated. With the point of a knife cut a small conical hole in the charcoal, as shown in section in Fig. 44, so as to form a small crucible for containing the substance; taking care that the surface of the charcoal extends some little way beyond the hole, so that any incrustation

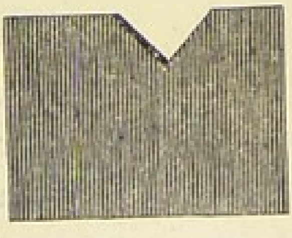

Fig. 44.

may be retained for examination. The charcoal may be conveniently held in a pair of crucible tongs, their bent points being turned upwards, as in Fig. 43.

3. Pieces of hard, difficultly fusible glass tubing, about 6 cm. in length and 3 mm. in diameter internally, sealed at one end and expanded into a small bulb. The shape and method of making these tubes is given at p. 37.

4. Pieces of similar tubing about 8 or 9 cm. in length, open at both ends.

Such supports are extremely useful, when the effect of simply heating a substance is to be tried, as a preliminary to its actual analysis; especially where there is reason to expect that any volatile products may be formed, since these will be retained in the cooler part of the tube for future examination. In general, however, the heat of a Bunsen's burner, or even a good spirit lamp, will be sufficient in such cases. Indeed, no glass tube will long withstand the intense heat of the blowpipe-flame without softening and enclosing the substance under examination.

Pieces of broken porcelain, such as the fragments of an evaporating dish, will almost always serve in place of the platinum foil, when it is not essential that silica or alumina should be excluded. Platinum foil should be used only when nothing else will serve the purpose, since it is not easy to keep it clean, and it is soon acted on and pierced with small holes which render it useless.

The pieces of platinum wire for blowpipe use may be conveniently preserved in the following way:—

Fit a cork to a wide-mouthed 1 oz. bottle; then with a sharp cork-borer cut two or more holes in the cork, about 5 mm. in diameter. Push out of the borer the small cylinders of cork extracted from the holes, and make a pin-hole along the axis of each. Pass one end of the piece of platinum wire through the pin-hole and bend it into a hook on the other side of the cork to secure the wire in its place. Lastly, fill the bottle nearly to the neck with dilute hydrochloric acid; cork it, and fit into each hole a cork cylinder with wire attached, so that the end of the wire may be immersed in the dilute acid. By this means the wires will be kept always clean and ready for use; while the cork cylinders will serve as convenient handles and prevent the wires from being lost. Fig. 45 represents a bottle thus fitted.

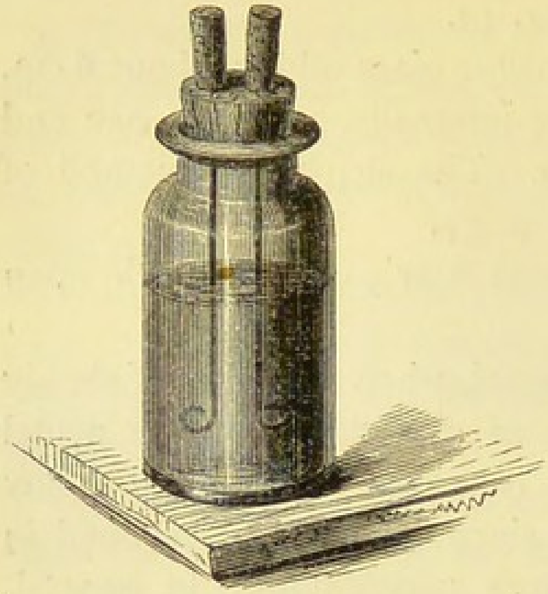

Fig. 45.

Having thus learnt the general principles on which the use of the blowpipe depends, you may proceed with the following experiments, which will serve to illustrate the practical employment of the instrument in analysis. Other experiments will be found in their proper places in Sect. **5**.

In the first place, light the lamp and see that it burns with a steady flame, and that the wick, if it is an oil lamp, is properly trimmed and divided down the middle. If the flame wavers, owing to draughts in the room, a screen of some kind must be arranged to protect it. Next examine the blowpipe-jet, and clean it out, if necessary, with a needle, until it deflects the flame into a steady pointed cone. Arrange the lamp at such a height that the hand which holds the substance in the flame may rest steadily on the table, while the other hand holding the blowpipe may also be firmly supported either on some part of the stand of the lamp itself (Fig. 43) or on a wooden block placed close to it. A little borax, sodium carbonate, and potassium cyanide in powder should be placed in watch-glasses near at hand.

1. Make a small loop at the end of one of the bits of platinum wire, moisten it with a drop of distilled water, dip it into the sodium carbonate, and hold it in the hottest part of the blowpipe-flame: notice the intense yellow colour imparted to the flame by the salt.

2. Repeat the preceding experiment, using strontium nitrate instead of sodium carbonate. The flame will be coloured crimson; and if barium nitrate be substituted for the strontium salt, it will tinge the flame yellowish green.

The platinum wire must be rendered perfectly clean between each of the above experiments by washing it with a few drops of dilute hydrogen chloride, and heating it strongly until it does not of itself impart any colour to the flame.

3. Heat the loop at the end of the platinum wire, and dip it into powdered borax placed in a watch-glass. A portion will adhere to the heated wire, and must be again brought into the blowpipe-flame. It will at first swell up and give off water, but will finally fuse into a colourless transparent bead, which undergoes no change on being further heated. When the bead is cold moisten it slightly with solution of cobalt nitrate, and again heat it slowly before the blowpipe, holding it in the hottest part of the flame, i. e. at the apex of the blue cone. When all action appears to have ceased and the bead is as clear as at first, withdraw it from the flame and allow it to cool. It will now be seen to have acquired a deep blue colour, if the cobalt salt has been taken in the right proportion. If the bead appears nearly black, too much of the cobalt nitrate has been added, and the wire after being again heated must be tapped on the edge of the table, so as to shake off the greater part of the still fluid bead, then dipped into the borax and again fused before the blowpipe. If the blue colour is very faint, a little more of the cobalt nitrate must be taken; but the delicacy of the reaction is so great that it is hardly possible to take too little of the substance.

4. Repeat the above experiment, using a clean platinum wire, and taking, instead of cobalt nitrate, potassium dichromate. The powdered salt may be placed in a watch-glass and the borax bead, while still red-hot, should be just brought into contact with it so as to take up a very minute quantity, and then held in the hottest part of the blowpipe-flame for a few seconds. It will, on cooling, appear of an emerald green colour.

In the preceding experiments it is immaterial into what part of the flame the substances are introduced, since the colour imparted to borax glass by cobalt and chromium is the same whether the oxidising or reducing flame be employed to melt the bead. In the following Exercises, however, the distinction between the two flames must be carefully observed, and the bead should be kept steadily for at least half a minute in the one or the other flame, as directed.

5. Form a borax bead as above, and, having added to it a trace of manganese dioxide, heat it in the oxidising flame, holding it 1 cm. at least in front of the visible flame. The bead will acquire an amethyst colour. Now heat it again, but this time in the reducing flame, holding it so that the luminous portion shall completely envelope it, and taking care that it does not, even for a moment, remain in the outer border of the flame. It will now be found that the amethyst colour has nearly or completely disappeared, and the bead is as colourless as at first. But if it be again held in the oxidising flame, the colour will return.

6. Repeat the last experiment, using iron peroxide instead of manganese. The bead will, in the oxidising flame, be orange coloured when hot, yellow when cold; in the reducing flame dull green when hot, becoming paler as it cools. It will be interesting and useful to keep a set of characteristic borax beads formed as above indicated, as a help to the memory, and as a standard of comparison. Such a set may be conveniently made as follows. Seal one end of a glass tube about 6 cm. long and 2 mm. internal diameter. Place near the blowpipe a deep porcelain dish, about 10 cm. in diameter (a large tea-cup will serve the purpose), perfectly clean and dry. After making the bead in the usual way, and while it is still fluid, tap the platinum wire somewhat obliquely on the edge of the porcelain dish. The bead will detach itself and roll round the side of the dish, finally coming to rest at the bottom. It will be found to have preserved its spherical shape; and if the colour is satisfactory, it should be placed at once in the glass tube, since its surface would soon effloresce in the air. Other beads may be formed

in the same way, and dropped one by one into the tube, from which they should not much differ in diameter. When the set is complete, the tube should be heated about 5 or 6 mm. above the last bead, drawn out and sealed, thus preserving the beads from further change.

The following list includes nearly all the substances which impart characteristic colours to borax glass:—Chromium, manganese, nickel, cobalt, iron, copper, uranium.

Two beads should be preserved in each case; the one showing the colour imparted by the substance in the oxidising, the other in the reducing flame.

7. Fuse a little sodium carbonate in a loop of platinum wire before the blowpipe. It will give a bead which is clear while hot, but opaque when cold. Now add to this bead a small quantity of powdered silica, and again fuse it in the hottest part of the flame; an effervescence will take place, owing to the liberation of carbon dioxide. You will, if enough silica has been added, obtain a bead which retains its transparency when cold.

A sodium silicate, such as you have here obtained, is the basis of ordinary glass.

8. Prepare a charcoal support as described, p. 132. Mix together in a mortar equal parts of tin dioxide and potassium cyanide, and transfer a small quantity of the mixture to the hole in the charcoal, taking care that none is spilled over the surface of the support. Bring the mixture into the reducing flame of the blowpipe, holding the charcoal slightly inclined towards the jet, so that the flame may play directly into the hole. The mass will readily melt, and bright globules of metallic tin will make their appearance, while the flux will be gradually absorbed by the charcoal. Maintain the heat steadily until the scattered particles of metal have run together into one globule, and the flux has disappeared; then withdraw it quickly from the flame and let it cool. Try the malleability of the metal by detaching it with a knife from the charcoal, placing it in the mortar and pressing the pestle strongly down upon it. It will be found to spread out under the pestle into a flat plate, not crumbling to powder or even tearing at the edges.

9. Make a hole in another part of the charcoal support, and place in it the globule of tin which you have just obtained. Fuse it in the reducing flame, and notice that its surface can be kept quite bright so long as it is held in that flame. Remove it into the oxidising flame, slightly increasing the blast of air. It will now become tarnished, a crust of white oxide being formed, which appears to grow out of the metal. Bring it again into the reducing flame, and a bright globule of metal will be again formed. The reduction should be assisted by the addition of a minute quantity of sodium carbonate.

10. Try similar experiments with metallic bismuth, and notice that the globule of metal is brittle and crystalline, crumbling under the pressure of the pestle; and that a yellow incrustation is formed on the charcoal, round the metal.

11. Repeat the above experiments, using cadmium instead of bismuth. The substance will fuse, and the charcoal will be covered with a reddish brown incrustation of cadmium oxide, formed (owing to the volatility of the metal) at some distance from the cavity.

12. Mix together a little plaster of Paris and charcoal (about equal parts of each) in a mortar; and place some of the finely powdered mixture on a piece of platinum foil, which should be turned up at the edges so as to form a cup. Heat it strongly in the reducing flame for half a minute, and when it is cool detach the ignited mass from the platinum foil, place it in a test-tube and pour on it a few drops of hydrogen chloride, diluted with twice its bulk of water. Notice the effervescence which takes place, the odour of the escaping gas, and its action on a piece of filter-paper moistened with solution of lead acetate, p. 110.

The calcium sulphate is reduced to sulphide by ignition with carbon, and the calcium sulphide when treated with hydrogen chloride is decomposed, calcium chloride being formed and hydrogen sulphide being given off[1].

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[1] $CaSO_4 + C_2 = CaS + 2CO_2$.
$CaS + 2HCl = CaCl_2 + H_2S$.

SECTION V.

EXAMINATION OF THE PROPERTIES OF THE PRINCIPAL RADICLES AND THEIR SALTS.

Introductory Remarks.

This Section will be devoted to a systematic examination of the properties of the simpler forms of matter, chiefly in order to acquire methods for recognising their presence in substances of unknown composition.

The examination of a substance for the purpose of ascertaining of what it consists is called Analysis (ἀνάλυσις), because we endeavour to break up, or dissect, the substance, and to find out whether each of the parts, into which we are able to divide it, agrees in properties with some one of the substances with which we are already acquainted. As one of the simplest instances of analysis, the following experiment may be taken:—

Place about 1 grm. of the red substance called 'red oxide of mercury' in a test-tube, and heat it strongly over a Bunsen's burner, closing the mouth of the tube loosely with your finger. The substance will become darker in colour, and, as the heat approaches redness, a mirror-like ring will be formed in the cool part of the tube. Continue the heat until the whole of the red substance has disappeared, then light a cedar match, blow it out, and while it is still glowing plunge it into the tube. It will burst into flame. This property of re-kindling a glowing match we have seen to belong to oxygen, and on trying other experiments we should find that the properties of the gas in the tube agree altogether with those of the substance which has been called oxygen. Next, examine the mirror-like deposit in the tube; you will see that it is a cluster of small globules which, on being touched with a glass rod, run together into a globule possessing a bright metallic lustre and considerable weight. These properties, together with others which you would discover on further examination of the deposit, coincide with the properties of the substance called mercury. Moreover, if we weigh (*a*) the red substance before heating it in the tube, (*b*) the gas evolved, and (*c*) the metal deposited, we should find that the weight of the original substance was equal to the sum of the weights of the gas and of the metal obtained from it.

Further, on making other experiments with the gas and the metal, we should find it impossible, with our present means, to break them up into simpler forms of matter.

Our inference, then, is that the red substance is composed of the substance called oxygen and the substance called mercury. In what state these substances exist in the compound we cannot say; all that we know is, that they, and they only, can be obtained from it.

This is an example of an analysis which can be effected by heat alone. In general, however, other methods have to be employed, which call into play the different affinities of the constituents of a substance. It is by no means necessary actually to obtain the simplest forms of matter (the so-called 'elements') from a substance in order to ascertain its composition. It is sufficient to cause the formation of compounds which, from their colour, solubility, &c., and the conditions under which they are formed, may be recognised as substances containing particular elements. For instance, it was observed (p. 112) that when iron sulphide (a compound of iron and sulphur) is acted upon by an acid such as hydrogen sulphate, a gas is evolved which blackens paper moistened with a solution of lead acetate. The formation of this black compound of lead, under the circumstances there given, is accepted as a proof of the presence of sulphur in the substance.

In many cases a group of two or more elements appears to be more easily detached from a substance than the individual elements; the substance, like a crystal, cleaving more easily in some directions than in others. An illustration of this will be found in Sect. 2, Ex. 6. The substance, marble, from which we have considerable difficulty in obtaining carbon oxygen and calcium individually, we found to yield by very simple treatment a gas, the properties of which we examined. Having once ascertained that this gas is composed of carbon and oxygen in certain proportions, we are satisfied, in the examination of other substances, if we can obtain the same gas from them by the action of hydrogen chloride. Substances which yield the gas when thus treated, we place under one head and call them 'carbonates.' Similarly, in examining marble for its other constituent, calcium, we do not attempt to isolate the element; we stop short of this point, and are satisfied with obtaining a compound with well-marked properties, from which it has once for all been proved possible to obtain calcium. Substances from which we can obtain this compound, we class together as 'calcium salts.'

We find, in fact, that certain groups of elements can be transferred from one compound to another, and the analysis of a substance is effected when it has been resolved into groups, the composition of which is known. These closely linked groups, as well as the elements themselves, are called 'radicles.'

In the following Exercises the radicles are, as a rule, arranged in the same order as that which is adopted by Professor Williamson in his 'Chemistry for Students.'

It has not been thought necessary to give, as hitherto, a list of apparatus, &c. required for each experiment. Before beginning each set of experiments, you should read over carefully all the directions given, note down what is required, and see that you have all that is necessary within your reach.

Some difficulty may be found in determining how much of a reagent is to be added to the substance under examination. In most cases some indication of the proper amount is given in the text, but the best rule will be, always to consider for what purpose the reagent is added, and in what way the use of too much or too little is likely to affect the chemical change. It may also be remembered that the error of adding too little of a reagent is, in general, more remediable than the error of adding too much.

In pouring a solution from a bottle into a test-tube, it should be a rule never to allow drops of the liquid to run down the side of the bottle. Apart from the unsightly appearance of a bottle encrusted with crystallised salts, the labels are likely to be obliterated or washed off, and shelves and tables will be damaged. These inconveniences may be entirely avoided by the simple method of pouring which is illustrated in Fig. 46.

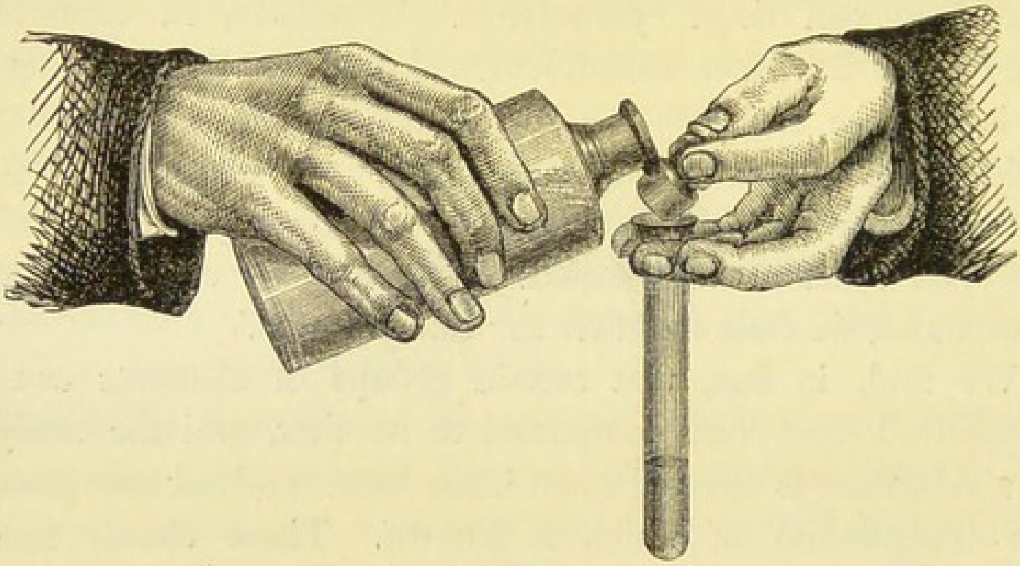

Fig. 46.

The test-tube, into which the liquid is to be poured, is held near the top between the third finger and little finger of the left hand. The stopper, grasped by the forefinger and thumb of the same hand, is loosened and wetted with the solution by slightly inclining the bottle; it is then withdrawn and held against the neck of the bottle, while the test-tube is brought directly under it. The stopper then (like the glass rod in Fig. 30, p. 43) forms a prolongation or lip down which the liquid runs, not a particle finding its way down the side of the bottle. Besides the greater facility with which drops may be measured out, this method has the further advantage, that the stopper is never out of the hands, and hence there is no danger of the solution being contaminated by impurities taken up by the stopper from a dirty table.

When a stopper is found to be fixed immovably in the bottle, try to loosen it by tapping it, first on one side then on the other, with a piece of wood such as the handle of a file, pressing the thumb against the opposite side of the stopper and taking care to direct the blows obliquely upwards, rather than directly across the stopper. If this does not succeed, heat the neck of the bottle by passing it to and fro over the flame of a spirit lamp, turning it constantly round. The neck will expand with the heat before the stopper, and if the latter is now tapped again, it will almost certainly be loosened. There is, of course, a risk of cracking the bottle if it is heated too suddenly, but as the success of the method depends upon the difference in temperature between the neck and the stopper, the heat should be applied quickly, and only for a short time. If this method fails put a drop of oil or glycerine round the stopper, and leave the bottle for some time in a warm room. The oil will work its way between the neck and the stopper, and the latter may generally be loosened by tapping. If the bottle contains potassium hydrate, a drop of hydrogen sulphate may be substituted for the oil, and will remove the alkaline cement.

NITRATES.

Preparation of Hydrogen Nitrate.

Set up an apparatus similar to that which was used for the distillation of water (Fig. 31, p 51); taking, however, a stoppered retort in place of the plain retort. Measure 20 c.c. of water into a beaker, and add to it by degrees 20 c.c. of strong hydrogen sulphate[1]. While the mixture is cooling, weigh out 30 grms. of potassium nitrate, powder the salt roughly in a mortar, transfer it to the retort (Fig. 37, p. 79), and replace the latter on the sand-bath. Pour the diluted acid through the tubulure by means of a funnel, taking particular care that none

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[1] For the precautions necessary in diluting the acid, see p. 86, note.

of the liquid passes down the neck of the retort; then replace the glass stopper and proceed to heat the mixture. Hydrogen nitrate will soon begin to distil over, not in the most concentrated form (since the hydrogen sulphate was slightly diluted), but sufficiently strong for experiments[1].

Collect the first 2 or 3 c.c. of the liquid which distils over in a large test-tube, then substitute for it the Florence flask receiver, and regulate the heat so as to keep the liquid in the retort gently boiling. It will be advisable to provide some means for cooling the receiver as well as the neck of the retort, to ensure the efficient condensation of the corrosive vapours of the acid. For this purpose lay over the flask as it rests in the mortar one or two folds of blotting-paper, and pour over it from time to time some cold water.

While the distillation is going on, you may examine the purity of the acid which came over first, and was collected in the tube. Add to it about 10 c.c. of water, and pour one half of the solution into another test-tube.

(*a*) Test the first half of the solution with a drop of solution of barium chloride; shake the mixture and hold it up to the light. If a turbidity is perceptible, hydrogen sulphate is present as an impurity.

(*b*) To the other half of the solution add a drop of solution of silver nitrate. If a precipitate is produced, chlorine is present as an impurity, derived from the potassium chloride which the nitre of commerce usually contains.

When crystals make their appearance in the liquid and on the sides of the retort, put out the lamp, and, as soon as the apparatus has cooled a little, withdraw the receiver and pour its contents into a clean stoppered bottle. The residue in the retort consists of potassium and hydrogen sulphate, and may when cool be washed out with a little warm water. The retort itself, after being thoroughly rinsed with distilled water, should be set aside to dry, supported with the tubulure downwards in a Bunsen's holder or otherwise.

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[1] $KNO_3 + H_2SO_4 = HNO_3 + KHSO_4$.

Test two small portions of the acid which you have obtained with solution of barium chloride and with solution of silver nitrate respectively. If the distillation has been carefully conducted, traces only (at most) of sulphate and chloride should be found to be present[1]. The liquid will perhaps be slightly yellow, owing to the presence of traces of lower nitrogen oxides (formed by the decomposition of the hydrogen nitrate which takes place if the temperature is somewhat too high), but this impurity is unimportant.

The following experiments may now be tried with the acid which you have obtained:—

1. Pour a little of the liquid into a basin, place in it some of the feather of a quill pen, and concentrate by evaporation. The feather will in a short time be stained yellow.

2. Pour some dilute solution of litmus into a test-tube and add one drop of the hydrogen nitrate. The blue colour will be at once changed to red, showing that hydrogen nitrate belongs to the class of substances which have an 'acid reaction.'

3. Pour about 5 c.c. of the liquid into a porcelain dish, and add about 3 c.c. of solution of ammonia. Stir the mixture with a glass rod, and place the drop of it which adheres to the rod upon a strip of blue litmus-paper laid on a clean glass plate. The liquid will redden the litmus-paper decidedly. Continue to add ammonia cautiously drop by drop, stirring the mixture, and placing a fresh drop from time to time on an untouched part of the strip of litmus-paper. You will find that a time will arrive when the liquid no longer alters the colour of the blue litmus-paper. When this is the case, place a drop of it on a piece of reddened litmus-paper, and observe whether the red colour is changed to blue. If so, pour a few drops of the hydrogen nitrate into a test-tube, dilute it with about ten times its volume of water, and add it drop by drop to the solution in the dish, constantly trying the action of the liquid on both

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[1] In order to obtain a perfectly pure acid, it will generally be necessary to re-distil it after addition of a crystal or two of silver nitrate and of potassium dichromate.

blue and reddened litmus-paper. You will eventually obtain a solution which does not alter the colour of either test-paper. Such a solution is said to be 'neutral' to test-paper (p. 100). When this point is reached, evaporate the solution on the sand-bath at a gentle heat, placing near it (for the sake of comparison) two watch-glasses, the one containing a little hydrogen nitrate, the other a little solution of ammonia. You will find that a white residue will be left in the porcelain dish, while there will be scarcely a trace of a residue left in the watch-glasses. By bringing hydrogen nitrate in contact with ammonia in a certain definite proportion, you have obtained a substance which differs from them in at least two respects, (*a*) in having no action on vegetable colours, (*b*) in being a crystallisable solid at ordinary temperatures. This 'salt' is, in fact, identical with the substance called ammonium nitrate, from which you obtained nitrogen protoxide (Sect. **2**, Ex. 4).

[For the following experiments crystallised potassium nitrate and a solution of the same salt (made by dissolving 2 grms. in 20 c.c. of water) should be used[1].]

4. Pour a little dilute hydrogen sulphate into a test-tube and add just sufficient solution of indigo sulphate to colour the liquid distinctly blue. Then add a drop of the solution of potassium nitrate and heat the mixture to boiling. The blue colour of the indigo will disappear entirely.

5. Place a small crystal of potassium nitrate on a piece of charcoal and direct the blowpipe-flame upon it. The salt fuses, and when the charcoal becomes red-hot, a vivid deflagration will take place owing to the decomposition of the nitrate and oxidation of the carbon; the oxygen uniting with the carbon and potassium to form potassium carbonate, while nitrogen gas and carbon dioxide are liberated.

6. Pour into a test-tube a few drops of the solution of potassium nitrate; add an equal volume of strong hydrogen

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[1] In cases where, as here, a solution of a salt is prepared for the purpose of being tested, the strength of the solution is such that a litre of it would contain (approximately) a molecule of the substance, expressed in grammes.

sulphate, and drop in a bit of copper wire. Nitrogen dioxide will be given off, and may be recognised by the red fumes which it forms with the air in the tube. These may be most distinctly seen by placing the tube on a piece of white paper and looking down into it.

7. Place a little solution of potassium nitrate in a tube, add an equal volume of hydrogen sulphate as in the last experiment, and allow the mixture to become quite cold. Then, holding the tube in a slanting position, pour as slowly as possible down the side a solution of iron protosulphate from another tube, so that the lighter solution may not mix with, but float upon, the heavier. A dark brown ring will be formed at the line of junction of the two liquids, which will, when the tube is heated, disappear with effervescence due to the escape of nitrogen dioxide.

8. Place some fragments of potassium hydrate in a test-tube, add a few drops of water, then put in a piece of granulated zinc, and heat the mixture, supporting the tube in a Bunsen's holder in a slanting position and covering the mouth loosely with a card. Hydrogen gas will be evolved when the solution is sufficiently concentrated, and may be recognised by holding a lighted match to the mouth of the tube, when the gas will take fire. Now drop in a small crystal of potassium nitrate, keeping the mixture heated. Ammonia will be formed in abundance, and may be recognised by its odour and its action on a piece of reddened litmus-paper held at the mouth of the tube.

AMMONIA.

(For the general properties of the gas, see p. 95.)

1. Form a piece of platinum wire, about 15 cm. long, into a close spiral[1], by coiling it round a glass rod or pencil,

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[1] The platinum should not be much thinner than No. 22 brass wire-gauge, otherwise it does not retain sufficient heat to effect the combination of the gases.

and attach it to a strip of card sufficiently wide to fit rather tightly into the neck of a flask about 400 c.c. in capacity. The coil should hang down freely in the centre of the flask, the card forming a diaphragm or partition in the neck. Withdraw the coil from the flask, pour a few drops of a strong solution of ammonia into the latter, and shake it so as to diffuse the ammonia gas through the air contained in the flask. Heat the platinum spiral to redness in a spirit or gas lamp, and while it is still red-hot, plunge it into the flask. The coil will continue to glow, and white vapours of ammonium nitrite will be almost immediately formed. The heated platinum wire has determined the union of the constituents of ammonia with the oxygen of the air. If, when the coil of wire has ceased to glow, the flask is gently heated for a few seconds over a lamp, more ammonia gas will be evolved from the liquid, and the platinum will again become red-hot. After the lapse of a minute or two, take out the coil of wire, add 2 or 3 c.c. of water, and shake it up in the flask; then pour the liquid (which will be slightly yellow, owing to the presence of nitrogen tetroxide) into a test-tube, add enough solution of hydrogen sulphate to render the solution strongly acid (when the smell of nitrogen oxides will be perceived), then add one drop of solution of indigo sulphate, and heat the mixture. The blue colour of the indigo will disappear, proving, under the conditions of the experiment, that a nitrite or nitrate is present.

In the last experiment (p. 147) we found that ammonia might be obtained from a substance in which nitrogen is associated with oxygen: we have here found it possible to effect the converse change, and obtain a nitrogen oxide from ammonia.

2. Place a little horse-hair or a few feathers in a large test-tube, and heat the tube rather strongly. The substance will soon blacken and be decomposed, giving off among other volatile products ammonium carbonate and ammonia. The presence of the latter may be recognised by its odour, by its action on a piece of reddened litmus-paper held near the mouth

of the tube, and by the white fumes formed when a glass rod dipped in strong hydrogen chloride is held in the issuing vapours. Horse-hair and other animal matters are compounds of nitrogen, hydrogen, carbon, and oxygen; and when they are decomposed a portion of the nitrogen and hydrogen unite to form ammonia.

3. Place a small piece of sodium, about as large as a pea, carefully freed from adhering naphtha by blotting-paper, in a dry test-tube, and add to it twice its volume of mercury. If the tube is very gently heated, the two metals will combine with a slight explosion, forming a semi-fluid alloy or amalgam. While this substance is cooling, make a strong solution of ammonium chloride by dissolving 7 or 8 grms. of the powdered salt in 20 c.c. of water, noticing the cold produced by the solution of the salt. Pour this solution into the test-tube containing the amalgam: the latter will immediately begin to increase in bulk, swelling into a grey porous mass which will probably half fill the tube. Shake it out upon a dish; notice its pasty consistence, and also that when no longer immersed in the solution of ammonium chloride it begins to decompose, shrinking in bulk, giving off ammonia and hydrogen, and finally leaving nothing but a globule of mercury. There is every reason to believe that this substance is a true amalgam or alloy of mercury with the metal ammonium, a body containing fourteen parts by weight of nitrogen united with four parts of hydrogen, while ammonia consists of the same weight of nitrogen united with only three parts of hydrogen. But this metal from its instability has never been isolated, and is only known in combination with mercury and acid radicals, e. g. chlorine.

4. Place a small fragment of ammonium chloride (common sal ammoniac) in a dry test-tube, and heat it. Notice that it volatilises entirely, forming a white sublimate in the upper part of the tube. Notice also that it passes at once from the solid to the gaseous state, without becoming a liquid; bearing a resemblance to arsenic trioxide in this respect.

5. Dissolve about 1 grm. of ammonium chloride in 20 c.c.

of water, and examine separate portions of the solution as follows:—

(*a*) To the first portion add a drop or two of solution of platinum perchloride, and shake the mixture. A yellow crystalline precipitate will be formed, consisting of a double salt, ammonium and platinum chloride.

(*b*) Place a little powdered quicklime or slacked lime in a small beaker, add just enough water to cover it, and then one or two drops of solution of ammonium chloride. Cover the mouth of the beaker with a watch-glass on the convex (and lowermost) side of which has been spread a moist slip of reddened litmus-paper and a similar slip of turmeric-paper. Heat the beaker very gently on the sand-bath. In a short time ammonia will be evolved by the action of the calcium hydrate on the ammonium salt, and will restore the blue colour to the litmus-paper and redden the turmeric-paper. When this is seen to be the case, remove the watch-glass, notice the odour of ammonia in the beaker, and also test for it by dipping into the beaker (without touching the sides) a glass rod moistened with strong hydrogen chloride or, better (since hydrogen chloride itself fumes perceptibly), hydrogen acetate.

CARBON.

1. Pour a few drops of turpentine into a small evaporating dish and place it in the centre of an ordinary china plate. Take out the stopper of a deflagrating jar and place it sideways on the neck, so as partially to close the opening. Apply a light to the turpentine, and place over it the jar, thus loosely stopped, and tilted slightly on one side so as to admit a very limited supply of air for the combustion of the turpentine. Under these circumstances the latter will not be completely burnt, and a quantity of finely divided carbon (the ordinary lamp-black) will be deposited on the sides of the jar. Oxygen has less affinity for carbon than for hydrogen, and hence, while

the whole of the latter is burnt, forming water, a part only of the former is converted into carbon dioxide, the rest appearing in the elemental condition. Compare Experiment 4, p. 105, which illustrates a similar difference in the affinity of chlorine for carbon and for hydrogen, respectively.

2. Put some pieces of leather or quills into a common clay crucible; place the cover on the crucible and heat it to redness for a few minutes in an ordinary fire. When it is cold, take out the porous mass of charcoal and grind it to powder in a mortar; then put some of it into a test-tube and add a little dilute solution of litmus. Shake up the mixture thoroughly, and filter it into a clean test-tube, passing it several times through the same filter. The filtrate will be nearly, or quite, colourless; the carbon having taken up the colouring matter in the solution[1].

Animal charcoal (or bone-black), which has been recently ignited in a covered porcelain crucible, may be used in this and the following experiment, and will be found to act even more powerfully than the charcoal prepared as above directed.

3. Put the remainder of the animal charcoal prepared in the last experiment into another test-tube, pour on it a few drops of solution of hydrogen sulphide, and shake the mixture thoroughly for half a minute. You will find that the liquid has now lost all the offensive smell of the gas, which has been absorbed by the charcoal. The value of charcoal as a disinfectant is due to the property of absorbing gases which it possesses.

4. The presence of graphite in cast-iron will be shown in a future experiment, under the head of 'Iron.'

5. Fill a large gas bottle with oxygen gas[2]. Select a slender splinter of charcoal, about 3 cm. in length; lay it across a deflagrating spoon so that the ends may project on either side;

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[1] The withdrawal of the colour appears to be more complete with reddened than with blue litmus.

[2] Sufficient gas for this experiment may be made in a tube-apparatus such as that which is described at p. 165 for making hydrogen iodide, a mixture of 10 grms. of potassium chlorate and 3 grms. of manganese dioxide being placed in the test-tube.

heat the ends until they glow, and immerse the spoon in the jar of gas. When the brilliancy of the combustion has died away, withdraw the charcoal, pour into the gas a little lime water (from a test-tube, p. 91), and shake the bottle. The lime water will immediately become turbid, owing to the presence of carbon dioxide, the oxygen having united with the carbon.

CARBONATES.

(For the general properties of carbon dioxide, see p. 88.)

1. Add a few drops of dilute hydrogen sulphate to some sodium carbonate in another test-tube. Effervescence will take place owing to the liberation of carbon dioxide. Close the mouth of the tube with the finger, to confine the gas; dip the end of a glass rod in lime water, remove the finger from the tube, and steadily lower the glass rod, with a drop of lime water adhering to it, into the tube, without touching the sides. The lime water will become turbid, as may be seen by withdrawing the rod carefully, and holding it against any dark background. Close the mouth of the test-tube with a cork to which is adapted a tube bent twice at right angles, like a siphon. The gas evolved may then be led into another test-tube containing lime water, which will be rendered turbid.

2. Adapt a cork to a large test-tube; bore two holes into it and fit into them elbow tubes similar to those used for the wash-bottle, Fig. 41, p. 109. The long branch of one elbow tube should reach nearly to the bottom of the test-tube. Fill the test-tube about half full of lime water; apply your mouth to the outer extremity of the short elbow tube, and suck air through the lime water for a few seconds. No turbidity will be produced, the quantity of carbon dioxide in the air being very small. Now, having taken a deep breath, apply your mouth to the other elbow tube (the long branch of which passes into the liquid), and blow air from the lungs through the lime water. The latter will immediately become turbid, showing

that much carbon dioxide is present in air which is expired from the lungs.

ACETATES.

1. Place a little saw-dust or a few splinters of wood in a test-tube, and heat the tube, supported in a Bunsen's holder, over a gas lamp. The wood will blacken or 'char,' and give off gases which will take fire if a lighted match is held at the mouth of the tube, while drops of a liquid will condense in the cool part of the tube. Bring a slip of blue litmus-paper in contact with the liquid: it will be found to redden the paper strongly; it consists, in fact, mainly of hydrogen acetate.

2. Place a small quantity of lead acetate in a test-tube, pour on it a few drops of strong hydrogen sulphate, and heat it gently. The substance will not blacken (as a tartrate does), but pungent smelling vapours of hydrogen acetate will be given off, which will redden a piece of litmus-paper held at the mouth of the tube.

If time permits, a little hydrogen acetate may be prepared from lead acetate in an apparatus similar to that which was used for the distillation of hydrogen nitrate (p. 144), 20 grms. of lead acetate being substituted for the potassium nitrate.

3. Repeat the above experiment, adding a few drops of of alcohol (not methylated) before the addition of the hydrogen sulphate. Vapours of ethyl acetate (acetic ether) will be given off, which possess a characteristic fragrant smell, not unlike that of fresh apples.

4. Place a little hydrogen acetate in a watch-glass and add solution of ammonia drop by drop until the liquid is just neutral to test-paper. If an excess of ammonia has been unintentionally added, it may be driven off by evaporating the solution for a few minutes on the sand-bath. Now place the watch-glass on a piece of white paper, and add one drop of solution of iron perchloride. No precipitate will be formed, but the solution will become dark red, and when boiled in a test-tube it will give a brown-red precipitate of basic iron acetate.

TARTRATES.

1. Place a small quantity of sodium and hydrogen tartrate in a test-tube, add a little strong hydrogen sulphate, and heat gently. The mixture will blacken and give off carbon dioxide and other gases with effervescence.

2. Heat a little of the same salt in a test-tube nearly to redness. It will become charred, and give off vapours possessing a characteristic smell, like burnt sugar. When the residue is cold, pour on it a few drops of dilute hydrogen chloride. Carbon dioxide will be evolved, as may be proved by holding within the tube a glass rod dipped in lime water (p. 152).

3. Dissolve 4 grms. of sodium and hydrogen tartrate in 20 c.c. of water, and test separate portions of the solution as follows:—

(*a*) To the first portion add one or two drops of solution of potassium hydrate, and shake the mixture. A crystalline precipitate of potassium tartrate will be gradually formed.

(*b*) To another portion add at least three or four times its volume of lime water. A white precipitate of calcium tartrate will be produced, which will dissolve on addition of a few drops of solution of ammonium chloride.

(*c*) Neutralise another portion carefully with solution of potassium hydrate, add a drop of solution of silver nitrate, and heat the mixture to boiling. The liquid will turn black owing to the reduction of the silver salt.

OXALATES.

1. Weigh out 8 grms. of loaf-sugar, reduce it to powder in a mortar, place it in a Florence flask, and pour upon it 60 c.c. of strong hydrogen nitrate; then heat the flask cautiously on a sand-bath (in a draught cupboard, if possible). Carbon dioxide and nitrogen oxides will be copiously evolved,

and hydrogen oxalate will be formed in the liquid. When the action has moderated, pour the solution into a porcelain dish, and evaporate it down until a drop taken out with a glass rod and placed on a watch-glass deposits crystals on cooling. The liquid may then be left to cool and crystallise. Transparent prisms of hydrogen oxalate will thus be obtained, which, after the liquid has been poured off, should be placed in a funnel, the neck of which has been obstructed by a bit of glass rod, and left to drain, and finally washed with a little water and dried by pressing them between folds of blotting-paper.

2. Place a few crystals of hydrogen oxalate in a test-tube and heat them slowly. The salt will melt and the greater part will sublime unaltered if the heat is carefully applied.

3. Pour some strong hydrogen sulphate on a few crystals of hydrogen oxalate in another test-tube, and heat the mixture. Carbon dioxide and carbon protoxide will be given off with effervescence (compare the method of making carbon protoxide, given in p. 92). The latter gas will burn with its characteristic blue flame if a lighted match be held to the mouth of the tube; the presence of the former gas may be shown by holding a glass rod dipped in lime water within the tube (p. 152).

4. Place a little manganese dioxide in a test-tube, add enough water to cover it, and then a few drops of strong hydrogen sulphate. If there should be any effervescence (manganese dioxide sometimes contains carbonates) wait until this has ceased, and then add a few drops of a solution of ammonium oxalate. Carbon dioxide will now be given off, and may be tested for with lime water, as above.

5. Fill a test-tube about one-third full of solution of ammonium oxalate, and add a few drops of solution of calcium chloride. A white precipitate of calcium oxalate will be produced. Now add more calcium chloride until no more precipitate is formed, and pour off a few drops of the mixture in which the precipitate is suspended into two other test-tubes. Add to one portion a little hydrogen acetate; the precipitate will remain undissolved. To the other portion add some dilute

hydrogen chloride; the precipitate will dissolve without effervescence. The rest of the liquid containing the calcium oxalate should be warmed and filtered through Swedish filter-paper; the precipitate should be washed on the filter by pouring a gentle stream of water on it from the washing bottle, and then left to drain for a short time. The damp filter should be cautiously taken out of the funnel, placed on a few folds of blotting-paper, and carefully spread open; it may then be left to dry in a warm place. When dry the calcium oxalate may be easily detached from the filter and placed in a test-tube. The white mass will, on being heated over a lamp, become grey at a temperature just below redness, but on continuing the heat it should become nearly white again. If a little dilute hydrogen chloride is poured on the residue an effervescence will take place, carbon dioxide being given off. The calcium oxalate has been decomposed by the heat into calcium carbonate and carbon protoxide[1].

CYANOGEN AND CYANIDES.

[This gas and its compounds are very poisonous, and great care is necessary in experimenting upon them. The experiments should be performed in a well-ventilated room, before an open window, or in the open air, and only small quantities of the substances should be used.]

1. Dissolve 3 grms. of silver nitrate and 1 grm. of potassium cyanide in separate portions of water, and add the latter solution to the former. A white precipitate of silver cyanide will be produced. Pour off small portions of the liquid in which the precipitate is suspended into two test-tubes; add to one a few drops of ammonia, which will readily dissolve the precipitate; allow the precipitate in the other tube to subside, pour off the liquid, retaining the precipitate in the test-tube, and boil the latter with some strong hydrogen nitrate. The silver cyanide will be slowly decomposed and dissolved by the acid.

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[1] $CaC_2O_4 = CaCO_3 + CO$.

The main portion of the silver cyanide is next to be filtered, washed, and dried (in the same manner as the calcium oxalate, p. 156). When dry place some of it in a small test-tube and apply heat. It will be decomposed, and cyanogen gas will be given off, which may be recognised by its smell, and will burn with a fine purple flame if a lighted match be held to the mouth of the tube. The black residue in the tube contains a part of the cyanogen in combination with the silver, as silver paracyanide.

2. Pour a little solution of potassium cyanide into a test-tube and add a few drops of solution of iron protosulphate, and then about the same quantity of solution of potassium hydrate, which will produce a bluish green precipitate. After warming the mixture add excess of dilute hydrogen chloride, which will leave a deep blue precipitate of iron ferrocyanide or Prussian blue.

3. Add one or two drops of yellow ammonium sulphide and a little ammonia to a very dilute solution of potassium cyanide in a small porcelain dish. Heat the mixture until it becomes colourless, then filter from the separated sulphur, and add to the clear solution a drop of dilute hydrogen chloride and a drop of iron perchloride. A deep red solution will be obtained, proving that a sulphocyanide has been formed by the action of ammonium sulphide on potassium cyanide, which forms a deep red salt with iron.

4. Heat a little solid potassium cyanide in a porcelain crucible over a Bunsen's burner or a gas blowpipe. The salt will fuse at a low red-heat; when it is in calm fusion, drop into it by degrees some dry lead protoxide. The potassium cyanide combines with the oxygen of the lead oxide, forming potassium cyanate, while the lead separates in the metallic form, and will be found under the fused layer of salt when the liquid contents of the crucible are poured out on a piece of iron or a plate. When cold, the mass may be warmed with water in a beaker, when the globule of lead will be left undissolved. If dilute hydrogen chloride be added to the solution of potassium cyanate thus obtained, the salt is decomposed; carbon dioxide

escapes with effervescence, while ammonium chloride is formed in the solution[1]. The presence of ammonia may be shown by gently warming a little of the solution with some quicklime in a beaker, when free ammonia will be given off and may be recognised by its smell and its action on reddened litmus-paper (see p. 150).

CHLORIDES.

(For the general properties of hydrogen chloride, see p. 99.)

[A solution of sodium chloride containing 2 grms. of the salt in 30 c.c. of water may be used.]

1. Mix a little strong hydrogen chloride with one-third its volume of strong hydrogen nitrate in a test-tube, and warm gently. The fluid will become yellow, and will give off an orange gas, smelling like a chlorine oxide: if scraps of platinum foil, or bits of gold-leaf, are thrown into the mixture they will be readily dissolved. A nitryl chloride (the orange gas) and free chlorine are formed in the reaction, the latter of which at the moment of its formation acts on metals with more than its usual energy.

2. Add a few drops of solution of silver nitrate to a little solution of sodium chloride in a test-tube. A white precipitate of silver chloride will be formed, which, on addition of a little hydrogen nitrate and after shaking the mixture, will readily separate in flocks, leaving the liquid almost clear. Pour off the solution, shake up the precipitate with some fresh distilled water, and again pour off the fluid, after allowing the precipitate to subside. Silver chloride is one of the easiest substances to wash thus, by decantation. Now pour a little ammonia upon the precipitate; it will readily dissolve, but will again be thrown down on addition of hydrogen nitrate to the ammoniacal solution.

3. Pour a few drops of solution of sodium chloride upon a little manganese dioxide in a rather wide test-tube, and add enough hydrogen sulphate to cover the mixture. On warming

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[1] $KCNO + 2HCl + H_2O = KCl + H_4NCl + CO_2$.

it gently chlorine will be evolved, which may be recognised by its smell, its colour (best seen by looking across the tube placed against a sheet of white paper), and its action on a slip of moist litmus-paper held in the tube, which will be first reddened, then bleached.

CHLORINE OXIDES AND HYDRATES.

1. Set up an apparatus for generating chlorine, as described in p. 113, and pass a stream of the gas through a moderately strong solution of potassium hydrate in a beaker or large test-tube, keeping the solution cool by placing it in a dish of water. When the gas has passed through the solution for about two minutes, pour off about one-third of the liquid, and reserve it for experiments. Saturate the remainder of the solution with chlorine, no longer cooling it, but on the contrary keeping it nearly boiling by a lamp. While the saturation is proceeding, you may examine the properties of that portion of the solution which you have reserved, which will contain potassium hypochlorite and chloride. It will be found to have a peculiar sweet chlorous smell, very different from that of chlorine itself.

(*a*) Pour a little of the solution into a test-tube and add a drop of solution of litmus, and warm gently. The colour of the litmus will be discharged.

(*b*) Acidify a weak solution of potassium iodide with dilute hydrogen sulphate (which should produce no colouration if the iodide is pure), and add one drop of the potassium hypochlorite. Iodine will be liberated, the solution becoming yellow, and giving a deep blue colour if a fresh solution of starch or arrowroot is added (compare p. 164).

(*c*) Add some solution of lead acetate to another portion of potassium hypochlorite. A white precipitate of lead hypochlorite will be first formed; which will, however, soon turn brown, especially if warmed, owing to the formation of lead dioxide.

2. Place a little ordinary bleaching powder at the bottom

of a large test-tube, and pour on it some strong hydrogen chloride. A violent action will take place, and a greenish yellow gas will fill the tube, which may be proved to be chlorine by its odour and bleaching action on litmus-paper.

3. Shake up a little bleaching powder with water, filter it, and try with the filtrate the same experiments as you have tried with potassium hypochlorite.

4. When the potash solution is thoroughly saturated with chlorine[1], filter it, pour it into a porcelain dish and evaporate it to one-half its bulk. Flat rhombic plates of potassium chlorate will be formed as the solution cools. These may be drained from the liquid, washed with a little water, and left to dry in a funnel. The liquid drained from the crystals will contain much potassium chloride, as may be proved by testing it with silver nitrate.

5. Pour about 2 c.c. of strong hydrogen sulphate into a test-tube, drop into it a crystal of potassium chlorate, and place it in a beaker containing a little hot water. The liquid will become deep orange, and a greenish yellow gas (chlorine tetroxide) will fill the tube. Bend a piece of copper wire, about 30 cm. long, at right angles, heat one end of it in a spirit lamp, and plunge it while hot into the gas in the test-tube. The gas will decompose with slight explosion, and its greenish yellow colour will almost entirely disappear, owing to the formation of chlorine, the colour of which is less intense, and oxygen.

6. Nearly fill a large wine-glass, or conical measure, with water, and drop into it a few crystals of potassium chlorate, which will sink to the bottom without much loss, as the salt is not very soluble in water. Drop upon the crystals one or two bits of phosphorus about as large as a pea, and support a tube funnel over the glass in such a position that the extremity of the tube may just touch the crystals at the bottom of the glass. Now pour down the funnel a few drops of strong hydrogen sulphate; chlorine tetroxide will be evolved when the

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[1] The cessation of an effervescence, which occurs towards the end of the reaction, and is due to the decomposition of the carbonate always present in ordinary potassium hydrate, will serve to mark this point.

acid comes in contact with the potassium chlorate, and the phosphorus will take fire, as in chlorine gas, and burn underneath the water.

This experiment is perfectly safe, if performed with small quantities of potassium chlorate and phosphorus. Larger quantities than are above indicated should on no account be used.

7. Dissolve 3 grms. of pure potassium chlorate in 25 c.c. of water, and try the following experiments with separate portions of the solution in test-tubes :—

(*a*) To one add a drop of solution of silver nitrate. No precipitate will be produced as in the case of the chloride, since silver chlorate (as indeed every other chlorate) is soluble in water[1].

(*b*) To another portion add a drop of solution of indigo sulphate, and boil. The colour of the indigo is discharged.

(*c*) To a third portion add solution of indigo as above, and then a few drops of solution of sulphur dioxide (or, what is equivalent, a solution of sodium sulphite acidified with hydrogen sulphate), which has been previously coloured with the same solution of indigo. The colour of the indigo disappears at once, even in the cold, owing to the reduction of the hydrogen chlorate, with formation of lower and more unstable chlorine compounds. This reaction serves to distinguish a chlorate from a nitrate, since the latter will not under the same conditions bleach indigo at once. The experiment should be repeated, using a solution of potassium nitrate instead of potassium chlorate, to prove this fact.

8. Place a few crystals of potassium chlorate in a dry test-tube, supported in a slanting position in a Bunsen's holder, and apply heat. The salt will fuse into a liquid, and at a temperature just above its melting-point will give off a gas, with effervescence, which may be shown to be oxygen by its power of re-kindling a glowing match introduced into the tube.

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[1] Commercial potassium chlorate generally contains a trace of chloride, sufficient to give a turbidity with silver nitrate, but it may be readily purified by dissolving in as little hot water as possible, and re-crystallising.

Continue heating the salt for a minute or two, then remove the lamp, and when the fused mass in the tube is cool, warm it with a little water, and test the solution with a drop of silver nitrate. A dense white precipitate will now be produced, insoluble in hydrogen nitrate, but soluble in ammonia. The chlorate, by the loss of oxygen, has been reduced to chloride.

BROMINE.

Mix together in a mortar 1 grm. of manganese dioxide with 0.5 grm. of potassium bromide. Transfer the mixture to a clean dry Florence flask and pour on it, through a tube funnel, sufficient strong hydrogen sulphate to cover it. On applying a gentle heat by some hot sand, the flask will be filled with deep red vapours of bromine, which will condense in the neck of the flask as a dark liquid. It will not be necessary to collect any of the liquid, which is extremely corrosive and poisonous, while its vapour is even more irritating than chlorine itself. The flask should be taken into the open air as soon as the experiment is performed.

BROMIDES.

[A solution of potassium bromide, containing 3 grms. of the salt dissolved in 25 c.c. of water may be used.]

1. Add a few drops of chlorine water to some solution of potassium bromide in a test-tube. The bromine will be displaced by the chlorine from its combination with potassium, and will dissolve in the excess of potassium bromide, colouring the solution yellow.

Now add a small quantity of carbon disulphide or chloroform, shake the mixture, and allow it to stand for a few moments in order that the scattered particles of the carbon disulphide may collect at the bottom of the tube in one globule, which will be found to have acquired an orange tint, while the fluid above it will be colourless. The bromine has been withdrawn entirely from the solution by the carbon disulphide, in which it is very soluble. Pour off the upper stratum of fluid, fill up the test-tube with water, and again pour it off; then add some solution of sodium hydrate, shake it up, and allow it to stand as before. The globule of carbon disulphide will have lost its colour, the bromine having combined with the sodium hydrate, to form sodium bromide and bromate. (Compare the action of chlorine on sodium hydrate, p. 160[1].)

2. Add a drop of solution of silver nitrate to solution of potassium bromide. A yellowish white precipitate of silver bromide will be formed. Divide the liquid in which the precipitate is suspended into two parts; add to the one some hydrogen nitrate; the precipitate will not dissolve. To the other portion add some ammonia; the precipitate will dissolve by degrees, but not so readily as silver chloride.

IODINE.

1. Repeat the experiment on the opposite page which illustrates the formation of bromine, substituting potassium iodide for potassium bromide. Deep violet vapours of iodine will be formed, and will condense in the neck of the flask in rhombic plates, the form of which may be readily made out with a small magnifying glass. Very fine crystals of iodine are often formed, by spontaneous sublimation on the stopper of a bottle containing it.

2. Put a small piece of carefully dried phosphorus into an evaporating dish and place on it a crystal or two of iodine. In a few moments the two bodies will combine, and will inflame from the heat produced by their union.

3. Pour a little of a solution of potassium iodide into a test-tube and add a crystal or two of iodine, which will readily dissolve forming an orange-red liquid. Pour the solution into a test-glass or beaker, and dilute it with about 200 c.c. of water.

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[1] If carbon disulphide is not at hand, ether will serve the purpose as it will form a coloured stratum on the top of the aqueous solution.

Add to it a little freshly made solution of starch, and stir with a glass rod. A deep blue colour will be formed even in so highly dilute a solution of iodine; a reaction which is very delicate and characteristic. Pour a little of the blue solution into a test-tube, and heat it nearly to boiling. The blue colour will disappear, but will again become apparent when the solution cools. Hence in testing for iodine by this method care must be taken that the liquid is cold. The starch solution, moreover, must be freshly made; since after a time it loses the property of forming a blue compound with iodine, probably owing to its conversion into dextrine.

4. Place a few crystals of iodine in a test-tube, pour over them about 5 or 6 c.c. of water, and shake the mixture. You will find that iodine is scarcely soluble in cold water, but enough will dissolve to colour the liquid pale yellow, and to give the deep blue colour when poured into another tube and tested with solution of starch. After pouring off the solution from the undissolved iodine, add to the latter 2 or 3 c.c. of alcohol, and observe that the substance readily dissolves, forming a dark red solution. Add to this four or five times its volume of water; the iodine will be almost wholly precipitated as a black powder. We have seen in the preceding experiment that it is not thus precipitated on dilution from the solution in potassium iodide.

IODIDES.

5. Place a crystal of potassium iodide in a test-tube, pour on it some strong hydrogen sulphate, and heat gently. A gas will be given off which forms white fumes in the air, like hydrogen chloride, but it will be mixed with violet vapours of iodine, resulting from the decomposition of a part of the hydrogen iodide by the hydrogen sulphate, sulphur dioxide, water, and iodine being formed. Hence it is not possible to obtain hydrogen iodide, as we obtained hydrogen chloride, by decomposing an iodide by hydrogen sulphate. To obtain the gas we must have recourse to the decomposition of

phosphorus iodide by water. We have seen, from Experiment 3, that the union of phosphorus and iodine takes place readily, but with a violence which is not easy to control. Nevertheless hydrogen iodide may be made without much danger by this method, if care be taken to add the phosphorus little by little to a large excess of iodine. The following modification of the process is much to be preferred, when the allotropic form of phosphorus, called red or amorphous phosphorus, can be obtained, since the action of red phosphorus on iodine is much less violent than that of ordinary phosphorus. (For the properties and preparation of red phosphorus, see p. 176.)

Take a large test-tube, about 2 cm. in diameter and 15 or 16 cm. in length; adapt to it a cork fitted with an elbow tube and right-angled delivery tube connected by a cork joint, such as was used in the preparation of carbon dioxide, p. 89, and support it in an inclined position in a Bunsen's holder thus, Fig. 47.

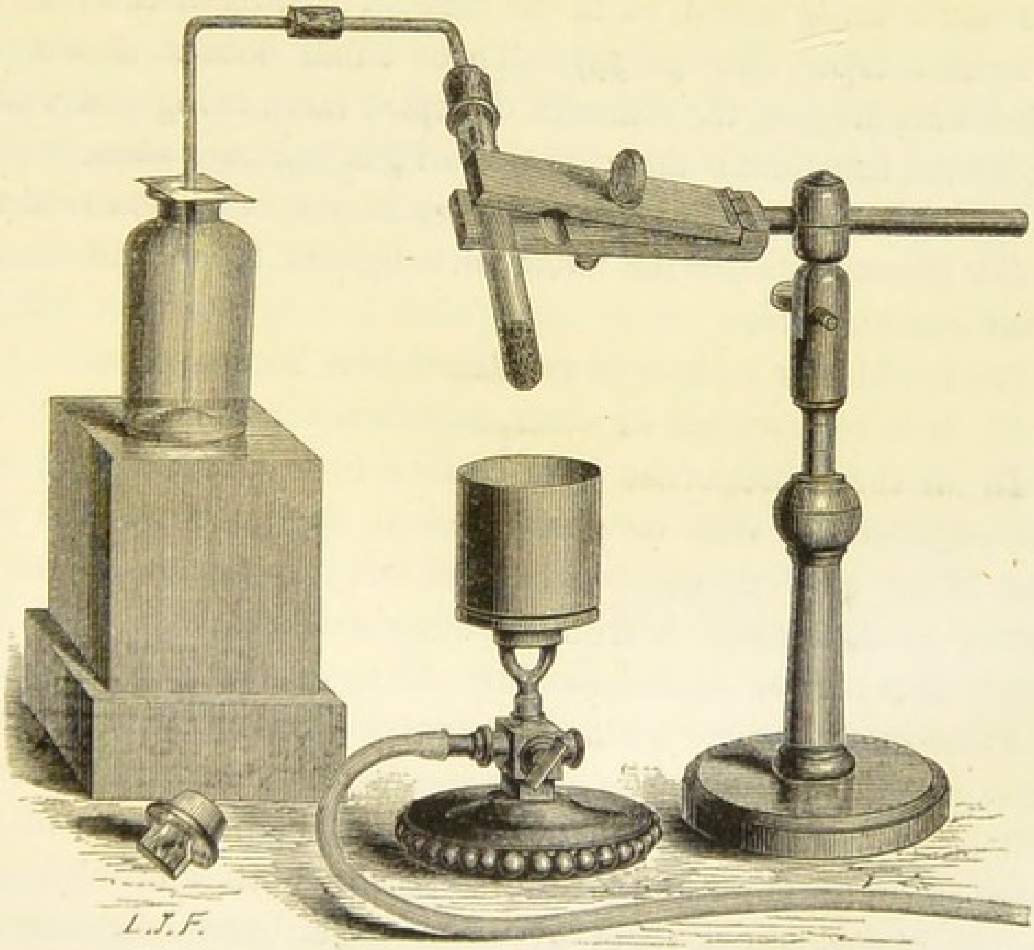

Fig. 47.

Weigh out 0.5 grm. of red phosphorus, grind it (if necessary) with care to a coarse powder in a mortar, covering it with water while crushing it with the pestle, in case any small particles of ordinary phosphorus may be present which might inflame by the friction and set the whole mass on fire; then pour off the water and transfer the moist powder to the test-tube. Pour upon it 1 c.c. of water and then add little by little 6 grms. of iodine, warming the mixture at first until the reaction begins, and afterwards cooling it by dipping the test-tube into water. Finally, replace the cork with delivery tube, and re-adjust the apparatus in the holder, placing under the end of the delivery tube a small wide-mouthed bottle, covered with a card. Now heat the mixture in the test-tube very carefully with a spirit lamp, until the gas is evolved[1]. The mixture froths up a little at first, but there is no explosive action, and the gas will soon pass over into the bottle. From its great density it may most readily be collected by downward displacement, its position in the bottle being tested, as in the case of hydrogen chloride, by a lighted taper (see p. 99). Three small bottles should be filled with the gas, the flame of the spirit lamp being just waved under the test-tube if the evolution of gas becomes slow.

The following properties of the gas may now be observed:—

(*a*) It extinguishes the flame of a lighted taper, and is not itself combustible.

(*b*) It reddens a piece of moistened blue litmus-paper.

(*c*) It is very soluble in water, and fumes in moist air.

In all these properties it resembles hydrogen chloride, and the experiments may be performed in the same way as with the latter gas (p. 99). The following property, however, serves to distinguish it from hydrogen chloride.

(*d*) It is readily decomposed by chlorine.

Place in a test-tube a little manganese dioxide and add some strong hydrogen chloride. On heating the mixture chlorine will be evolved and will fill the tube. Bring the mouth of the tube close to the mouth of one of the bottles of hydrogen iodide placed on a sheet of white paper, and take out the

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[1] $P_4 + 6 I_2 + 12 H_2O = 4 H_3PO_3 + 12 HI$.

stopper. The chlorine will gradually pass into the bottle and violet vapours of iodine will be formed from the decomposition of the gas. An excess of chlorine must be avoided, and the experiment will be more striking if the gases are diluted with air, as may be done by allowing them to pour simultaneously into a large empty bottle.

After the experiment, wash out the test-tube containing the chlorine with plenty of water, that the gas may not escape unnecessarily into the room.

6. Pour a few drops of solution of potassium iodide (containing 4 grms. of the salt dissolved in 25 c.c. of water), and add two or three drops of solution of silver nitrate. A yellow precipitate of silver iodide will form, which should be divided into two portions. To the one add some dilute hydrogen nitrate, which will fail to dissolve it; to the other add excess of ammonia, which will also fail to dissolve it. It is, however, readily soluble in excess of potassium iodide and sparingly so in excess of silver nitrate.

7. Add solution of mercury dichloride drop by drop to some solution of potassium iodide. The first drop will produce a yellow precipitate, rapidly changing to scarlet, which will redissolve on agitation. Continue to add the mercury salt until some quantity of the precipitate is obtained; pour off a little of this, and add to it a little more of the solution of mercury dichloride; the precipitate will redissolve. Pour the rest of the liquid, in which the precipitate is suspended, on a filter, and wash it once or twice with a gentle stream of water from the wash-bottle; then spread out the filter on some folds of blotting-paper, and put it in a warm place to dry. Detach the dried mercury iodide from the filter, place it in a small test-tube, and heat it over a lamp. The scarlet iodide will sublime and condense in yellow prismatic crystals in the cool part of the tube. If these crystals be rubbed with a glass rod they will be reconverted into the scarlet modification, which has the same chemical composition as the yellow iodide but differs in crystalline form. If you possess a moderately good microscope, you will find it interesting to watch the progress of the

change by subliming a little of the scarlet mercury iodide from a watch-glass to one of the ordinary microscope slides, placing it on the stage of the instrument, examining the crystals with a one-inch power, and lightly touching one of them with a needle. The change will begin in the crystal which was touched and gradually spread through the whole, while its progress may readily be watched; the yellow rhombic prisms becoming aggregations of red octohedra with a square base, belonging to a different crystallographic system.

8. Add one drop of weak chlorine water to a very dilute solution of potassium iodide. Iodine will be liberated and will colour the solution yellow. On addition of a few drops of solution of starch, the characteristic deep blue compound will be formed. Now add some stronger chlorine water, and notice that the starch reaction will entirely disappear; owing to the formation of a colourless iodine chloride which does not act upon starch. Hence in testing for iodine it is important not to use an excess of chlorine. It is, in fact, best not to use chlorine at all for the purpose of liberating iodine, but hydrogen nitrite, as described in the next experiment.

9. Add a few drops of hydrogen sulphate to solution of potassium iodide, and then a small quantity of a recently-made solution of potassium nitrite. Shake up the yellow solution with a little carbon disulphide, and allow it to stand for a minute. The carbon disulphide will be found to have withdrawn the iodine from the solution, collecting into a deep purple globule at the bottom of the tube. Repeat the similar experiment with bromine, given at p. 162, and compare the colours of the two globules, the bromine imparting a bright orange, the iodine a fine purple colour to the carbon disulphide. Notice also that sodium nitrite will not decompose hydrogen bromide, but that chlorine water must be used for the purpose. If some chlorine water is added to the contents of both tubes, and the mixture shaken and again allowed to settle, the globule containing iodine will be found to have lost its colour, while the globule containing bromine remains unchanged.

FLUORINE.

1. Make a small leaden cup, by turning up the edges of a circular piece of thin sheet lead, about 7 or 8 cm. in diameter. This is conveniently effected by placing the piece of lead in a mortar just large enough to hold it, and pressing forcibly on its centre with the pestle, so as to mould it to the shape of the interior of the mortar. If you have a platinum crucible, this will serve the purpose of the following experiment even better. Now take a piece of sheet glass, about 8 cm. in diameter, warm it before the fire or over a lamp, and when it is thoroughly hot, rub over it a piece of bees'-wax, and incline the plate in different directions, so that the melted wax may run over it and form an even coating. Set it up edgeways to cool, on a piece of paper, and meanwhile grind a small bit of fluor spar to powder, and place it in the leaden dish. Place the piece of glass coated side upwards on the table, and trace on it with a pointed piece of wood (a match cut like a pencil answers very well) any letters or device which may occur to you, taking care that the lines are drawn quite through the wax so as to expose the bare glass. Pour a little strong hydrogen sulphate upon the fluor spar in the leaden dish, and cover the dish with the piece of glass, the coated side being downwards. Heat the dish very gently by placing it on some warm sand, and leave it for a short time undisturbed. The hydrogen sulphate will decompose the fluor spar, forming calcium sulphate, while gaseous hydrogen fluoride will be evolved, which will act upon the glass where it is unprotected by the wax. In about five minutes, the glass may be removed from the dish; the pungency of the fumes of hydrogen fluoride should be noticed, and the contents of the dish washed away at once in order that the glass bottles in the room may not be damaged. The wax may be removed from the glass by warming the glass and rubbing it with a cloth, and the device will be found deeply and permanently etched into the glass.

2. The preparation of silicon fluoride has been given at p. 114. Its formation may be illustrated on a small scale by placing a mixture of equal parts of finely powdered fluor spar and glass or fine sand in the leaden dish used in the last experiment, adding some strong hydrogen sulphate, heating gently, and covering the dish with a bit of glass on the centre of which a drop of water has been deposited. The drop will soon become coated with a film of silica, owing to the decomposition of the silicon fluoride by moisture.

SULPHUR AND SULPHIDES.

1. Place 4 or 5 grms. of 'flowers of sulphur' in a test-tube, and pour over them about 10 c.c. of carbon disulphide. Hold the tube, loosely corked, in a dish of warm water for a few minutes, shaking it occasionally. Care must be taken that there is no lighted lamp or fire near at hand, on account of the inflammability of the carbon disulphide. There will be a residue left, not necessarily because there is not sufficient disulphide to dissolve the substance, but because the flowers of sulphur consist of two varieties of the element, of which one only is soluble in carbon disulphide. Filter the fluid quickly into another tube, through an ordinary paper filter[1]. Pour about 2 c.c. of the solution into a watch-glass, and leave it on the table to evaporate. The carbon disulphide, from its great volatility, will quickly pass off, and small transparent crystals of sulphur will be formed and rapidly increase in size. They may be easily recognised, especially if a magnifier is used, to be right rhombic octohedra, Fig. 48.

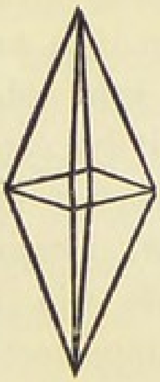

Fig. 48.

2. Place some pieces of roll sulphur or of flowers of sulphur in a common clay crucible about 10 cm. high, and heat it on a common fire, or, embedded in sand, over a lamp until the

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[1] Both the filter and the tubes, &c. used in this experiment must be quite dry.

sulphur is just melted. Then allow it to cool until a thick crust has formed on its surface; after the lapse of three or four minutes break this crust with a glass rod, and, taking up the crucible wrapped in a cloth, pour out the portion which is still fluid into a dish of water. If the rest of the crust be carefully removed with a knife, the interior of the crucible will be found lined with needle-shaped, transparent crystals, Fig. 49, which are referred to the oblique prismatic system, a system totally distinct from that to which the crystals obtained from solution in Experiment 1 belong, viz. the right prismatic, Fig. 48. In a few days the prismatic crystals lose their transparency and are spontaneously converted into aggregations of minute octohedra.

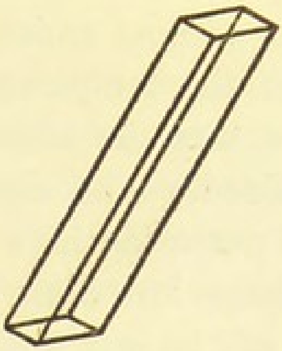

Fig. 49.

3. Put about 30 or 40 grms. of roll sulphur into a Florence flask, support it over a lamp in the retort-stand, placing a piece of wire-gauze under it. While it is being gently heated, fill a jug with cold water, and place in it mouth downwards one of the largest funnels you have. The sulphur will first melt to a clear pale-yellow fluid, almost as mobile as water, but as the temperature rises higher it will darken in colour and become thicker and thicker, until it has so far lost its fluidity that the flask may be inverted for a moment without spilling any of it. When the heat is further raised it becomes again fluid, but not so much so as at first. When it has reached this point, take hold of the neck of the flask with a cloth, and pour its contents in a thin stream into the cold water, immediately round the stem of the funnel. By being thus suddenly cooled, the sulphur will be preserved in the allotropic condition into which it has been converted by heat, and on lifting the funnel out of the water, the threads of sulphur which surround it will be found to be semi-transparent, soft, almost as elastic as india-rubber, and scarcely soluble in carbon disulphide. If the residue in the flask be watched as it cools, it will be seen to undergo the same changes as when it was being heated, but in a reverse

order, becoming thick, then losing its dark colour and becoming fluid again, and finally solidifying to a yellow, crystalline, brittle mass. It may be heated just above its melting-point and suddenly cooled by pouring it into the cold water without undergoing any change of properties. It is only when the temperature has been raised nearly to its boiling-point that the above modification, called 'plastic sulphur,' is obtained. The elastic threads should be dried with a cloth, and put aside in a bottle. In a few days they will be found to have lost both their transparency and their plasticity, but will still be in a great measure insoluble in carbon disulphide.

4. Heat a little more sulphur in the flask used in the last experiment; raise the temperature until it boils, and the flask is filled with the brownish vapours. Now immerse in the flask a close spiral of very thin copper wire, so that the end of the spiral may nearly touch the surface of the boiling sulphur. The wire will burn in the sulphur vapour with incandescence, and be converted into a brittle sulphide.

5. Mix about 2 grms. of iron filings with an equal weight of sulphur, and heat it quickly in a test-tube, until combination takes place with low incandescence. Allow the tube to cool, detach the bronze-coloured friable mass of iron sulphide with a glass rod, transfer it to another tube, and pour a little dilute hydrogen sulphate upon it. Effervescence will take place owing to the evolution of a gas, which will be recognised by its smell to be hydrogen sulphide. Hold a slip of blotting-paper made damp, not wet, with a solution of lead acetate, in the mouth of the tube; it will be at once blackened owing to the formation of lead sulphide. The gas may be kindled at the mouth of the tube, and will burn with a blue flame.

6. Powder a small fragment of iron pyrites in a mortar and place it in a small hard glass tube sealed at one end (Fig. 13). Heat it to redness in the lamp-flame, and notice that a yellowish brown sublimate of sulphur is formed in the cool part of the tube. It may be proved to be sulphur by cutting off the sealed end of the tube (which may be effected by touching it while still hot with a drop of water) and heating the

sublimate gently while the tube is held obliquely so that a current of air may rise through it. The sulphur will be oxidised to sulphurous acid, which may be recognised by its odour and by its acid reaction on a piece of moist blue litmus-paper held at the upper end of the tube.

7. Mix another small portion of powdered iron pyrites with an equal quantity of sodium carbonate; fuse the mixture on a piece of porcelain, pour a little water on the residue and warm it; then dip a bright silver coin into the solution. The silver will be stained black, owing to the formation of a superficial coat of silver sulphide.

SULPHUR DIOXIDE.

The properties of this substance are given at p. 106.

SULPHITES.

[A solution of sodium sulphite containing 5 grms. of the salt dissolved in 20 c.c. of water may be used.]

1. Add a few drops of strong hydrogen sulphate to a little of the solution of sodium sulphite placed in a test-tube. Sulphur dioxide will be given off, and may be recognised by its smell. If a slip of paper moistened with solution of lead acetate is held within the tube it will not be altered in colour. Now drop in a fragment of granulated zinc; the hydrogen produced will decompose the sulphur dioxide, forming water and hydrogen sulphide[1], which latter gas will be recognised by its smell, as well as by its action on the lead acetate.

2. Acidify a little solution of potassium iodide with hydrogen chloride, and add one drop of chlorine water, or of solution of potassium nitrite in order to liberate iodine, which, on addition of some solution of starch, will give a deep blue solution (p. 164). Add to this a few drops of solution of sodium

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[1] $SO_2 + 3H_2 = H_2S + 2H_2O$.

sulphite; the solution will immediately become colourless, the iodine being converted into hydrogen iodide[1].

In Experiment 1 we removed oxygen from sulphur dioxide, while in Experiment 2 we have effected a reverse change, and have added oxygen to it. This latter is the change which sulphur dioxide, in presence of water, is most prone to undergo, and it is, on this account, a very useful reducing agent for laboratory purposes.

HYPOSULPHITES.

[A solution of sodium hyposulphite containing 6 grms. of the salt in 25 c.c. of water may be used.]

1. Add to a portion of the solution of sodium hyposulphite a few drops of dilute hydrogen chloride. The solution will almost immediately become turbid, owing to precipitation of sulphur, while sulphur dioxide is given off.

2. To another portion of the solution add some solution of silver nitrate. A precipitate is produced (soluble in excess of the hyposulphite), which is at the first moment white, but rapidly becomes yellow, brown, and finally black. These changes are due to the instability of the silver hyposulphite which is first precipitated; in presence of water it is rapidly converted into silver sulphide[2].

SULPHATES.

1. Place about 30 c.c. of water in a dish or beaker, and add to it quickly an equal volume of strong (common) hydrogen sulphate. The heat evolved by the union of the acid and the water is very great, as has been before noticed (p. 86), and if a little alcohol (or even water) is placed in a test-tube and immersed in the solution, it will soon boil. A white turbidity

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[1] The general nature of the change is shown in the following equation:

$$I_2 + H_2O + H_2SO_3 = 2HI + H_2SO_4.$$

[2] $Ag_2S_2O_3 + H_2O = Ag_2S + H_2SO_4.$

will, in all probability, be produced in the diluted acid: this consists of lead sulphate (an impurity always present in the common acid, and derived from the lead cisterns used in its manufacture) which is soluble in the strong acid, but is thrown down on dilution. Add another 30 c.c. of water, and leave the liquid to cool, and the precipitate to settle. The clear solution may then be poured off and used in Experiments 3 and 4. Any that remains should be reserved for galvanic batteries and other uses.

2. Dissolve sufficient white sugar in 20 c.c. of water to form a thick syrup, place it in a beaker standing on a plate, add to it an equal volume of strong hydrogen sulphate, and stir the mixture. It will become hot and blacken, and will finally swell up into a porous pasty mass of charcoal. Sugar may be regarded as a combination of water and carbon, and hydrogen sulphate has so strong an affinity for water that it withdraws it from combination, leaving the carbon.

3. Add a drop of dilute hydrogen sulphate to a solution of barium chloride in a test-tube. A dense white precipitate of barium sulphate will be formed, which will not dissolve on the addition of a little strong hydrogen chloride, even on boiling.

4. Add a drop of dilute hydrogen sulphate to solution of lead acetate in a test-tube. A white precipitate of lead sulphate will be formed, insoluble in cold hydrogen chloride, but slowly decomposed by it on boiling.

5. For a method of detecting a sulphate by its reduction by means of carbon with formation of a sulphide, see Experiment 12, p. 138.

PHOSPHORUS.

[It is hardly necessary to say that whenever phosphorus is to be used for an experiment, it must be handled with the greatest care, owing to the low temperature at which it catches fire. It should be cut under water and should never be touched by the fingers when dry; a pair of crucible tongs being used to transfer it from one place to another. A jug of water should always be at hand, in case the phosphorus should, in spite of precautions, catch fire[1].]

1. Place a small fragment of phosphorus, carefully dried by blotting-paper, on a watch-glass, and float it on hot water in a basin. The phosphorus will melt and inflame spontaneously giving off white fumes of phosphorus pentoxide, and leaving a small residue consisting of red phosphorus.

2. Place about 1 c.c. of carbon disulphide in a test-tube, add a small fragment of dry phosphorus about half the size of a pea, and cork the tube. Leave it to digest for a few minutes, shaking it occasionally to promote the solution of the phosphorus, then pour the clear solution obtained upon a piece of blotting-paper placed in a plate. The phosphorus will, as the carbon disulphide evaporates, be left in a state of fine division upon the paper; it will emit white fumes and will shortly catch fire and burn away. The paper itself will be found to be only superficially charred, owing to the protective action of the phosphorus pentoxide formed.

3. Set up a small apparatus for preparing carbon dioxide (p. 89). Support a large test-tube vertically in a Bunsen's holder, at such a height above the table that a lamp may be placed under it. Pass into it a stream of carbon dioxide, and when it is full (which may be ascertained by bringing a lighted match or taper just within its mouth) drop into it sufficient phosphorus, carefully dried, to form a stratum about 1 cm. in depth. Heat the tube very gently and cautiously with a lamp, maintaining the current of gas. The phosphorus will melt to a clear fluid, but will not inflame, since it has no action on carbon dioxide. Now remove the lamp, place a plate containing a little water underneath the tube, and drop in a very

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[1] As the burns produced by phosphorus are unusually severe and slow to heal, it may be worth mentioning that lint soaked in water containing a little common washing soda is the best application at first. After the hydrogen phosphate is by this means neutralised, nothing is better than lint soaked in a mixture of equal volumes of glycerine and water, the whole being covered with oilskin to prevent evaporation.

small fragment of iodine. A violent action will commence, either at once or on cautiously raising the temperature a little higher, and the phosphorus will be converted into the red modification. Intense heat is evolved at the moment of change, and the tube is sometimes cracked; hence the advisability of placing a plate or dish of water underneath it, to catch the phosphorus if the tube should break. The product, a hard, brittle, red mass, should be allowed to cool in the tube, the current of carbon dioxide being still maintained, since traces of unaltered phosphorus always remain, which by their inflammation would set the whole on fire. In order to detach the phosphorus it will generally be necessary to break the tube, which may be done by striking it forcibly against the bottom of a dish containing water. It is, perhaps, scarcely worth while to attempt the purification of the product, which is effected by repeated washings with carbon disulphide (or with caustic soda), and is not free from danger.

4. Seal the end of a piece of glass tube about 5 mm. in diameter and 15 or 16 cm. in length. When it is cold, drop into it a small fragment of amorphous phosphorus and heat the latter gently; notice that it will sublime without previous fusion, and will condense in the cool part of the tube in amber-coloured drops of ordinary phosphorus.

The amorphous phosphorus used in this experiment should not be that which has been prepared by the use of iodine, which latter has the property of volatilising unaltered, probably owing to the presence of a trace of iodine from which it seems impossible to free it.

HYPOPHOSPHITES.

Place about 15 c.c. of solution of potassium hydrate in a small beaker, add a piece of phosphorus about twice as large as a pea, and heat the solution nearly, not quite, to boiling on the sand-bath for about ten minutes. Potassium hypophosphite will be formed[1], while a gas, hydrogen phosphide, will be given off, which differs from all the other gases which we have examined, in being spontaneously inflammable in the air; each bubble, as it rises to the surface of the liquid, catching fire and producing a white smoke-wreath of hydrogen phosphate. The experiment should be made in a draught-cupboard or in the open air, since hydrogen phosphide is an extremely poisonous and offensive gas. Allow the liquid to become quite cold, then decant it into a test-tube, and fill up the beaker with water, to prevent the remaining phosphorus from catching fire before it can be thrown away. Filter the liquid, render it slightly acid with dilute hydrogen sulphate, and examine it as follows:—

1. Evaporate a small portion to dryness, and heat the residue more strongly. It will be decomposed into potassium phosphate and hydrogen phosphide, the latter burning with a greenish flame.

2. Add to another portion a drop or two of solution of copper sulphate (made by dissolving a small crystal of the salt in 1 or 2 c.c. of water), and heat the solution gradually to boiling. A yellow turbidity will at first appear, rapidly changing to a dull red precipitate of metallic copper[2].

PHOSPHATES.

1. Place a piece of carefully dried phosphorus on a plate, set fire to it with a hot wire, and cover it with a deflagrating jar, between the stopper and the neck of which a slip of card has been inserted, to prevent the stopper from becoming fixed in its place. Dense white fumes of phosphorus pentoxide will be formed, and will be deposited partly on the sides of the jar,

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[1] $P_4 + 3KHO + 3H_2O = 3KPH_2O_2 + H_3P$.

[2] This reaction serves to distinguish hypophosphites from phosphites and phosphates, neither of which latter reduce copper salts. The yellow substance formed at first is copper hydride, and is one of the few examples of the combination of a metal with hydrogen.

partly on the plate. Remove the jar and scrape together, with a spatula, the snow-white powder on the plate. Place it in a test-tube, and pour a few drops of water upon it; it will hiss like a red-hot iron from the intensity of the combination, and a solution of hydrogen phosphate will be formed. A piece of blue litmus-paper dipped into the solution will be at once reddened. Notice also that the phosphorus pentoxide deposited on the sides of the jar will, after a few minutes exposure to the air, deliquesce into an oily liquid. From its affinity for water, which has been thus illustrated, phosphorus pentoxide is one of the most efficient substances for drying gases.

Pour the solution of hydrogen phosphate thus obtained into a beaker, and evaporate it until the liquid becomes syrupy, then add a little more water, and boil the solution again for a short time; finally, add solution of sodium carbonate until the addition of it no longer produces effervescence and the liquid is neutral to test-paper. You have now a solution of sodium orthophosphate to which the following tests may be applied:—

(*a*) Place a small portion in a test-tube, and add a drop of solution of silver nitrate. A canary-yellow precipitate will be formed. Pour off one half of the fluid in which the precipitate is suspended into another test-tube, and add a few drops of dilute hydrogen nitrate; to the remaining half add the same quantity of ammonia; in both cases the precipitate will be re-dissolved.

(*b*) To another portion add a few drops of solution of ammonium chloride, then a little ammonia, and test it with a drop of solution of magnesium sulphate. A white crystalline precipitate of magnesium and ammonium phosphate will be formed, which will readily dissolve in dilute hydrogen nitrate.

(*c*) Pour some solution of ammonium molybdate, acidified with hydrogen nitrate, into a test-tube, and add a drop or two of sodium phosphate. The solution will become yellow, and on warming it (it should not be boiled) a yellow precipitate will fall.

(*d*) Acidify another portion with hydrogen acetate and add a few drops of solution of albumen. No change beyond a slight opalescence will be produced, since albumen is not coagulated by hydrogen orthophosphate.

2. Dissolve a crystal of sodium and hydrogen phosphate (the ordinary 'phosphate of soda') in a little water, and apply tests *a*, *b*, and *d* to the solution. Similar results to those given above will be obtained. Now place a few crystals of the same salt in a porcelain crucible, and heat for some time to redness. The salt will first melt, then give off water, and finally will leave a white earthy-looking residue; this should be dissolved in cold water, and tested with the same tests as the original salt.

(*a*) A white granular precipitate of silver pyrophosphate will be obtained, soluble in hydrogen nitrate and ammonia.

(*b*) A white precipitate will fall, which is soluble in excess of the magnesium sulphate, but reprecipitated on boiling.

(*c*) The result will be the same with the orthophosphate.

3. Repeat Experiment 3, using sodium ammonium and hydrogen phosphate ('microcosmic salt'). The salt will give off ammonia and water, and leave a transparent glassy residue of sodium metaphosphate, which on being tested as above will give the following reactions:—

(*a*) A white precipitate.

(*b*) No precipitate, if enough ammonium chloride has been previously added.

(*c*) A white precipitate of coagulated albumen will form at once.

This last test is especially characteristic of hydrogen metaphosphate.

4. Add a few drops of hydrogen nitrate to some of the solution either of sodium pyrophosphate or of sodium metaphosphate, and boil for ten minutes in a beaker. Hydrogen orthophosphate will be reproduced, as may be proved by the above tests.

BORATES.

1. Make a concentrated solution of sodium borate by boiling the crystals with water in a test-tube until no more is dissolved. Add to the solution, while still warm but not boiling, about half its volume of strong hydrogen sulphate, and cool the mixture. Shining scales of hydrogen borate will be precipitated, from which the solution of sodium sulphate may be decanted, and the crystals left to drain on a porous tile.

2. Place a little of the hydrogen borate obtained in the last experiment on a piece of porcelain, or a piece of platinum foil bent up to form a cup, and heat over a lamp. The crystals will fuse, give off water, and leave a clear glassy residue of boron trioxide.

3. Place a little hydrogen borate in a watch-glass, pour over it some common alcohol and set fire to it. The alcohol flame will be coloured yellowish green, especially if the mixture be stirred with a glass rod and warmed. The same result will be obtained if, instead of the hydrogen borate, a little sodium borate mixed with strong hydrogen sulphate be used.

4. Place a little sodium borate in a watch-glass, add a few drops of water, and then enough hydrogen chloride to make the solution give an acid reaction to test-paper. Dip into the solution one-half of a slip of turmeric-paper, and dry it at a gentle heat, by holding it before the fire; the dipped portion will be coloured red. If a drop of sodium carbonate be placed on the reddened portion of the turmeric-paper, the colour will be changed to black.

SILICATES.

1. The best method of obtaining silicon dioxide in a pure state has already been given, p. 114.

2. Heat a piece of flint to full redness in a common fire, and quench it suddenly in water. It will then form a white friable

mass, about 1 grm. of which should be ground to powder in a mortar, mixed with four times its weight of sodium carbonate, and fused in a platinum crucible, or on a piece of platinum foil turned up at the edges. A full red-heat, such as that of a gas blowpipe, will be required for the fusion. Pour a little water over the fused mass when it is cold, and boil it rather quickly over a lamp. By this means the whole of the sodium silicate may generally be detached from the crucible, and should then be boiled for some time in a beaker. You will thus obtain a solution of sodium silicate, similar to that which is called 'soluble glass,' with which the following experiments should be tried:—

(*a*) Add a few drops of hydrogen chloride to a portion of the hot solution, in a test-tube. A dense gelatinous precipitate will be formed of hydrogen silicate, and, if the solution of sodium silicate is strong, the whole will appear to solidify.

(*b*) Place some dilute hydrogen chloride in a test-tube, and add drop by drop a little of the solution of sodium silicate, which should be diluted, if strong. No permanent precipitate will now be formed, since the hydrogen silicate is retained in solution when thus formed in presence of an excess of acid.

(*c*) Add a little hydrogen chloride to a dilute solution of sodium silicate, and evaporate to complete dryness. Pour on the white residue some dilute hydrogen chloride, warm it, and notice that the silicon dioxide, which is formed, remains completely undissolved. Decant the solution, dry the residue, and heat a small portion of it with a bead of sodium ammonium and hydrogen phosphate, made in the same way as a borax bead, p. 135. The residue will float in the melted bead, without dissolving or altering in appearance. Another portion may be tested by fusion with sodium carbonate in a loop of platinum wire, p. 137, when a globule of clear glass will be obtained.

[The student is recommended, before passing on to the study of the different metals, to refer to the account of the course of analysis given at the commencement of Part II, and to practise himself in the analysis of single salts containing some one of the foregoing non-metallic radicles associated with an alkali-metal.]

ARSENIC.

1. Place a little arsenic trioxide in a small dry test-tube, and heat it over a lamp; the substance will volatilise completely, and form a ring of crystals, often iridescent, in the cool part of the tube. Examine these crystals with a magnifier; they will be found to be octohedra, with a square base.

2. Mix a small quantity of arsenic trioxide with an equal quantity of sodium carbonate and the same amount of potassium cyanide. Place the finely-powdered and dried mixture in a small tube of hard glass about 5 cm. long, having a bulb blown at one end (for the method of making these tubes, see p. 37), and wipe off any adhering particles of the mixture from the sides of the tube with a twisted strip of filter-paper. Heat the bulb, gently at first, over the lamp, and absorb any moisture which may condense in the tube with a strip of filter-paper; then increase the heat, and notice that a shining ring of metallic arsenic will be deposited in the tube. Cut off the bulb, retaining the metallic sublimate in the tube; hold the latter in a slanting position (the part containing the arsenic being lowest), and heat the ring by a spirit lamp with a very small flame. The arsenic will volatilise, will be oxidised by the current of hot air ascending in the tube, and will condense near the top of the tube as arsenic trioxide, in small crystals. The heat should not be too strong, or a portion of the arsenic will volatilise unchanged and interfere with the distinctness of the crystalline ring. If this should occur, allow the tube to cool, and re-sublime the deposit as above directed.

3. Boil a little arsenic trioxide in a test-tube with some water; notice the difficulty with which it is wetted by water, the powder showing a tendency to float as a film upon the surface of the liquid, or remain at the bottom in lumps.

It will, however, dissolve to a certain extent in water alone, and much more readily if a few drops of solution of sodium carbonate be added. Filter the solution, if necessary, and test separate portions as follows:—

(*a*) To one portion add some solution of hydrogen sulphide; the liquid will be coloured yellow, and on addition of a few drops of hydrogen chloride a bright yellow precipitate of arsenic trisulphide will be formed. Pour some of the liquid, in which the precipitate is suspended, into another test-tube, add some strong hydrogen chloride, and boil. The precipitate will remain almost unaltered, but will be readily decomposed and dissolved on the addition of a drop of hydrogen nitrate.

Neutralise the rest of the solution with ammonia, add a few drops of ammonium sulphide, and warm. The arsenic trisulphide will be dissolved, ammonium sulpharsenite being formed. If a few drops of dilute hydrogen chloride be now added to the clear solution, arsenic trisulphide will be reprecipitated.

(*b*) Add a few drops of hydrogen nitrate to another portion of the solution, and then add a drop of silver nitrate. If the hydrogen nitrate or sodium carbonate contained any chloride, a slight white precipitate will form on addition of the silver nitrate, which should be filtered off. Mix in another test-tube some solution of ammonia with an equal volume of water, and pour the diluted ammonia very slowly and carefully down the side of the test-tube containing the arsenic solution, so that the two fluids may not mix, but the lighter solution (the ammonia) may rest on the surface of the heavier. The tube containing the arsenic solution should be held in an inclined position, in order that the ammonia may flow less rapidly down the side. It is best to add the ammonia by means of a pipette (a straight piece of glass tube, about 20 cm. long, terminating in a jet, p. 30), which should be dipped into the tube containing the dilute ammonia, the top closed by a moistened finger, the pipette withdrawn, and the fluid it contains allowed to drop slowly down the side of the test-tube containing the arsenic solution, by slightly relaxing the pressure of the finger on the top of the tube. A yellow film of silver arsenite will be formed at the surface of contact of the two fluids. We have in the test-tube three fluid strata: a lower one containing excess of hydrogen nitrate, an upper one containing excess of ammonia, and an intermediate one consisting of a neutral solution of ammonium nitrate. Silver arsenite is insoluble in water, but soluble in excess both of acid and of alkali, and therefore appears only where the fluid is neutral. On shaking the mixture the yellow film will disappear altogether, unless just enough ammonia has been added to neutralise the acid.

(*c*) Add to a third portion a drop of solution of copper sulphate, and then, if a precipitate is not produced at once, add a drop or two of potassium hydrate. A grass-green precipitate of copper arsenite will be formed, readily soluble in hydrogen nitrate as well as in ammonia.

(*d*) Add excess of potassium hydrate to another portion, and then a drop of dilute solution of copper sulphate. A clear blue solution will be formed, which will become turbid on boiling, and deposit a red precipitate of copper suboxide.

(*e*) Acidify another portion with hydrogen chloride, and drop into it a piece of rather fine copper wire, cleaned with emery-paper and doubled up into a bundle. On boiling the solution, the arsenic will be thrown down on the more electropositive metal, the copper, in the form of a grey metallic film, which will, if the action be continued, separate in scales from the copper. Decant the solution, and pour some fresh water upon the coated copper, to wash away the last traces of the solution; then shake the copper out of the tube upon a piece of blotting-paper and dry it by gentle pressure, taking care not to disturb the film of arsenic; complete the drying by warming the copper over a lamp-flame for a few seconds, then place it in a tube of hard glass sealed at one end, similar to that used in Experiment 2, and heat it over the lamp. A ring of crystals of arsenic trioxide will be formed beyond the heated portion of the tube, and should be examined with a lens.

This test, which is usually called Reinsch's test, has the advantage that the presence of organic substances (e. g. milk, beer, in cases of poisoning) does not interfere with the decomposition of arsenic on the copper. But other metals, such as antimony, bismuth, silver, are deposited on the copper under the same conditions as arsenic. Moreover, when the deposit of arsenic is heated, only a portion of the metal is volatilised, the rest forming a non-volatile alloy with the copper. The test, therefore, is by no means so reliable and characteristic as the following one.

4. If a solution containing arsenic is brought into contact with nascent hydrogen, a volatile compound of arsenic and hydrogen is formed, corresponding in composition to the compound of nitrogen and hydrogen, or ammonia, and to one of the compounds of phosphorus and hydrogen. This reaction furnishes us with a most delicate test for arsenic, since the hydrogen arsenide is very readily decomposed, and thus the presence of arsenic in it may be demonstrated. The best method of applying the test, which is known as Marsh's test, is as follows:—

Place a few pieces of granulated zinc in a small flask fitted with an acid funnel and right-angled elbow tube, and connect the latter with a drying tube filled with fragments of calcium chloride, and supported in a horizontal position by a Bunsen's holder. Take a piece of infusible glass tubing about 4 mm. in diameter and 40 cm. in length; soften it in the middle by means of the blowpipe-flame, and draw the two ends asunder, until the softened portion is contracted to a diameter of 2 mm. When it is cool, make a scratch with a file at the middle of the contracted portion, and break it at that point. You have now two tubes each about 18 cm. in length and terminated by a jet[1]. Reserve one for an experiment with antimony, and connect the other with the drying tube, supporting it horizontally on the largest ring of the retort-stand at such a height

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[1] The jet shown in the figure is turned up. This, which is a slightly preferable form, may be obtained by moving the hands, in drawing out the glass, laterally (the one hand towards, the other from the body).

that a lamp may be placed underneath it. The whole apparatus will then be arranged thus, Fig. 49.

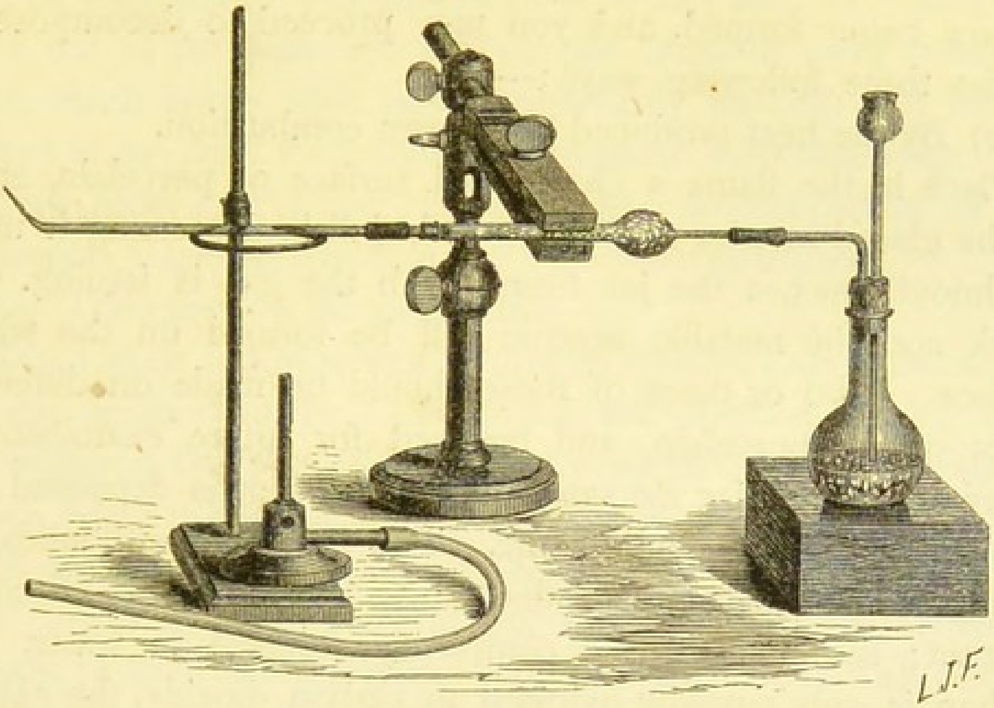

Fig. 49.

Pour a little water on the zinc in the flask, and add a few drops of strong hydrogen sulphate, so as to generate a moderately rapid current of hydrogen. Allow the gas to pass through the apparatus for about two minutes, in order to sweep out all traces of air. While this is going on, you may get ready a right-angled elbow tube with one long branch, a test-tube half filled with a dilute solution of silver nitrate acidified with one drop of dilute hydrogen nitrate, and one or two pieces of clean porcelain, such as the lid of a crucible, or fragments of an evaporating dish. Now light the hydrogen gas at the jet, and depress into the flame, for a few seconds, one of the pieces of porcelain. If any stain appears on the white surface (as may happen if the zinc or the hydrogen sulphate is contaminated with arsenic) allow the stream of gas to pass a little longer, and do not proceed with the experiment until the porcelain depressed on the flame remains unstained. When this is the case, pour into the flask a very few drops of the solution of arsenic trioxide. The first effect of this will be to increase the evolution of gas, and the flame at the jet will shortly become tinged with grey (not unlike the colour imparted to flame by the presence of potassium vapour). The compound of arsenic with hydrogen is now being formed, and you may proceed to decompose it in the three following ways:—

(*a*) By the heat produced by its own combustion.

Place in the flame a clean, cold surface of porcelain, such as the glazed interior of an evaporating dish, depressing it until it almost touches the jet from which the gas is issuing. A black spot of metallic arsenic will be formed on the white surface. Two or three of these should be made on different parts of the porcelain, and reserved for future examination. They are formed for the same reason that soot is deposited on a plate held in a candle-flame. The gaseous compounds of carbon and hydrogen are decomposed by the heat of combustion; the hydrogen is completely oxidised to water, the carbon is only partially oxidised to carbon dioxide, the excess of carbon being suspended in the flame and deposited on any cold surface brought in contact with it. Similarly in the present experiment the hydrogen of the hydrogen arsenide is oxidised into water, while an arsenic soot is deposited on the porcelain.

(*b*) By heat applied to the tube through which the gas is passing.

Place a lamp underneath the tube near the end at which the gas enters, and heat that part of the tube to redness. A mirror-like ring of arsenic will be deposited just beyond the heated portion of the tube, while the flame at the jet will lose its grey colour, showing that the arsenic has been arrested in its course to the jet.

(*c*) By a solution of silver nitrate.

Detach the tube containing the arsenic deposit, and fit in its place the elbow tube, the longer branch of which should reach to the bottom of the test-tube containing the solution of silver nitrate: the latter may be supported in an upright position by the smallest ring of the retort-stand. Pour, if necessary, a little more acid and solution of arsenic trioxide into the flask.

The solution of silver nitrate will soon become turbid, and a black deposit of metallic silver will be formed while silver arsenite will remain in solution in the hydrogen nitrate.

The evolution of gas should now be stopped, by taking the cork out of the flask and pouring the solution away from the zinc, which latter, after being thoroughly washed, may be retained in the flask for a similar experiment with antimony.

You have now three results of the decomposition of the hydrogen arsenide, which are to be further examined.

(*a*) The spots on the porcelain.

Dip a glass rod in strong hydrogen nitrate, and moisten one of the spots with it. The metal will dissolve in the acid, especially if the porcelain be gently warmed, and on evaporation a white residue of hydrogen arsenate will be left. Do not raise the temperature beyond the point necessary to drive off the excess of hydrogen nitrate, lest the hydrogen arsenate should be itself decomposed. Moisten the residue with a drop of solution of silver nitrate and again evaporate to dryness. A red deposit of silver arsenate will be left, readily seen on the white porcelain.

(*b*) The mirror formed in the tube.

Connect the tube with the calcium chloride drying tube, and arrange them as before, when the arsenic mirror was to be formed. In place of the hydrogen generating flask attach to the drying tube a test-tube fitted with a gas funnel and elbow tube, and containing one or two lumps of iron sulphide and a little water. Pour down the funnel a few drops of strong hydrogen sulphate, just sufficient to liberate a slow stream of hydrogen sulphide gas. When the air has been expelled from the apparatus, light the gas at the jet (merely to prevent its escape into the room), and heat the arsenic mirror by a spirit lamp, beginning at its outer border, or that farthest from the end at which the gas enters. The arsenic as it volatilises will decompose the hydrogen sulphide, combining with its sulphur and forming a yellow ring of arsenic trisulphide in the cool part of the tube, which is very volatile and may be driven about from one part of the tube to another by the heat of the lamp.

(*c*) The solution containing silver arsenite.

Filter this from the reduced silver, and test the clear solution as directed in Experiment 3 *b*, p. 184, by carefully neutralising with ammonia, when a yellow stratum of silver arsenite will be precipitated at the point where the solution is neutral.

These reactions should be carefully compared with those of antimony, given at p. 192, and it is best so to arrange your work that the analogous experiment with antimony should immediately follow the test for arsenic.

5. Place about 1 grm. of arsenic trioxide in a small evaporating dish, and pour over it sufficient strong hydrogen nitrate to cover it. Heat the mixture gently over a lamp in the open air, or at any rate in some place where there is a sufficient current of air to carry off the vapours of nitrogen oxides which are evolved. Evaporate it to complete dryness, then add some water, boil, and filter the solution which now contains hydrogen arsenate, formed by the decomposition of the hydrogen nitrate by the arsenic trioxide. Apply the following tests to separate portions of the filtrate:—

(*a*) Add a few drops of dilute hydrogen chloride and then some solution of hydrogen sulphide. No precipitate will be produced, and even on heating only a slight yellow precipitate will fall. Add to another portion some solution of hydrogen sulphite, boil the liquid until it no longer smells of sulphur dioxide, and then test with hydrogen sulphide. A yellow precipitate of arsenic trisulphide will be formed, the hydrogen arsenate having been reduced to hydrogen arsenite by the action of the hydrogen sulphite.

(*b*) Acidify a portion with hydrogen nitrate, add a few drops of solution of silver nitrate, and pour on the solution dilute ammonia, as directed, p. 184. A dull red precipitate of silver arsenate will form at the point where the solution is neutral.

(*c*) Add a drop of copper sulphate to another portion, and then a drop of potassium hydrate. A blue precipitate of copper arsenate will be formed.

(*d*) Add a few drops of ammonium chloride to another portion, then ammonia until the solution is strongly alkaline, and lastly a drop of magnesium sulphate. A white crystalline precipitate will be formed, very similar in appearance, and analogous in composition, to the magnesium ammonium and hydrogen phosphate, the phosphorus being replaced by arsenic.

ANTIMONY.

1. Place a small fragment of metallic antimony on an anvil and strike it with a hammer. It will split to pieces, and may readily be crushed into a fine crystalline powder. Put a little of this powder into a test-tube and pour over it some strong hydrogen nitrate. Red vapours of nitrogen tetroxide will be produced, and the antimony will be converted into a white substance (the trioxide), but will not dissolve. Place some more of the powdered metal into another test-tube, add some hydrogen chloride and heat to boiling. The metal will dissolve very slowly; its solution will be hastened by the addition of one or two drops of hydrogen nitrate. When the action has ceased allow the liquid to cool, and decant the clear solution of antimony trichloride into another test-tube for use in experiments.

2. Mix a little antimony trisulphide, or tartar emetic (antimony and potassium tartrate), with sodium carbonate and potassium cyanide, and heat the mixture on charcoal before the blowpipe. Brilliant globules of metal will be produced, which lose their lustre at once on being removed from the reducing flame, and white fumes of antimony trioxide are produced and form an incrustation on the cool part of the charcoal. Owing to the volatility of the metal, these white fumes of oxide are emitted for some time after the globule is removed from the flame, especially if a stream of air from the blowpipe is directed upon it. When cold, the globule should be detached from the charcoal, placed in a mortar, and the pestle pressed forcibly down upon it. It will, as already observed in Experiment 1, be crushed to powder instead of spreading out into a plate.

3. Pour a little of the solution of antimony chloride, obtained in Experiment 1, into a test-tube and add two or three times its volume of water. A white precipitate consisting of antimony oxychloride will be formed. Pour off some of the milky fluid into another test-tube and add a few drops of strong hydrogen chloride, which will dissolve the precipitate. To the remainder of the fluid add some solution of hydrogen tartrate. The precipitate will in this case also be redissolved. (Compare the reaction of the corresponding bismuth salt, p. 212.)

4. Test another portion of the solution of antimony chloride with hydrogen sulphide. A characteristic orange precipitate of antimony trisulphide will be obtained. Allow this to subside, pour off the fluid, and shake up the precipitate with a little water. After thus roughly washing it by decantation, divide it into two portions: pour on the one portion a little strong hydrogen chloride, and heat to boiling; the precipitate will dissolve. Digest the other portion with some ammonium sulphide at a gentle heat; the precipitate will readily dissolve, but will be again thrown down on addition of a little dilute hydrogen chloride.

5. Place a piece of platinum foil in an evaporating dish; put on it a small fragment of granulated zinc, and pour into the dish a little dilute hydrogen sulphate or chloride. When the evolution of hydrogen has begun from the surface of the platinum, add a drop or two of the solution of antimony terchloride. The antimony will be thrown down on the platinum as a black coating, owing to galvanic action. If the black deposit is heated with a drop of ammonium sulphide it becomes orange.

6. Set up an apparatus for Marsh's test, and, referring to pp. 188–190, repeat the experiments there given, using the antimony salt instead of an arsenic salt. Antimony, like arsenic, forms a volatile compound with hydrogen, which may be decomposed by heat and silver nitrate, but with results which enable us to distinguish it without difficulty from hydrogen arsenide.

(*a*) The dark spots formed on the porcelain are more like soot, and not so brown and lustrous as those of arsenic. They leave a white residue when treated with hydrogen nitrate, but yield no red deposit with silver nitrate.

(*b*) An orange-red deposit of antimony tersulphide will be formed, close to the mirror in the tube, which is only volatilised with difficulty, by the utmost heat of the lamp.

(*c*) The whole of the antimony is precipitated as silver antimonide. No yellow layer or precipitate, therefore, is seen when the filtrate is neutralised by ammonia. Wash the precipitate on the filter; then place the filter with the precipitate in a beaker and boil it with a little solution of hydrogen tartrate, which will dissolve the antimony. Filter the solution and test it with hydrogen sulphide, which will give the characteristic orange precipitate of antimony tersulphide.

It is obvious that in cases where we have both antimony and arsenic in the same solution this last test gives us a means of separating the two metals, the antimony being found in the precipitate produced when the gases are passed into the silver nitrate solution, while the arsenic remains in solution.

The two metals may also be discriminated by the difference in volatility of their sulphides. When the mirror in the tube is heated in a current of hydrogen sulphide, the yellow ring of arsenic tersulphide is always deposited considerably in advance of the orange sublimate of antimony tersulphide.

TIN.

1. Granulate some tin, by melting it in an iron ladle or in a crucible, and pouring it from a height into a jug of water, as directed in the case of zinc, p. 23. The granulated metal may be placed in a funnel, washed with distilled water, allowed to drain, and reserved in a bottle for use.

2. Take a strip of tin (the best tin is usually sold in strips

or thin rods), bend it quickly backwards and forwards several times, holding it to the ear while doing so. A crackling sound will be heard, and the metal will become sensibly warm at the point of flexure. Cast-tin although to a certain extent malleable and ductile is crystalline in structure, and the crackling sound and heat is due to the motion of the crystals over each other, and their mutual friction. The crystalline structure may be rendered evident by immersing the end of a rod of tin in a little mercury in a test-tube; in a few days the rod will be dissected, as it were, and the faces of the crystals will be plainly seen, if the mercury is brushed away. Or, the surface of a piece of tin plate may be washed first with alcohol to free it from grease, and then with a mixture of one part of hydrogen chloride, one part of hydrogen nitrate, and three parts of water. The mottled, watered surface thus produced owes its appearance to the unequal reflection of light from the faces of the crystals.

3. Reduce a little tin dioxide on charcoal before the blowpipe, as directed, p. 137.

4. Place a few pieces of granulated tin, or of tinfoil, in a test-tube, and pour on them some hydrogen nitrate diluted with a few drops of water. Notice the violent action, the evolution of orange vapours of nitrogen tetroxide, and the formation of a white powder, tin peroxide, which is insoluble in excess of the acid.

5. For the preparation of tin protochloride and perchloride, see p. 113. The solutions there mentioned may be used in the following experiments:—

6. Place about 20 c.c. of tin perchloride in a beaker; dilute it with about 150 c.c. of water, and place in it a strip of thick sheet zinc, or a zinc rod, suspended by a thread from a piece of glass rod or tube lying across the rim of the beaker. Leave it undisturbed for a few hours. You will find that the tin is slowly deposited on the zinc in the form of shining metallic leaves, while an equivalent weight of zinc enters into solution.

7. Place a few drops of solution of tin protochloride in each

of four test-tubes, dilute it with about four times its bulk of water, and test as follows:—

(*a*) Add one drop of solution of potassium hydrate. A white precipitate of tin protohydrate will be formed, which will redissolve when more potash solution is added. If the solution of tin protochloride contains much free acid it may require two or three drops of the solution of potassium hydrate to produce a permanent precipitate, the first drop being employed in neutralising the excess of acid.

(*b*) Add some solution of hydrogen sulphide. A dark brown precipitate of tin protosulphide will be formed. Allow this to subside, wash it once or twice by decantation, then add ammonia to alkaline reaction, and lastly a few drops of solution of ammonium sulphide, and warm the mixture. The precipitate will redissolve, if the ammonium sulphide contains an excess of sulphur, as may be known by its colour being yellow. Now add a few drops of dilute hydrogen chloride to the clear solution. A yellow precipitate of tin persulphide will be thrown down, instead of the brown protosulphide.

(*c*) Add a drop of solution of mercury perchloride. A white precipitate of mercury protochloride will be produced, the colour of which changes, however, immediately to grey (the tin salt being in excess) owing to its reduction to metallic mercury.

(*d*) Add one drop of tin perchloride, dilute with more water, and add a drop of solution of gold perchloride. A brownish purple precipitate will be formed, called from its discoverer, the purple of Cassius, containing gold, tin, and oxygen, but not, apparently, of definite composition. When the solutions are very dilute, the colour of the liquid changes to brownish red, but no precipitate falls which can be separated by filtration.

8. Place a few drops of solution of tin perchloride in four test-tubes, dilute with water as above, and apply the above tests to separate portions of the solution.

(*a*) A white precipitate of tin perhydrate, or hydrogen stannate will be formed, which will dissolve, like the protohydrate, in excess of the precipitant.

(*b*) A yellow precipitate of tin persulphide will be formed more quickly if the solution is warmed. It will dissolve in ammonium sulphide, and be reprecipitated unchanged on addition of hydrogen chloride.

(*c*) No precipitate will be produced.

(*d*) No precipitate will be obtained.

GOLD.

1. Spread a piece of gold-leaf on a glass plate, to examine its transparency. This will be most readily done in the following way. Clean the glass plate, which should be about 15 cm. square, with a tuft of cotton-wool dipped in alcohol, dry it with a clean cloth, and lay it flat on the table. Slightly damp the edge of a paper-knife and lay it on one edge of the leaf of gold, to which it will adhere; you can then transfer the leaf to the plate and lay it approximately flat on the glass. Detach the paper-knife from the gold by slightly rubbing it against the glass, and then, still keeping the glass horizontal, direct a gentle stream of water from a wash-bottle under the leaf of gold. The water will spread quickly over the glass, and the leaf will float on it, losing all inequalities and wrinkles. You have now only to raise very carefully one corner of the glass, holding a glass rod at the opposite corner to guide the water in flowing off, and to absorb the remaining water with filter-paper, finally holding the plate over a lamp until it is perfectly dry. The leaf of gold is left as a perfectly smooth film on the glass. Hold it up to the light and notice the deep green colour of the transmitted light. Examine it with a magnifier; many holes will be seen, and much inequality of thickness, but the greater portion of the leaf is a continuous film, even under high magnifying powers.

2. Place a leaf of gold in a test-tube, and pour on it some hydrogen chloride. It will not dissolve in the acid, even on boiling. Treat another leaf of gold with hydrogen nitrate in

a similar way; it will also be found to be unacted upon. Now mix the contents of the two tubes; the metal will at once disappear, and a yellow solution of gold perchloride will be obtained. Pour this into a porcelain dish, add a few more leaves of gold, and a little more hydrogen chloride, and evaporate nearly, but not quite, to dryness on a sand-bath. If you wish to drive off the whole of the excess of acid, the evaporation must be finished on the water-bath, since, if the temperature is raised much above 200°, an insoluble protochloride is formed. For the following experiments a slight excess of acid is of no consequence. Dilute the solution with about 20 c.c. of water, divide it between four test-tubes, and try the following reactions:—

(*a*) Add some solution of hydrogen sulphide. A black precipitate of gold persulphide will be formed, insoluble in hydrogen chloride. Add to the solution in which the precipitate is suspended, ammonia in slight excess, and a few drops of ammonium sulphide. The black precipitate will redissolve, not very readily, but more quickly on boiling; and will be reprecipitated on addition of hydrogen chloride in excess.

(*b*) Add some solution of iron protosulphate. The liquid will rapidly darken, and metallic gold will be precipitated as a brownish powder (the liquid in which the precipitate is suspended having a blue colour by transmitted light), or if the solution is strong, as a spongy mass. If the quantity of precipitate is sufficient, it may be washed by decantation and dried in a watch-glass; if a portion of it be then burnished with the end of a glass rod or of a test-tube, it will acquire the metallic yellow lustre of gold.

(*c*) Add a drop of tin protochloride, which will produce a purple precipitate. The same reaction, it will be remembered, was employed to detect the presence of tin, p. 195.

It is worth while to preserve all waste solutions containing gold in a bottle labelled 'Gold Residues.' The contents of the bottle may be treated in the same way as platinum residues. (See Appendix B.)

PLATINUM.

1. Hold a piece of platinum wire in the hottest part of the blowpipe-flame. It will be found to be quite infusible, unless oxygen gas, instead of common air, be supplied to the blowpipe from a bladder or gas-holder, when the end of the wire will be fused into a bead with slight scintillation.

2. Measure 15 c.c. of strong hydrogen chloride into a flask, add 5 c.c. of strong hydrogen nitrate, drop in a few pieces of thin platinum foil, and heat the flask on the sand-bath. Platinum, like gold, is not soluble either in hydrogen chloride or in hydrogen nitrate, but is readily acted upon by the chlorine evolved during the mutual decomposition of these acids, and dissolves as perchloride. Evaporate the solution in a dish, in the open air if possible, observing the same precautions as in the case of gold, since an insoluble platinum protochloride is formed if the temperature is high. Dilute the solution with water and test separate portions as follows :—

(*a*) Add some solution of hydrogen sulphide, and warm the liquid. A black precipitate of platinum persulphide will be formed, soluble, like gold, in ammonium sulphide, and reprecipitated by hydrogen chloride.

(*b*) Add two or three drops of solution of potassium hydrate. A yellow crystalline precipitate will be formed, which is not an oxide but a double salt containing platinum, potassium, and chlorine.

(*c*) Repeat the last experiment, using ammonium chloride in excess instead of potassium hydrate. A yellow precipitate will be formed in this case, similar in appearance and analogous in composition to the potassium compound. Wait until it has completely subsided, which result is hastened by shaking the mixture and adding a little alcohol. Filter the liquid, and wash the precipitate with water to which has been added one-fourth its bulk of alcohol; then leave it to dry in a warm place, while the other experiments are proceeding. When it is dry, detach it from the filter, place it in a porcelain dish, and heat it strongly over a lamp. All the ammonia and chlorine will be driven off, and a grey spongy mass of metallic platinum will be left. This spongy platinum, as it is called, possesses in a high degree the property of causing certain chemical combinations to take place. Thus if a small strong glass jar be filled with a mixture of oxygen and hydrogen gases, in the proper proportions to form water (see p. 72), and, instead of a lighted match, a small piece of spongy platinum be introduced, the gases will explode. Another method of illustrating this property of platinum, is the following :—Coil a piece of platinum wire two or three times round a few fibres of asbestus, so as to form a small ball. Dip the ball into the precipitated platinum and ammonium chloride while still moist on the filter, and hold it in the flame of the lamp until the salt is decomposed. Prepare some hydrogen according to the directions given in Part II, Sect. **2**, Ex. 2, adapting to the generating flask the glass jet mentioned at p. 74. Allow a stream of gas to escape for at least a minute, in order to expel the air; then hold in front of the jet the platinised asbestus ball. It will become red-hot, and will ignite the gas at the jet. Extinguish the flame and hold the ball some little distance from the jet, in the path of the stream of gas, supporting beyond it a clean glass beaker. The latter will be covered with dew, showing that the platinum has caused the hydrogen to combine with the oxygen of the air, although the combination is not so rapid as to evolve heat enough to inflame the gas. If the ball is held in a stream of ordinary coal gas, combination takes place and the ball becomes hot, but the temperature does not rise high enough to inflame the gas, as in the case of pure hydrogen.

Another instance of chemical combination produced by platinum is the oxidation of alcohol or ether. Pour a few drops of ether into a beaker or wine-glass, and cover the glass partially with a card, to the centre of which is attached the platinised ball (or a coil of platinum wire) previously heated to redness in a lamp, so that it may hang down within the glass. Pungent vapours of aldehyd are at once produced, and this compound is further oxidised to hydrogen acetate.

(*d*) Platinum may be obtained in a state of division still finer than platinum sponge—in particles so fine, in fact, that they cease to reflect light, and form a black impalpable powder. The readiest method of preparing 'platinum black' is to add excess of sodium carbonate to platinum perchloride, and then to boil the liquid with a little sugar. Carbon dioxide is evolved with strong effervescence, and a black powder is formed, which should be washed by decantation successively with dilute alcohol, potassium hydrate, hydrogen chloride, and finally water, and then dried at a gentle heat. This powder is still more effective than spongy platinum in producing chemical combination.

SILVER.

1. Place two or three pieces of silver-leaf in a test-tube (this may be done in the same way as in the case of gold-leaf, p. 196), add a little water, and then a few drops of strong hydrogen nitrate. The metal will readily dissolve, and a clear solution of silver nitrate will be obtained[1]. Add to this solution a drop of hydrogen chloride. A white precipitate of silver chloride will be formed, insoluble even on adding more hydrogen chloride and applying heat. Hydrogen chloride cannot therefore be used to dissolve silver.

2. Place a small silver coin in a flask, pour on it 8 or 10 c.c. of concentrated hydrogen nitrate, and warm the flask very gently on a sand-bath. A strong action will commence, and nitrogen tetroxide will be evolved, the fumes of which should not be allowed to escape into the room. In a short time a clear bluish green solution will be formed, containing

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[1] It must be borne in mind that solutions of silver salts, if allowed to touch the skin or clothes, stain it black. The quickest way of removing these stains is to moisten them first with a solution of potassium iodide, and then with strong solution of potassium cyanide. Great care must, however, be taken in using the latter, as it is very poisonous: all traces of it should be washed away as soon as it has removed the stains.

silver nitrate and copper nitrate, since the coin consists of an alloy of silver with about one-thirteenth of its weight of copper. In order to separate the copper, dilute the solution with four or five times its volume of water, and add hydrogen chloride until no more precipitate is formed. The silver will be completely precipitated as chloride, while the copper chloride, being soluble in water, remains in the liquid. Cork the flask and shake it for a few seconds: the suspended silver chloride will then separate in curdy masses, and the liquid may be readily poured off. The precipitate should next be thoroughly washed on a filter, first with dilute hydrogen nitrate, and then with several changes of water until the liquid which runs through the filter ceases to redden blue litmus-paper. While the last portions of water are draining off, take a strip of sheet zinc about 5 × 8 cm., attach a short piece of platinum wire to one corner, fixing it in a nick cut in the zinc by a strong pair of scissors or a knife, and lay the strip at the bottom of a large evaporating dish[1]. Take the filter containing the silver chloride carefully out of the funnel, place it on a piece of blotting-paper and open it out gently so as not to tear it; then lay it upon the piece of zinc in the dish, and bend over the piece of platinum wire attached to the zinc until its extremity dips into the mass of silver chloride. When this is done, fill the dish about half full of dilute hydrogen sulphate (one volume of acid to seven volumes of water), taking care to pour in the acid very slowly, and near the edge of the dish, so as not to disturb or scatter the silver chloride. A galvanic action will be set up, and the hydrogen which is evolved in contact with the silver chloride will reduce it to the metallic state. The dish should be set aside, and the action allowed to go on until the whole mass has become grey, which may take a quarter of an hour

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[1] It will be advisable, though not absolutely necessary, to cover the zinc with a thin coating of mercury, in order to prevent 'local action' (see p. 233). This may be simply done by laying it in a dish, pouring in sufficient dilute hydrogen sulphate to cover it, and adding 2 or 3 c.c. of solution of mercury perchloride. In two or three minutes a sufficient coating of mercury will be deposited, and the zinc after being washed may be used as above directed.

or so. Finally, take out the filter containing the spongy mass of reduced silver, transfer the latter to a beaker and wash it thoroughly first with dilute hydrogen sulphate, and then with several changes of hot water, to get rid of all traces of zinc sulphate. The finely divided metal will readily dissolve in a little moderately strong hydrogen nitrate, and the solution should be evaporated to the crystallising point, and then left to cool. Square tabular crystals of silver nitrate will be formed, which should be drained in a funnel, washed with a very little water, and dried with blotting-paper.

[Liquids containing silver, such as the mother liquor from the crystals of silver nitrate, and the residues from the following experiments should on no account be thrown away, but reserved in a bottle labelled 'Silver Residues.'

For the method of recovering the silver from these residues, see Appendix B.]

3. Place a small crystal of silver nitrate in a mortar, powder it and mix it with about twice as much sodium carbonate, then heat the mixture on charcoal in the blowpipe-flame. Bright globules of metallic silver will be formed, which remain untarnished in the oxidising flame, and no incrustation is formed on the cooler part of the charcoal.

(For the following experiments a solution of silver nitrate containing 4 grms. of the salt in 25 c.c. of water may be used.)

4. Pour some of the silver nitrate solution into six test-tubes, and test it in the following way :—

(*a*) Add to one portion a few drops of dilute hydrogen chloride. A white curdy precipitate will, as we have seen, be produced, quickly subsiding if shaken, and insoluble in excess of hydrogen nitrate or chloride, or in aqua regia, but soluble in excess of ammonia. The precipitate, if exposed to daylight, soon turns grey, owing to a superficial reduction.

(*b*) Acidify another portion with hydrogen nitrate, and test it with solution of hydrogen sulphide. A black precipitate of silver sulphide will be formed. To the fluid in which the precipitate is suspended add ammonia to alkaline reaction, then ammonium sulphide, and warm the mixture. The precipitate will remain undissolved, thus differing in character from the corresponding compounds of the metals which we have hitherto examined, all of which form combinations with sulphur which dissolve in ammonium sulphide.

(*c*) Add to another portion some hydrogen sulphide, allow the precipitate to subside, pour off the fluid, and boil the precipitate with strong hydrogen nitrate. It will dissolve, leaving only yellow clots of sulphur.

(*d*) To another portion add a few drops of solution of potassium hydrate. A brown precipitate of silver oxide will be formed, insoluble in excess of the precipitant.

(*e*) Repeat the last experiment, using ammonia instead of potassium hydrate. The brown oxide will readily dissolve in excess of ammonia.

(*f*) Add dilute ammonia, drop by drop, to another portion, until the precipitate is partially redissolved. Add a drop of a solution of hydrogen tartrate to the ammoniacal solution, and warm it gently. The liquid will turn black, and the silver will be deposited on the sides of the tube, forming a brilliant mirror-like coating. In order to obtain the best mirror, the solution should be dilute, there should be no excess of ammonia, and the heat should be gentle, and gradually applied.

A watch-glass may be silvered in a similar way, by embedding it in sand, filling it with the solution, and heating the sand gently. A very good convex mirror may be thus obtained.

5. Make a solution of ammonium chloride, containing 1 grm. of the salt in 50 c.c. of water, and filter it into a flat-bottomed plate. Take a piece of fine drawing-paper about 12 cm. square, make a small pencil-mark in one corner, then taking it up by the two opposite corners lower it gently upon the solution in the plate, keeping the marked side downwards, so that the centre touches the liquid first, the two corners being lowered afterwards, in order that no air-bubbles may be retained between the solution and the paper. Allow the paper to float upon the solution for five or ten minutes, then take

it up quickly by one corner, and pin it to a shelf or the back of a chair, to drain and dry. The next stage of the preparation of the paper, since it involves the formation of a compound sensitive to light, must be performed by candle-light, and therefore in a cellar, or a room with closed shutters, or may be deferred until the evening. Dissolve 4 grms. of silver nitrate in 50 c.c. of water, filter the solution into a perfectly clean plate, carry it into a room lighted only by a candle, and float upon it the piece of salted paper (with the marked side downwards), taking particular care to avoid air-bubbles. The silver nitrate in the solution will act upon the ammonium chloride in the paper, and ammonium nitrate and silver chloride will be formed at the surface of the paper. After the lapse of three minutes, raise the paper by one corner and pin it up, as before, to dry in a dark cupboard or drawer. Obtain two pieces of flat window-glass, rather larger than the piece of prepared paper; lay one of them flat on the table in a room lighted by a candle only, or at any rate in the darkest corner of a room in which the blinds are drawn down. Place upon the glass a piece of black velvet or green baize; lay upon the latter the piece of prepared paper, having the marked side upwards; place upon the paper a piece of lace or coarse muslin, or a leaf, or a small engraving (which should be on thin paper), and on the top of all lay the other plate of glass. Bind the whole together by two letter clips or india-rubber bands, one on each side, and bring the extempore printing-frame into full daylight. You will observe the portions of the prepared paper, which are not protected by the lace or the engraving, to pass through shades of red and purple, finally becoming black. When this is the case, remove the apparatus again to the dark room, and take out the prepared paper. The lace, or other opaque object, will be found to have protected the paper from the action of the light, and an image of it will be formed, white on a black ground. Of course, if the paper be now brought out to the daylight, it will blacken all over, and the image will be obliterated. If it be thought worth preserving, it must be soaked in common water to remove all excess of silver nitrate, and then placed in a solution of sodium hyposulphite (containing 25 grms. of the crystallised salt dissolved in 100 c.c. of water). This solution will dissolve away all the unaltered silver chloride, leaving the reduced silver untouched. After the print has remained about ten minutes in the solution it should be removed, and washed in several changes of water; after which it may be brought out to the light without damage. The black portions will have become reddish brown, and will be reduced in intensity, since no toning process has been employed; but the experiment will serve the purpose of illustrating the action of light on silver chloride, and the principles of the art of photography.

MERCURY.

1. Pour a few drops of mercury on a clean plate; if the metal is pure, the drops will preserve their roundness as they roll about; but if it contains lead or other impurity, the drops will become elongated when the plate is inclined, and will move about sluggishly, leaving a tail behind them[1].

2. Place about 2 grms. of mercury in a small dish, and pour upon it about 5 c.c. of hydrogen nitrate diluted with an equal volume of water. Allow the action to go on for ten minutes or so (while you proceed with Experiments 3, 4, and 5), stirring the liquid frequently, but applying no heat. Pour off the

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[1] In order to purify it, pour it into a plate or shallow dish in sufficient quantity to form a thin stratum at the bottom, and pour over it enough dilute hydrogen nitrate (1 vol. of the strong acid to 20 vols. of water) to cover it. Leave it in contact with the metal for three or four hours, stirring it occasionally with a glass rod to expose a fresh surface of the metal to the acid; then pour off the acid, wash the mercury with a stream of water poured from a jug, and finally, after pouring off as much water as possible, absorb the remainder with a clean cloth or blotting-paper. Fit a filter in a funnel, and make a fine hole with a pin at the point, support the funnel over a beaker or bottle, and pour into it the dry mercury. The metal which runs through will be found very nearly pure; it may be further purified by distillation, but, except for special purposes, the gain in purity will scarcely compensate for the trouble and risk.

solution into a test-tube, dilute it with two or three times its volume of water, and label the tube 'Mercury Protonitrate.' Pour upon the remainder of the mercury 3 or 4 c.c. of concentrated hydrogen nitrate, and allow the action to go on until all the metal is dissolved, boiling the liquid and adding a little more hydrogen nitrate if the action becomes slow. Drive off any great excess of acid by evaporation, adding one drop of hydrogen chloride, to convert any remaining protosalt into persalt, then dilute the solution with water, and pour it into a test-tube labelled 'Mercury Pernitrate.'

3. Place a few drops of mercury in a test-tube, and heat it over a lamp, holding the tube nearly horizontal. The metal will boil below a red heat, and, if pure, will wholly volatilise, its vapour condensing in the cooler parts of the tube, and forming a bright metallic ring, which, when looked at through a magnifier, will be seen to consist of minute liquid globules like dew. These, if touched with a glass rod, may be made to run together into one large drop.

4. Place a small crystal of mercury perchloride in a tube, and heat it gently over the lamp. The salt will fuse, and at a rather higher temperature volatilise entirely, forming a white crystalline ring in the cool part of the tube. If the experiment is repeated with mercury protochloride (calomel) a similar result will be obtained, but the substance will volatilise without previous fusion.

5. Mix a little mercury protochloride, or perchloride, with an equal quantity of dry sodium carbonate in a mortar, introduce it into a bulb-tube (p. 11), in quantity just sufficient to fill the bulb, and cover it with a thin layer of the dry sodium carbonate. Wipe the tube clean with a twisted strip of filter-paper, and heat it very gently over the lamp. At first, if the substances are not carefully dried, some moisture will be given off, which must be absorbed by strips of filter-paper, before a stronger heat is applied; otherwise it will be difficult to obtain a well-defined sublimate. When no more water condenses in the tube, increase the heat, until the bulb is nearly red-hot. A metallic mirror will form in the tube which, when examined

with a magnifying glass, will be seen to consist of small round globules of metal, very different in appearance to the crystalline crust of arsenic, p. 183. To remove any doubt as to the character of the mirror, introduce a thin slip of wood (e. g. the end of a match) into the tube, and employ it to scrape the sublimate from the sides of the tube. The small globules will run together into one large globule, which from its metallic lustre and fluidity can only be mercury[1].

6. Apply the following tests to each of the solutions which you have obtained in Experiment 2, and which will serve as examples of the two series of salts formed by the metal mercury.

1. Mercury Protonitrate.

(*a*) Pour a few drops of the solution into a test-tube, and add dilute hydrogen chloride, drop by drop. A white precipitate of mercury protochloride will be formed, which will not dissolve in excess of the precipitant. Pour some of the fluid in which the precipitate is suspended into another tube, add a few drops of hydrogen nitrate, and boil. The mercury protochloride will be converted into mercury perchloride, which will dissolve. To the remainder of the precipitate add ammonia in slight excess. The precipitate will turn black.

(*b*) Test another portion with solution of hydrogen sulphide. A black precipitate of mercury protosulphide will be formed. Divide the contents of the test-tube into two portions; render one portion alkaline with ammonia, add some ammonium sulphide, and warm the mixture; the precipitate will remain undissolved. Allow the precipitate in the other portion to subside; pour off the fluid, add some strong hydrogen nitrate and boil. The mercury protosulphide will be decomposed, part dissolving as nitrate, part remaining as persulphide; which latter will be readily decomposed on the addition of a few drops of hydrogen chloride.

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[1] Mercury iodide is the only commonly occurring mercury compound which under the above conditions volatilises unreduced, in spite of the superposed layer of sodium carbonate.

(*c*) Add to a third portion some solution of potassium hydrate. A black precipitate of mercury protoxide will be formed, insoluble in excess.

(*d*) Add to another portion some tin protochloride in excess. A grey precipitate of metallic mercury will be produced. If this be allowed to subside, the fluid poured off, and the precipitate, after being once washed by decantation to remove traces of nitrate, warmed with a little strong hydrogen chloride, the finely divided particles will unite into a globule of mercury.

(*e*) Place a drop of the solution on a clean surface of copper; allow it to remain for half a minute, and then wash it off. A dull white stain will be left on the copper, which when rubbed with a cloth will become bright and silvery. The mercury salt has been decomposed, copper nitrate being formed, while the mercury is deposited on the more electro-negative metal, the copper, with which it forms an alloy or amalgam.

2. Mercury Pernitrate.

(*a*) No precipitate will be formed.

(*b*) If the hydrogen sulphide is added drop by drop, and the liquid shaken, a precipitate will be produced which is at first white, but on further addition of hydrogen sulphide becomes yellow, brown, and finally black. The cause of this characteristic reaction is, that a combination of mercury persulphide and nitrate is first thrown down, which is converted entirely into mercury persulphide by excess of the precipitant. The mercury persulphide, like the protosulphide, will be found insoluble in ammonium sulphide and also in hydrogen nitrate, but decomposable by aqua regia.

(*c*) A yellow precipitate of mercury peroxide will be formed.

(*d*) A white precipitate of mercury protochloride will be produced by the addition of a drop of tin protochloride, which will become grey on further addition of the reagent, owing to its reduction to metallic mercury.

(*e*) The result will be the same as in the case of the protosalt.

7. Add ammonia to a solution of mercury perchloride. A white precipitate will be formed, which has the composition of ammonium chloride in which part of the hydrogen is replaced by the metal mercury[1].

8. Mercury iodide has been already prepared and examined (p. 167). It may be added that, to obtain good crystals for the microscope, it is generally necessary to resublime the crystals first obtained. To do this, place over the slip of glass on which the crystals have been deposited, another similar slip of glass, separating the two by strips of card placed between them at each end. Hold the plates in this position between the finger and thumb, and heat both of them over a spirit lamp. When they are pretty hot, apply a higher temperature to the one on which the mercury iodide is deposited, waving the lamp-flame to and fro under it. The sublimation thus takes place slowly, and large crystals are formed.

LEAD.

1. Cut off a small piece of sheet lead with a strong knife, and hammer it thin on an anvil, noticing its malleability, softness, and want of elasticity. Fold up the thin sheet thus obtained, place it in a flask or beaker, pour on it some strong hydrogen nitrate diluted with an equal volume of water, and heat gently on the sand-bath. The metal will readily dissolve, orange vapours of nitrogen tetroxide being formed. Keep the solution of lead nitrate, after driving off any great excess of acid by evaporation, to be tested as directed in Experiment 4.

2. Mix a little lead acetate with sodium carbonate, and heat the mixture on charcoal before the blowpipe. A metallic globule will be obtained, which is malleable; and a light yellow

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[1] $2\,H_3N + HgCl_2 = (H_4N)\,Cl + (Hg\,H_2N)\,Cl.$

incrustation will be formed round the margin of the hole in the charcoal.

3. Pour some dilute hydrogen nitrate on a little red lead in a test-tube, and warm the mixture. A chocolate-coloured powder will remain undissolved, which is lead dioxide.

4. Add a few drops of dilute hydrogen chloride to some of the solution of lead nitrate obtained in Experiment 1. A white crystalline precipitate will be formed, insoluble in excess of the acid. Divide the fluid containing the precipitate in suspension into three portions, and test them as follows:—

(*a*) Add to the first portion three or four times its volume of water. The precipitate will gradually redissolve.

(*b*) Heat the second portion to boiling. It will in this case also redissolve, but will be reprecipitated as the solution cools, in snow-like flakes, which, when examined by a magnifier, will be seen to consist of groups of slender prisms.

(*c*) To the last portion add ammonia. The precipitate will undergo no alteration. (Compare Experiment 1 *a*, p. 207.)

5. Test another portion of the solution of lead nitrate with solution of hydrogen sulphide. A black precipitate will be produced, insoluble in hydrogen chloride but decomposed on being boiled with excess of hydrogen nitrate. It will be remembered that this property of lead, of forming a black compound with sulphur, furnished us with a very delicate test of the presence of a sulphide (p. 172).

6. Add to another portion of lead nitrate a few drops of solution of potassium hydrate. A white precipitate of lead hydrate will be formed, which will redissolve in excess of the precipitant.

7. Test another portion of lead nitrate with dilute hydrogen sulphate. A white precipitate will be formed, insoluble in excess of the acid, slightly soluble in strong hydrogen chloride, but readily decomposed and dissolved if warmed with solution of ammonium acetate[1].

8. Add a few drops of solution of potassium iodide to

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[1] This may be made by taking 2 or 3 c.c. of hydrogen acetate, and adding nearly sufficient solution of ammonia to neutralise it.

another portion of lead nitrate (which should be only slightly acid). A yellow precipitate will be produced, which will dissolve slowly if the solution is diluted and warmed, and will be reprecipitated as the solution cools in golden-yellow spangles.

9. Add to another portion of lead nitrate a drop of solution of potassium chromate. A yellow precipitate will be formed. Divide the liquid into two portions: to the first add some dilute hydrogen nitrate; the precipitate will remain undissolved. To the second portion add some solution of potassium hydrate, which will readily redissolve the precipitate.

BISMUTH.

1. Place a small fragment of bismuth on an anvil, and strike it with a hammer. It will not spread out, like lead, into a thin plate, but will be crushed, like antimony, into a crystalline powder. Dissolve the metal in hydrogen nitrate, as directed in the case of lead. It will be found to dissolve easily, and the solution of bismuth nitrate may be reserved for testing.

2. Weigh out 4 grms. of bismuth, 2 grms. of lead, and 2 grms. of tin. Melt the bismuth in a clean iron spoon, and add to it the lead and the tin, stirring the melted alloy with a glass rod or piece of iron wire. Pour it out on a clean iron plate or on a tile and allow it to cool. The melting-points of bismuth, lead, and tin, are all above 230°, but this alloy is so fusible that if a piece of it be cut off and thrown into boiling water in a test-tube, it will readily melt.

3. The blowpipe reaction of bismuth will be found at p. 138.

4. Pour a little of the solution of bismuth nitrate into a test-tube and add four or five times its volume of water. The solution will gradually become cloudy and deposit a precipitate of bismuth oxynitrate, which will dissolve in excess of hydrogen chloride, but is insoluble in hydrogen tartrate. (Compare the reaction of antimony, p. 192.) If a drop of hydrogen chloride

is added to the solution of bismuth nitrate and the mixture then diluted with water, a precipitate is formed far more readily than in the case of the nitrate, since bismuth chloride is very easily decomposed by water.

The best method of applying this test is, to evaporate a few drops of the solution of bismuth salt to dryness in a watch-glass, to dissolve the residue in a drop of moderately dilute hydrogen chloride, and then, placing the watch-glass on a black surface, to fill it up with water from a wash-bottle. A very slight turbidity may thus be rendered evident.

5. Acidify another portion of bismuth nitrate with hydrogen chloride, adding the acid until any precipitate which may be formed is redissolved, and test the solution with hydrogen sulphide. A black precipitate of bismuth trisulphide will be formed, insoluble, after neutralisation of the solution, in ammonium sulphide, but decomposed by strong boiling hydrogen nitrate.

6. Add ammonia to another portion of the solution of bismuth nitrate until a permanent precipitate is produced, then add a drop of solution of potassium chromate. A yellow precipitate of bismuth chromate will be formed, which will dissolve in dilute hydrogen nitrate, but not in solution of potassium hydrate. (Compare the reaction of lead chromate, p. 211.)

COPPER.

1. Clean a strip of sheet copper with emery-paper, heat it to redness in the flame of a Bunsen's burner, and hold it in the outer part of the flame for one or two minutes. The metal will combine with the oxygen of the air, and be covered with a film of oxide. If it be now plunged into cold water, the oxide will detach itself in scales (since it does not contract, as the temperature falls, in the same degree as the metal), and the surface of the metal will show the characteristic red colour of copper.

2. Make a small quantity of a mixture of equal parts of

copper sulphate and sodium carbonate, and heat it on charcoal before the blowpipe-flame. You will obtain small grains of metallic copper, but the heat of the blowpipe-flame will be scarcely sufficient to fuse these into one globule. They will be better seen if the fused mass is placed in a mortar and washed with water until the soluble salts are dissolved; the red metallic particles may then be readily distinguished with a magnifier.

3. Make a borax bead, add to it a trace of copper sulphate, and heat it in the oxidising flame of the blowpipe. The bead will be coloured green while hot, becoming blue as it cools. After being heated in the reducing flame, it will appear red by reflected light, owing to the presence of suspended particles of copper.

4. Dissolve 1.5 grms. of fine copper wire in hydrogen nitrate diluted with an equal volume of water, heating the flask gently if the action becomes slow. Red vapours of nitrogen tetroxide will be seen above the liquid (p. 84), and a blue solution of copper nitrate will be obtained. Evaporate this solution nearly to dryness in a dish, to drive off any excess of hydrogen nitrate, dilute it with water to 20 c.c., and examine separate portions of the solution with the following tests:—

(*a*) Place a piece of platinum foil in an evaporating dish, put on it a fragment of granulated zinc, and pour into the dish some dilute hydrogen sulphate. If a drop of the solution of copper nitrate be now added, the surface of the platinum will be coated with metallic copper[1].

(*b*) Pour a little of the solution into a test-tube, and dip into it a piece of clean iron wire or the blade of a knife. Metallic copper will be thrown on the surface of the iron, while the latter is slowly dissolved, taking the place of the copper in the solution.

(*c*) Acidify another portion of the solution with hydrogen chloride, and add solution of hydrogen sulphide. A black

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[1] After the experiment the platinum should be warmed with some dilute hydrogen nitrate, which will dissolve the coating of copper, without injury to the platinum.

precipitate will be formed, consisting of copper sulphide. Allow this to subside, pour off the clear fluid, and wash the precipitate once or twice by decantation. Divide it into two portions: to one of these add some ammonium sulphide, and warm it gently; the precipitate will not dissolve. Boil the other portion with strong hydrogen nitrate, which will readily decompose and dissolve it, with separation of sulphur.

(*d*) To another portion add a few drops of solution of sodium hydrate. A light blue precipitate of copper hydrate will be formed, which will not dissolve in excess of the precipitant, and will turn black when the solution is heated to the boiling-point, owing to its conversion into copper oxide.

(*e*) To another portion add one drop of solution of ammonia. A light blue precipitate will be formed, as in the last experiment, but will readily dissolve on addition of a few drops more of ammonia, and a deep blue solution will be obtained.

(*f*) Test another portion with a drop of a solution of potassium ferrocyanide. A reddish brown precipitate of copper ferrocyanide will be produced.

5. Dissolve a small lump of grape sugar in 2 or 3 c.c. of water, filter the liquid if necessary, and add to it a small portion of the solution of copper nitrate. If solution of potassium hydrate be now added, no precipitate of copper hydrate will be formed, or if formed (owing to the grape sugar not being present in sufficient quantity) it will be redissolved on addition of more potassium hydrate. But if the mixture is heated to the boiling-point, a yellow precipitate of copper protohydrate is obtained, which quickly changes to red copper protoxide.

CADMIUM.

1. Mix a small quantity of cadmium sulphate with an equal amount of sodium carbonate, and heat it on charcoal before the blowpipe. No metallic globule will be obtained, owing to the volatility of cadmium, but the surface of the charcoal will

be covered with a characteristic reddish brown coating of cadmium oxide.

2. Dissolve 3 grms. of cadmium sulphate in 12 c.c. of water, and test separate portions of the solution as follows:—

(*a*) Acidify one portion with hydrogen chloride, and add solution of hydrogen sulphide. A yellow precipitate of cadmium sulphide will be produced. Add to the solution in which the precipitate is suspended ammonia in slight excess, then a few drops of ammonium sulphide, and warm the mixture. The precipitate will remain unaltered, differing in this respect from the two other yellow sulphides, those of arsenic and tin, which are dissolved under the same circumstances.

(*b*) Add a drop of ammonia to another portion. A white precipitate of cadmium hydrate will be formed, which will readily dissolve on addition of more ammonia.

IRON.

1. Clean a small strip of sheet iron with a file or with emery-paper, until it shows a bright metallic surface; then hold it with a pair of pliers or crucible tongs in the flame of a Bunsen's burner or spirit lamp. The surface will soon lose its lustre, becoming in succession light yellow, orange, blue, and finally dark grey, owing to the formation of an extremely thin film of oxide. If the strip of iron be maintained at a red heat in an ordinary fire for about ten minutes, the film of oxide will increase in thickness, and will be detached in the form of black scales when the iron is quenched in cold water. These scales consist of the same iron oxide as that which was formed when the watch-spring was burnt in oxygen gas (p. 66). When a magnet is brought near them they will be attracted by it; and this oxide, from this property and from its identity in composition with the native loadstone, is called the magnetic oxide.

2. An experiment illustrating the combination of sulphur with iron is given at p. 172.

3. Take a moderately stout steel knitting-needle; notice that you can file it flat, and bend it into a loop with the pliers, without much difficulty, although it is harder and stiffer than iron wire of the same thickness. Heat 2 or 3 cm. at the end of it to redness in the lamp-flame, and while it is red-hot, plunge it quickly into a beaker of cold water. If you now attempt to file it, you will find that the file glides over the surface without cutting into it, and, instead of bending, it will break short off, like a piece of glass. The sudden cooling has rendered it extremely hard and brittle; but if it be again heated to redness and allowed to cool very slowly it will be found to have regained its former properties of softness and pliability. Between these two extremes any intermediate degree of hardness may be imparted to steel by the process of tempering. To illustrate this process, you may take a piece of thick watch-spring, straighten it as directed, p. 65, harden it in the manner above mentioned, and then lay it flat upon a piece of board and rub it with a piece of moistened whetstone (or with a bit of wood dipped in fine emery made into a paste with water) until it becomes quite bright. Now lay it upon a strip of sheet iron and heat it again very carefully, holding it at some distance above the flame. The surface of the steel will soon begin to show the same succession of tints as the iron in Experiment 1, passing from yellow to orange and blue. When it has just reached this latter tint, plunge it into cold water, to stop any further action of the heat. You will now find that it has regained the properties of the original watch-spring; that it is stiff and elastic, and that its surface can be filed away, though with difficulty, and much to the damage of the file. We have in the various colours of the film of oxide a guide to the amount of heat communicated to the hardened steel, which becomes softer in proportion as it is more strongly heated. If the application of heat had been stopped when the surface became straw-yellow, the hardness would have been less reduced, and the temper would have been such as is required for razors and tools for cutting steel.

4. Obtain a thin piece of cast-iron (a fragment of a broken

saucepan will do), place it on an anvil and strike it forcibly with a hammer. The cast-iron, unlike wrought-iron, will break into pieces, the surfaces of the fracture showing a crystalline structure, which should be compared with the fibrous structure of iron wire observed when a thick piece of the latter is placed in a vice and broken by being repeatedly bent in opposite directions. Place 6 or 8 grms. of cast-iron (crushed into small fragments) in a large test-tube, and add 8 or 10 c.c. of dilute hydrogen sulphate. An action will begin almost immediately, hydrogen gas being given off with effervescence. Its presence may be recognised by holding a lighted match to the mouth of the tube, when the gas will take fire with a slight explosion. Observe the strong, peculiar smell of the gas, which is owing to the presence of small quantities of some volatile compounds of carbon and hydrogen. Leave the tube in a warm place for an hour or two, occasionally adding a few drops of strong hydrogen sulphate if the action becomes slow. After all the heavy metallic particles have disappeared, there will still remain floating in the liquid some black flakes, which consist of graphite. Filter the solution into an evaporating dish and concentrate it until a drop placed on a watch-glass deposits crystals on cooling; then cover the dish with a piece of filter-paper, and leave it undisturbed to cool. Light green crystals of iron protosulphate (ferrous sulphate) will be formed, which may be placed in a funnel to drain, washed from the mother liquor by a little distilled water, dried on blotting-paper, and kept in a closely-stoppered bottle.

5. Take a piece of fine iron wire (the finest is the purest) about 20 cm. in length; clean it from rust, if necessary, by drawing it between folds of emery-paper, form it into a small, close coil, and place it in a test-tube to which you have fitted a cork in the side of which a small nick has been cut to allow gas to escape. Pour on the iron wire about 9 or 10 c.c. of dilute hydrogen sulphate, cork the test-tube and leave it in the stand, while you proceed with the two following experiments:—

6. Place a crystal of iron protosulphate in a small bulb-tube, and heat it strongly in the flame of a Bunsen's burner. The

salt will first melt in its water of crystallisation, then it will turn white, give off aqueous vapours, and lastly, as the temperature rises nearly to redness, it will decompose, giving off vapours of sulphur dioxide and trioxide, which will redden litmus-paper held at the mouth of the tube, and a red powder will be left behind, which consists of iron peroxide, or 'rouge.'

7. Crush a crystal of iron protosulphate to powder in a mortar. Form a bead of borax in a loop of platinum wire (see p. 135), and while the bead is still hot bring it in contact with the powdered iron salt, so that it may take up a very minute quantity of the latter. Heat the bead again, holding it near the tip of the blue flame until the iron salt is dissolved, then bring it into the oxidising flame and hold it there steadily for a few seconds. On withdrawing it from the flame, you will find that (if the right proportion of iron has been taken) it is orange-coloured while hot, becoming light yellow as it cools. In the next place, heat it in the reducing flame for half a minute; its colour will now be found to have changed to a dull green (like that of bottle-glass), which becomes paler on cooling.

8. The iron wire which you placed in dilute hydrogen sulphate a short time ago will now have dissolved in sufficient quantity to enable you to examine the properties of iron salts with the solution. Iron forms at least two series of salts:

(*a*) The proto- or ferrous salts.

(*b*) The per- or ferric salts.

The former class are converted into the latter class of salts, when their solutions are placed in contact with air, with such extreme facility that special precautions are necessary in order to obtain them in a pure state. You have in the test-tube in which iron is being acted upon by hydrogen sulphate, the metal being in excess, and air being excluded by the loosely-fitting cork, a solution of the protosulphate alone. Pour off one half of the solution into a porcelain dish, replacing the cork as quickly as possible; heat the dish gently over the lamp and add dilute hydrogen nitrate, drop by drop, as long as a transient brown colour is produced in the solution. Leave the dish exposed to a gentle heat (in order to drive off any excess of hydrogen nitrate) for some minutes, while you examine the properties of iron protosalts in the remainder of the solution, as follows:—

(*a*) Pour into a test-tube about 2 c.c. of solution of hydrogen sulphide, and add about two drops of the solution of iron protosulphate[1]. No change of colour or precipitation will occur.

(*b*) To the contents of the same test-tube add solution of ammonia to alkaline reaction. A black precipitate of iron protosulphide will be formed, which will readily dissolve on addition of a drop or two of dilute hydrogen chloride.

(*c*) Pour into another test-tube a few drops of solution of potassium hydrate, add a little water, and then a drop or two of the solution of iron protosulphate. A greyish white flocculent precipitate of iron protohydrate will be formed, which, if the tube be shaken for half a minute, will rapidly become darker, and be finally converted into a reddish brown perhydrate, by absorbing oxygen from the air.

(*d*) Pour into another test-tube a little solution of potassium ferrocyanide, add water, and then a drop of the solution of iron protosulphate. A precipitate will be formed which is at the first moment nearly white, but rapidly changes to deeper and deeper shades of blue, when the test-tube is shaken, or when the solution is poured backwards and forwards several times from one test-tube to another.

(*e*) Repeat the last experiment, using potassium ferricyanide instead of ferrocyanide. A deep blue precipitate will be at once formed, which will undergo no further change in the air.

(*f*) Repeat the last experiment using potassium sulphocyanide instead of ferricyanide. No change will take place at first in the solution, but on shaking the test-tube the liquid will become light red, and eventually of the colour of dark sherry. The appearance of this red colour is an extremely delicate test for iron persalts.

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[1] Remember always to replace the cork in the test-tube as quickly as possible, in order to prevent access of air.

9. Pour into a test-tube the solution of iron salt which has been heated with hydrogen nitrate; notice that it has a yellow colour, while the original solution was nearly colourless; add a little water, and examine portions of the solution with the tests given in the last experiment.

(*a*) The liquid will become milky, owing to precipitation of sulphur, and will lose its red colour, the persalt being reduced to protosalt.

(*b*) On addition of ammonia a black precipitate of iron protosulphide will be formed, soluble in dilute hydrogen chloride.

(*c*) A reddish brown precipitate of iron perhydrate will be formed, which will undergo no change in the air.

(*d*) A deep blue precipitate (Prussian blue) will be formed.

(*e*) No precipitate will be formed, but the colour of the solution will become brown (or green if the solution of potassium ferricyanide has been made for some time, a small quantity of ferrocyanide being formed in such a case).

(*f*) No precipitate will be formed, but the colour of the solution will become intensely red.

10. We have seen that iron protosalts can be converted into persalts by the action of hydrogen nitrate. The same change may be effected by other oxidising agents. Pour the remainder of the solution of iron protosulphate into a test-tube, and add a few drops of solution of chlorine. The solution will now give the characteristic reactions of iron persalts, viz. a deep blue precipitate with potassium ferrocyanide, and an intensely red liquid with potassium sulphocyanide.

11. We have seen from Experiment 7 *a*, that hydrogen sulphide is decomposed by iron persalts, the protosalt being formed. Other reducing agents may be employed to produce the same effect. Hydrogen in the nascent state is one of the best for the purpose. Add a few drops of dilute hydrogen sulphate to the remainder of the solution of iron persulphate, place a fragment of granulated zinc in the solution, and close the tube with a cork having a nick cut in its side. Hydrogen will be evolved by the action of the zinc on the acid, and th

solution will gradually lose its colour, and will give the reactions of iron protosalts, viz. a deep blue precipitate with potassium ferricyanide, and a colourless solution with potassium sulphocyanide[1].

12. Dissolve a crystal of iron protosulphate in a little water, and leave it exposed to the air for a short time; filter the solution if necessary, and add one or two drops of infusion of nut-galls, or solution of hydrogen gallate. A black precipitate will be formed, which will, if a little gum-water be added, remain for a long time suspended in the liquid. Ordinary writing ink is thus made.

ALUMINIUM.

1. If you can obtain a few pieces of leaf-aluminium you may try the following experiment, which illustrates its combination with oxygen :—

Take a cylindrical gas jar, about 18 cm. in height, place in it two or three leaves of aluminium, cover it with a card, and fill it with oxygen by displacement, passing the gas through a bent tube which reaches to the bottom of the jar. When it is full of the gas, take in the crucible tongs a small bit of tinder or of a cigar-light (p. 65), light it, and (having removed the card) drop it upon the leaves of aluminium. They will burn with a brilliant flash, and a white powder (aluminium oxide) will be formed.

2. Place a small crystal of aluminium and ammonium sulphate (ammonia alum) in a cavity cut in a piece of charcoal, and heat it in the hottest part of the blowpipe-flame. The salt will at first swell up like borax, white fumes of ammonium sulphate and hydrogen sulphate will then be given off, and finally a white, infusible, incandescent residue of aluminium

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[1] The presence of the zinc salt will not interfere with these reactions, if the solution contains hydrogen sulphate.

oxide will be left on the charcoal. Allow this to cool, then moisten it with a drop of solution of cobalt nitrate (transferred from the bottle on a glass rod), and ignite it again for several seconds. The mass, when cool, will be found to have acquired a bright blue colour[1].

3. Dissolve 4 grms. of re-crystallised alum (p. 44) in 40 c.c. of water[2], and use the solution in the following experiments (the presence of ammonium in the alum will not interfere with the tests):—

Pour a few drops of the solution into a test-tube, dilute with water, and add solution of potassium hydrate, drop by drop. A white gelatinous precipitate of aluminium hydrate will be formed at first, but on addition of more potassium hydrate it will readily redissolve. Divide the solution into two parts:

(*a*) To one portion add one or two drops of solution of hydrogen sulphide. No precipitate will be produced, since aluminium sulphydrate is not formed in presence of water.

(*b*) To the other add about half its volume of solution of ammonium chloride, and warm the mixture; aluminium hydrate will be precipitated.

4. To another portion of the solution of alum add a few drops of solution of ammonium sulphide. A gelatinous precipitate will be formed, consisting of aluminium hydrate and not aluminium sulphydrate.

5. Add some solution of cochineal to another portion of the solution of alum; then add enough ammonia to precipitate all the aluminium as hydrate, and filter the liquid. The filtrate will be quite colourless, the colouring matter of the cochineal being retained on the filter in combination with the aluminium hydrate, forming one of the 'lakes' used in painting. This

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[1] This reaction, however, is not absolutely characteristic of aluminium, since some silicates, calcium phosphate, and one or two other substances behave in a similar way.

[2] The proportions here given, and also in the case of chromium (p. 223), are sufficient to form a solution of one-fifth the strength of the normal solution (see p. 146), since the weight of the molecule of alum, expressed in grammes, cannot be contained in a litre of solution at the ordinary temperature.

affinity of aluminium hydrate for colouring matters is of great importance for dyeing purposes, and the use of aluminium salts as mordants may be illustrated in the following way:—

6. Dissolve 3 grms. of lead acetate in about 10 c.c. of water, adding a few drops of hydrogen acetate if the solution is turbid. Measure 30 c.c. of the solution of alum into a beaker and add to it the solution of lead acetate. Warm the mixture and let it stand for a short time to allow the precipitate of lead sulphate to subside, then filter it. The filtrate contains aluminium acetate together with a small quantity of undecomposed alum, and is the solution which is usually employed in dyeing. Dip one half of a strip of white calico in the solution, and hang it up to dry in a warm place, until the next day. During the process of drying, the aluminium acetate is decomposed, hydrogen acetate being given off, and aluminium hydrate being deposited in the fibres of the calico. Warm some moderately strong solution of cochineal or logwood in an evaporating dish, immerse the strip of calico, and heat the liquid to boiling for about ten minutes; then take out the calico, and wash it thoroughly in several changes of water. The part of it which was soaked in the 'mordant' solution will be found to be permanently dyed, while all the colouring matter will be washed out of the other portion.

CHROMIUM.

1. Make a borax bead (p. 135), add to it a very minute quantity of potassium chromate, and heat it in the oxidising flame. The colour of the bead, on cooling, will be emerald green, and will not be altered in the reducing flame.

2. Dissolve 10 grms. of potassium chromate in 50 c.c. of water, and test portions of the solution as follows:—

(*a*) Add to a portion a drop of solution of silver nitrate.

A purple-red precipitate of silver chromate will be formed, which will readily dissolve on addition of a few drops of hydrogen nitrate.

(*b*) Add to another portion a drop of solution of lead acetate. A bright yellow precipitate of lead chromate will be formed. Pour off a portion of the liquid in which the precipitate is suspended into another test-tube, add some solution of potassium hydrate, and warm the mixture; the lead chromate will dissolve.

(*c*) Place a small fragment of barium peroxide[1], about as large as a pea, in a mortar, pour upon it 3 or 4 c.c. of dilute hydrogen chloride, and grind them together until the peroxide has entirely dissolved. A slight effervescence will probably be noticed, owing to the escape of oxygen, and a solution containing barium chloride and hydrogen peroxide will be obtained.

Place about one-third of this solution in a test-tube, and add one or two drops of the solution of potassium chromate acidified with hydrogen chloride. The colour of the solution will change to a deep blue, but in a second or two this will disappear, while oxygen is given off with effervescence, and a pale green solution will be obtained. These changes of colour are due to the formation of a higher chromium oxide, which is very unstable, and decomposes, when hydrogen chloride is present, into oxygen and chromium chloride. This blue chromium oxide (of which the constitution is uncertain) is much more stable when dissolved in ether. To illustrate this, add to another portion of the solution of potassium chromate sufficient ether to form a stratum about 1 cm. in depth; pour in some more of the solution of hydrogen peroxide, close the mouth of the test-tube with the thumb and shake the mixture. If it is now allowed to remain undisturbed for a minute, the ether will rise to the surface, forming a magnificent blue stratum, while the liquid below is nearly colourless.

(*d*) Acidify another portion of the solution of potassium

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[1] For the method of making this substance, see p. 235.

chromate with hydrogen chloride, and add excess of solution of hydrogen sulphide. A white precipitate of sulphur will be formed, and the red colour of the solution will change to pale green, owing to the reduction of the chromate, and formation of a chromium salt.

(*e*) A similar reduction of chromates is effected by other oxidisable substances, e. g. alcohol. Add to another portion (about 10 c.c.) of the solution of potassium chromate, a few drops of dilute hydrogen chloride, and a little alcohol, and warm the mixture. Vapours of aldehyde will be evolved, which may be recognised by their peculiar pungent smell, and a green solution of chromium chloride will be obtained. Pour the liquid into a dish and evaporate it on the sand-bath until all the excess of hydrogen chloride and of alcohol is driven off; then dilute it, if necessary, with water, and reserve it for testing in Experiment 4, p. 226.

3. Dissolve 4 grms. of potassium dichromate in about 30 c.c. of hot water in a flask, and set it aside to cool, when a portion of the salt will be deposited. Pour off the cold saturated solution into a small beaker, and place the latter in a dish of cold water. Measure 25 c.c. of strong hydrogen sulphate, and add it to the solution of potassium dichromate. Chromium trioxide will be deposited in crimson needles, since, while it is soluble in strong hydrogen sulphate and in water, it is insoluble in a mixture of the two made in the proportions above indicated. Cover the beaker with a watch-glass, and set it aside until the precipitate has subsided; then pour off the liquid carefully, and scrape out the crystals with a platinum spatula or glass rod upon a dry porous tile or clean brick. Cover them with an evaporating dish, and leave them until the liquid has been absorbed as far as possible by the brick. Ten minutes will generally be sufficient for this purpose, and as the chromium trioxide is very deliquescent it should be protected as far as possible from the air, and not left longer than is necessary on the brick. When it is dry, the following experiments may be tried with portions of it; the remainder, if the crystals are good, may be kept as a specimen in a small

stoppered bottle, or in a stout test-tube, hermetically sealed, p. 35.

(*a*) Heat a portion of the chromium trioxide on a piece of platinum foil over a lamp. The substance will melt, and on being further heated will decompose with incandescence, leaving a green residue of chromium sesquioxide, while oxygen is given off.

(*b*) Pour one or two drops of strong alcohol into a bottle, shake it up so as to diffuse the vapour; then throw into the bottle some of the chromium trioxide. The latter will be reduced to sesquioxide, the action being generally so violent as to set the alcohol on fire.

4. Dissolve 2 grms. of chrome alum in 20 c.c. of water[1], and examine its properties as follows:—

(*a*) Add to a portion of the solution a drop of ammonium sulphide. A light bluish green, gelatinous precipitate of chromium hydrate, and not chromium sulphydrate, will be formed.

(*b*) To another portion add, drop by drop, solution of potassium hydrate, shaking the mixture after each addition. The chromium hydrate, which is at first precipitated, will readily dissolve in excess of potassium hydrate to a clear green fluid. Boil this fluid for two or three minutes; it will become turbid, and eventually the whole of the chromium hydrate will be reprecipitated, in a form in which it is no longer soluble in potassium hydrate.

5. Reduce a small crystal of chrome alum to powder in a mortar, and mix with it an equal quantity of sodium carbonate and about one-fourth the quantity of potassium nitrate. Fuse the mixture, over the gas blowpipe, on a piece of porcelain (or on platinum foil, since the proportion of nitrate is small), and boil the yellow fused mass, when cold, with a little water. You will obtain a yellow solution containing a chromate, since the chromium has been oxidised by the potassium nitrate. To prove the presence of a chromate, apply to the solution the

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[1] Instead of chrome alum, the solution of chromium chloride, obtained in Experiment 2 *e*, may be used.

tests given in Experiment 2 *a* and *b*, remembering to decompose the excess of sodium carbonate which is present, by adding a little hydrogen acetate until there is no further effervescence, and boiling the solution.

COBALT.

1. Make a borax bead, dip it into a solution of cobalt nitrate, and heat it in the oxidising flame. A deep blue bead will be obtained, which will undergo no alteration in colour when exposed to the reducing flame.

2. Dissolve 5.8 grms. of crystallised cobalt nitrate in 20 c.c. of water, and test portions of the solution as follows:—

(*a*) Add to the first portion four or five drops of dilute hydrogen chloride, and test it with solution of hydrogen sulphide. No precipitate will be produced, but on the addition of some solution of ammonia a black precipitate of cobalt sulphide will be formed.

(*b*) Test another portion of the solution with solution of ammonium sulphide, and warm the mixture, adding more ammonium sulphide as long as it produces a precipitate. The black precipitate of cobalt sulphide will soon separate in flakes. Filter the solution, and wash the precipitate twice, on the filter, proceeding, meanwhile, with other experiments: then make a hole in the filter with a glass rod, and wash the precipitate into a test-tube. Allow it to subside, and pour off the greater part of the supernatant fluid; then add six or eight drops of dilute hydrogen chloride, and warm the mixture. The cobalt sulphide will be found to be soluble only with great difficulty in the acid, although the addition of a small quantity of the latter was sufficient in the previous experiment to prevent the precipitation by hydrogen sulphide.

You will observe that in this, as in many other cases, it is much easier to prevent precipitation than to dissolve a precipitate when formed.

(*c*) Test another portion with solution of potassium hydrate. A light blue precipitate, consisting of a basic cobalt salt, is formed, which does not dissolve in excess of the precipitant, and becomes reddish brown when the solution is boiled, owing to its conversion into cobalt hydrate.

(*d*) Test another portion with solution of ammonia. A light blue precipitate is formed as in the last experiment, but it will redissolve in excess of the precipitant, a reddish brown solution being formed.

(*e*) Add to another portion solution of potassium cyanide, drop by drop. A light red precipitate of cobalt cyanide will be at first formed, but will readily dissolve in excess of potassium cyanide. Divide the solution into two parts. To the one add excess of dilute hydrogen chloride; cobalt cyanide will be precipitated. To the other portion add a few more drops of the solution of potassium cyanide, and then one or two drops of dilute hydrogen chloride, but not sufficient to produce a permanent precipitate: boil the solution for two or three minutes, allow it to cool, and then add excess of dilute hydrogen chloride. No precipitate will, in this case, be produced; the reason being that, when a solution of cobalt cyanide in potassium cyanide is boiled in presence of free hydrogen cyanide (which you have formed by adding the one or two drops of hydrogen chloride), potassium cobalticyanide (corresponding to potassium ferricyanide) is formed, which is not decomposed by dilute acids.

NICKEL.

1. Make a borax bead, add to it a minute quantity of nickel sulphate, and heat it in the oxidising flame. A reddish brown colour will be imparted to the bead, and when it is heated in the reducing flame it will become grey and turbid.

2. Dissolve 5.3 grms. of nickel sulphate in 20 c.c. of water, and test portions of the solution in precisely the same way as you tested the cobalt salt.

(*a*) With solution of hydrogen sulphide. No precipitate will be formed until ammonia is added, when a black precipitate of nickel sulphide will be produced.

(*b*) With solution of ammonium sulphide. The nickel sulphide will dissolve sparingly in excess of ammonium sulphide, forming a dark brown solution. In other respects it resembles cobalt sulphide.

(*c*) With solution of potassium hydrate. A light green precipitate of nickel hydrate will be formed, which is not altered when the solution is boiled.

(*d*) With solution of ammonia. The green precipitate will readily dissolve in excess of the precipitant, forming a blue solution, but it is reprecipitated from this solution on addition of solution of potassium hydrate.

(*e*) With solution of potassium cyanide. The yellowish green precipitate of nickel cyanide is readily dissolved by excess of the precipitant, and is reprecipitated by excess of dilute hydrogen chloride, even after the solution has been boiled. No salt analogous to a cobalticyanide appears to be formed in the case of nickel.

MANGANESE.

1. Make a borax bead, add to it a minute quantity of manganese dioxide, and heat it in the oxidising flame of the blowpipe. The bead will be coloured violet, but when heated for a short time in the reducing flame, it will become colourless.

2. Make a bead of sodium carbonate (p. 137), add to it a trace of manganese dioxide, and heat it in the oxidising flame. The opaque bead will, when cold, be of a bluish green colour.

3. Make a mixture of 4 grms. of manganese dioxide with

3 grms. of potassium chlorate, and place it in a small porcelain dish. Dissolve 6 grms. of potassium hydrate in 5 c.c. of water, and add the solution to the mixture in the dish. Stir the whole together, and evaporate it to dryness over the lamp; then heat the dish nearly to redness, and keep it at that temperature for four or five minutes. The dark green, semi-fused mass which is formed consists of potassium manganate, produced by the action of the potassium chlorate and hydrate upon the manganese dioxide[1]. When it is cold, detach some of it with a knife from the dish, powder it in a mortar, transfer it to a test-tube and pour on it 10 or 12 c.c. of cold water. Allow any insoluble particles to subside and pour off some of the deep green solution into two other tubes.

(*a*) Heat the contents of one tube to boiling; the colour of the solution will change from green to purple, while a flocculent brown precipitate of manganese hydrate is formed.

(*b*) To the solution in the other tube add one or two drops of dilute hydrogen sulphate; the same change of colour will be produced, without any precipitate.

The change in both cases is due to the instability of the potassium manganate, which decomposes into a permanganate and a manganese salt[2].

4. Test two portions of the solution of potassium permanganate obtained in the last experiment (acidified with hydrogen sulphate), the one with solution of sodium sulphite, the other with solution of iron protosulphate. The purple colour will immediately disappear in both cases, owing to the decomposition of the permanganate and formation of a colourless manganese salt[3]. The facility with which the permanganates give up oxygen, and the total disappearance of colour

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[1] $3\,MnO_2 + 6\,KHO + KClO_3 = 3\,K_2MnO_4 + KCl + 3\,H_2O.$

[2] $3\,K_2MnO_4 + 4\,H_2O = 2\,KMnO_4 + 4\,KHO + MnH_4O_4.$
$5\,K_2MnO_4 + 4\,H_2SO_4 = 4\,KMnO_4 + 3\,K_2SO_4 + MnSO_4 + 4\,H_2O.$

[3] $2\,KMnO_4 + 5\,Na_2SO_3 + 3\,H_2SO_4 = K_2SO_4 + 2\,MnSO_4 + 5\,Na_2SO_4 + 3\,H_2O.$
$2\,KMnO_4 + 10\,FeSO_4 + 8\,H_2SO_4 = K_2SO_4 + 2\,MnSO_4 + 5\,Fe_2(SO_4)_3 + 8\,H_2O.$

which marks their decomposition, render them of great use in volumetric analysis.

5. Place 1 grm. of manganese dioxide in a porcelain dish, add 6 or 7 c.c. of strong hydrogen chloride, and heat the mixture gently on a sand-bath, taking great care that none of the chlorine evolved escapes into the room[1]. When all the black oxide has disappeared, evaporate the liquid to complete dryness, and heat the light pink residue rather strongly (but not to redness), in order to decompose any iron chloride which has been formed owing to the presence of iron as an impurity in the manganese dioxide. Warm the residue, when cool, with 10 c.c. of water, filter the solution of manganese chloride, and examine it as follows:—

(*a*) Test one portion with a drop of solution of potassium hydrate. A nearly white precipitate of manganese protohydrate will be formed, which will soon become brown on agitation, owing to absorption of oxygen from the air.

(*b*) Test another portion with a drop of solution of ammonium sulphide. A pink-coloured precipitate of manganese sulphide will be formed, which will readily dissolve on addition of a few drops of dilute hydrogen chloride.

ZINC.

1. Take a strip of sheet zinc about 20 cm. in length, hold it by its extremities and bend it double; notice that it is stiff, and requires some little force to bend it. Now heat it in the middle gently over a lamp, and observe that its pliability is greatly increased. If, however, it is still further heated it becomes quite brittle as the temperature approaches its melting-point.

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[1] Instead of this, the residue of manganese chloride obtained in the preparation of chlorine gas (p. 101) will answer very well.

2. Place 1.5 grms. of granulated zinc (p. 23) in a test-tube, and pour over it about 10 c.c. of dilute hydrogen chloride. An effervescence will commence owing to the liberation of hydrogen (Part I, Sect. 2, Ex. 2). While the solution of the metal is proceeding, you may go on to the next experiment. A little more strong hydrogen chloride should be added when the action becomes slow.

3. Heat a small fragment of zinc on charcoal in the hottest part of the blowpipe-flame. It will, when the heat rises to full redness, begin to burn with production of zinc oxide, light flakes of which will be swept away by the current of air, but the greater part will remain on the charcoal, as an incrustation which is yellow while hot, white when cold. Moisten this incrustation with a drop of solution of cobalt nitrate on the end of a glass rod, and heat it again before the blowpipe. The mass will now acquire a fine green colour.

4. Pour the contents of the test-tube used in Experiment 2 into a small porcelain dish, and evaporate to dryness on the sand-bath in order to drive off any excess of acid. Warm the white residue of zinc chloride with 20 c.c. of water, filter the solution, and test portions of it as follows:—

(*a*) Add to one portion a drop of solution of potassium hydrate. A white precipitate of zinc hydrate will be formed, which will readily redissolve on addition of a few more drops of the solution of potassium hydrate. Divide the clear liquid into two parts:

(α) To one portion add a drop or two of solution of hydrogen sulphide. A white precipitate of zinc sulphide will be formed.

(β) To the other portion add about half its volume of solution of ammonium chloride, and warm the mixture: no precipitate will be formed[1]. (Compare the reactions of aluminium, p. 222.)

(*b*) Test another portion of the solution of zinc chloride

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[1] If the potassium hydrate contains aluminium, a precipitate will, of course, be formed (see p. 222).

with a drop of solution of ammonium sulphide. A white precipitate of zinc sulphide will be formed, which will readily dissolve on addition of a drop or two of dilute hydrogen chloride.

5. Make a solution of lead acetate by dissolving 10 grms. of the salt in 200 c.c. of water, with addition of a few drops of hydrogen acetate. Place the solution in a wide-mouthed bottle, and suspend in it a strip of thick sheet zinc, or, better, a rod of the metal about 1 cm. in diameter[1]. A short piece of glass tubing should be laid across the mouth of the bottle, from which the zinc may be suspended by a piece of string, so that it may not touch the bottom of the bottle. Leave the whole undisturbed for twelve or fourteen hours: the rod of zinc will soon be covered with thin brilliant plates of pure metallic lead, while a proportional quantity of zinc is dissolved. This is a good illustration of the replacement of one metal by another in a combination; and, if we ascertained the weight of zinc dissolved and the weight of lead deposited, we should find that the former weight was to the latter as 65 : 207, numbers which are the accepted atomic weights of zinc and lead respectively.

In a day or two the whole of the lead will have been withdrawn from the solution, which may then be tested as directed in p. 210, Experiment 7, to prove the absence of lead, and as directed in p. 232, Experiment 4 *a*, to prove the presence of zinc.

6. Fill a test-tube about half full of dilute hydrogen sulphate, and immerse in the liquid a small strip of zinc. Hydrogen will be evolved from the surface of the metal. Now add about 2 c.c. of a solution of mercury perchloride; a bright film of mercury will be deposited on the zinc, and the evolution of hydrogen will cease entirely. The mercury has, as in the case of lead, been replaced by the zinc, but the deposited metal has

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[1] As the shape of the rod is immaterial, it may be easily made by melting some zinc in a ladle, and casting it in a shallow trough formed by pressing a thick pencil or a piece of glass tube into a mass of fine, slightly moistened sand, or clay.

formed a combination or 'amalgam' with the zinc, which protects the metal from the action of the hydrogen sulphate[1]. Immerse in the solution a strip of copper; no action occurs until it is brought into contact with the zinc, when hydrogen is again evolved, not, however, from the surface of the zinc, but from the copper. You have formed what is called a 'galvanic couple,' since the dilute acid acts unequally on the copper and on the zinc amalgam immersed in it. The two plates assume opposite electric states, and the electro-positive substance, the hydrogen, is evolved from the surface of the electro-negative plate, the copper.

BARIUM.

1. Dissolve 1 grm. of barium chloride in 17 c.c. of water, and examine separate portions of the solution as follows:—

(*a*) Hold a loop of platinum wire in the flame of a Bunsen's burner, or, better, of a gas blowpipe, until it no longer imparts any colour to the flame, dip it into the solution of barium chloride, and hold it again in the flame. The barium salt will, as it volatilises, impart to the flame a bright green colour.

(*b*) To a portion of the solution add a drop of solution of ammonium sulphide. No precipitate will be produced.

(*c*) To another portion add a drop of solution of ammonium carbonate. A white precipitate of barium carbonate will be formed, which will readily dissolve, with evolution of carbon dioxide, on addition of a few drops of dilute hydrogen chloride.

(*d*) To another portion add about one-third its volume of solution of calcium sulphate. A white precipitate of barium sulphate will be immediately formed.

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[1] Absolutely pure zinc is not acted on by dilute hydrogen sulphate. In the case of ordinary zinc the action arises from the presence of particles of carbon, lead, &c., which form, like the copper, galvanic couples, and thus originate local action. The protective action of the mercury amalgam is due mainly to the fact that it covers these impurities, and presents a surface of pure metal to the acid.

The same reaction has already been employed to detect the presence of a sulphate (p. 175).

(*e*) To another portion add a few drops of solution of hydrogen and silicon fluoride. A crystalline precipitate of barium and silicon fluoride will be produced.

2. Place a small lump of barium oxide in a test-tube, and pour on it a few drops of water. If the barium oxide is pure and has not been exposed to the air, it will swell up, evolving much heat as it combines with the elements of the water to form barium hydrate. (Compare the action of water on calcium oxide, p. 45.) Pour about 8 or 10 c.c. of water upon the mass, heat it to boiling, and filter it while hot into a clean test-tube. Crystals of barium hydrate will be deposited as the liquid cools, since the salt is much more soluble in hot than in cold water. Observe also the alkaline reaction of the solution on reddened litmus and turmeric-paper.

3. Place a small lump of barium oxide in a mortar, grind it to powder, then add rather more than the same quantity of potassium chlorate and mix the two substances intimately with the pestle. Place the mixture in a small porcelain crucible, and heat it rather strongly over a Bunsen's burner. A low incandescence will spread through the mass, the barium oxide burning in presence of the chlorate (precisely in the same way as carbon or any other combustible body) with formation of barium dioxide[1]. Allow the semi-fused mass to cool, then detach it from the crucible and grind it to a fine powder in a mortar, adding, finally, sufficient water to make a thin paste. Pour into the mortar about 5 or 6 c.c. of dilute hydrogen chloride, and mix it quickly with the semi-fluid mass, which ought to dissolve readily and completely; if not, a little more hydrogen chloride must be added at once. A slight effervescence will take place, owing partly to the presence of carbonate as an impurity in the barium oxide, partly to the decomposition of the dioxide with evolution of oxygen gas. If, however, the hydrogen chloride is kept in excess, the amount of decomposition is small, and a solution is obtained containing barium

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[1] $3\,BaO + KClO_3 = 3\,BaO_2 + KCl$.

chloride and hydrogen dioxide[1]. The following experiments may next be made with the solution:—

(*a*) Render a portion of it alkaline with solution of barium hydrate, and filter it quickly into a clean tube from the precipitate of iron hydrate and aluminium hydrate which will be formed, since common barium oxide is never free from these impurities. Add a little more solution of barium hydrate, and shake the mixture. Brilliant pearly scales of barium dihydrate will be gradually formed.

(*b*) Add a few drops of the solution to some solution of potassium iodide acidified with hydrogen chloride. Iodine will be liberated by degrees, colouring the liquid yellow and giving the characteristic blue compound if a little solution of starch is added.

(*c*) Add a little of the solution to some solution of potassium permanganate. The liquid will become colourless, a manganese protosalt being formed, while oxygen is evolved with effervescence. The hydrogen dioxide has here acted as a reducing agent, although in Experiment *b* we saw that it acted upon an iodide as an oxidising agent[2]. (Compare its action on chromates, p. 224.)

STRONTIUM.

1. Dissolve 4 grms. of strontium nitrate in 20 c.c. of water, and repeat with the solution the experiments which you made with the barium salt, carefully noticing the points in which the two substances differ and agree.

(*a*) When heated in the lamp-flame. A deep crimson colour will be imparted to the flame.

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[1] $BaO_2 + 2\,HCl = BaCl_2 + H_2O_2$. Potassium chloride is, of course, also present.

[2] The changes may be expressed by the following equations:—

In the case of the iodide,

$$2\,HI + H_2O_2 = 2\,H_2O + I_2.$$

In the case of the permanganate,

$$2\,HMnO_4 + 4\,HCl + 5\,H_2O_2 = 2\,MnCl_2 + 8\,H_2O + 5\,O_2.$$

(*b*) Tested with ammonium sulphide. No precipitate will be produced.

(*c*) Tested with ammonium carbonate. A white precipitate of strontium carbonate will be formed, soluble in dilute hydrogen chloride.

(*d*) Tested with calcium sulphate. No precipitate will be formed at once, but after the lapse of some minutes a white precipitate of strontium sulphate will gradually appear.

(*e*) Tested with hydrogen and silicon fluoride. No precipitate will be formed.

2. Place about 1 grm. of crystallised strontium nitrate in a porcelain crucible, or on a piece of a broken evaporating dish, and heat it to full redness before the gas blowpipe. The salt will fuse and effervesce owing to the evolution of nitrogen oxides and oxygen, and finally a dark grey porous mass of strontium oxide will be left. When this is cool pour a few drops of water upon it, and notice that it 'slakes' like the corresponding barium and calcium salts, and that its solution in water has an alkaline reaction on test-paper.

CALCIUM.

1. Dissolve 2.2 grms. of calcium chloride in 20 c.c. of water, and test the solution in the same way as you have tested the barium and strontium salts.

(*a*) When heated in the lamp-flame. An orange-red colour will be imparted to the flame.

(*b*) Tested with ammonium sulphide. No precipitate will be produced.

(*c*) Tested with ammonium carbonate. A white precipitate of calcium carbonate will be produced, soluble in dilute hydrogen chloride.

(*d*) Tested with calcium sulphate. No precipitate, of course, will be produced, even on allowing the mixture to stand for a quarter of an hour.

(*e*) Tested with hydrogen and silicon fluoride. No precipitate will be produced.

(*f*) Add to another portion of the solution a drop of solution of ammonium oxalate. A white precipitate of calcium oxalate will be formed. The same reaction was employed (p. 155) to detect the presence of an oxalate.

2. Place a piece of reddened litmus-paper in a porcelain dish; lay on the paper a small fragment of marble, about as large as a pea, and pour a few drops of water from a washing bottle over the marble. The colour of the litmus-paper will not be altered, since marble is quite insoluble in water. Place the fragment of marble on a piece of platinum foil, fold down the corner of the platinum foil to retain the marble in its place, and heat it to full redness before the blowpipe for a minute or two. Place it, when cool, upon the litmus-paper, and add a drop or two of water. The paper will now turn blue, since the marble has been decomposed by heat into carbon dioxide and calcium oxide and the latter substance unites with the elements of water to form calcium hydrate, as we have seen in Part I, Ex. 5.

3. Roll a strip of paper, about 2 cm. broad and 20 cm. long, round a penny, or medal, so as to form a shallow trough, and fasten down the end of the strip by sealing-wax or by a fold or two of string. Place 20 c.c. of water in a porcelain dish, and shake into it gradually, from a paper gutter, sufficient fresh plaster of Paris to form a thin paste, stirring the mixture continually with a glass rod. As soon as the paste is thoroughly mixed, pour over the coin sufficient of it to fill the trough, and stir it with a feather or splinter of wood, in order to detach any bubbles of air which may remain adhering to the surface of the coin. Leave the whole at rest for about half an hour, in which time you will find that the paste has solidified, or 'set,' as it is termed, the calcium sulphate having (like calcium oxide) combined chemically with the water. Unroll the paper rim, and carefully detach the coin from the plaster by inserting the point of a pen-knife between them. You will thus obtain a copy of the coin in plaster, but reversed, the raised parts

of the coin forming depressions in the plaster. The cast should be left for an hour or two in a warm place, and when it is thoroughly dry it may be used as a mould from which to obtain a facsimile of the original coin. For this purpose its surface must be lightly dabbed with a tuft of cotton-wool dipped in sweet oil[1]; a paper rim must then be placed round it, and some freshly mixed paste poured into it as above directed.

4. Reduce to powder a little of the remaining paste, which has solidified in the porcelain dish, and heat it in a dish on the sand-bath, stirring it continually. The water which it contains will be given off (as may be shown by holding a cold beaker over the dish), and it will again acquire the property of setting when made into a paste with water.

MAGNESIUM.

1. Place a piece of reddened litmus-paper in a porcelain dish, and moisten it with water. Cut off a piece of magnesium ribbon or wire about 12 cm. in length, hold it in the crucible tongs over the porcelain dish, and apply the lamp-flame to one extremity. The metal will take fire and burn with an intense white light, forming white flakes of magnesium oxide, which should be allowed to drop upon the test-paper. The latter will have its blue colour restored at the points where it is touched by the magnesium oxide.

2. Place a bit of magnesium ribbon about 25 cm. in length in a test-tube and pour on it some dilute hydrogen sulphate. Hydrogen gas will be evolved with effervescence, and the metal will gradually dissolve. When the action has ceased, pour off the liquid into a small porcelain dish, and evaporate it down until it begins to crystallise. Long prismatic crystals of magnesium sulphate (Epsom salts) will be deposited in abundance

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[1] This is done in order to prevent the adhesion of the liquid plaster to the mould. A better expedient is, to saturate the mould with wax by placing it, plain side downwards, in a porcelain dish containing a little melted wax or paraffin. The wax will soon rise through the porous plaster, and saturate it thoroughly.

as the solution cools: these should be redissolved in about 20 c.c. of water, and separate portions of the solution tested as follows:—

(*a*) To a portion add a drop or two of solution of ammonium sulphide. No precipitate will be formed, since magnesium sulphide, unlike zinc sulphide, is soluble in water.

(*b*) To another portion add a drop of solution of sodium carbonate. A white precipitate of magnesium carbonate will be formed, which will readily redissolve on addition of a few drops of solution of ammonium chloride.

(*c*) Test another portion with solution of ammonium carbonate. No precipitate will be produced until after the lapse of some time, although we have seen in the preceding experiment that magnesium carbonate is insoluble in water. The reason of this non-precipitation is that magnesium salts have a great tendency to combine with ammonium salts to form compounds which are, in general, more soluble than the simple magnesium salts. Thus we saw in the last experiment that the addition of ammonium chloride caused the magnesium carbonate to redissolve. In the next experiment we shall form one of these double salts, which is extremely insoluble.

(*d*) Add to another portion of the solution five or six drops of solution of ammonium chloride, then a drop or two of solution of ammonia, and lastly a drop of solution of sodium phosphate. A white granular precipitate will be formed consisting of magnesium and ammonium phosphate. To illustrate the delicacy of this reaction, you may place one drop of the solution of magnesium sulphate in a clean test-tube, add about 10 c.c. of water, and then successively a few drops of solution of ammonium chloride, a drop of ammonia, and a drop of solution of sodium phosphate. If no precipitate is produced at first, dip a clean glass rod into the solution and gently rub the sides of the test-tube with it. Crystals will soon begin to form along the lines at which the rod touched the sides of the tube, especially if the liquid is agitated for a few seconds.

(*e*) Place a small crystal of magnesium sulphate on charcoal and heat it strongly before the blowpipe. Moisten the white infusible residue with solution of cobalt nitrate, and heat it again. The mass, when cool, will be found to have acquired a faint pink colour.

POTASSIUM.

1. Pour some distilled water into a large porcelain dish, and wet the sides of the dish above the surface of the water by rubbing them with the moistened finger[1]. Take a small piece of potassium, about as large as a pea, out of the bottle, free it from adhering naphtha by pressing it between folds of blotting-paper, and cut it in two with a knife. Notice the softness of the metal, the brilliant lustre of the freshly cut surfaces, and the rapidity with which the latter are tarnished in the air, owing to the formation of a coat of oxide. Take up one of the pieces of potassium in the crucible tongs and dip it in the centre of the dish of water. It will immediately take fire, burning with a violet flame as it floats from side to side of the dish. The water is decomposed by the metal, a portion of its hydrogen being replaced by potassium, with formation of potassium hydrate[2]. The heat produced is sufficient to kindle the hydrogen evolved, and to volatilise a small portion of potassium which gives the flame its violet tinge. Immediately on the cessation of the flame, you will observe the globule of potassium hydrate floating red-hot upon the water. Cover it at once with an inverted funnel or beaker, and observe that in a second or two it disappears with a slight explosion. The globule, while red-hot, was not in actual contact with the water, but was supported on a cushion of steam. As it cooled, a point arrived at which the layer of steam was condensed, and

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[1] This is done in order to prevent the globule of potassium adhering to the sides instead of floating freely on the water. It is a good plan to rinse out the dish, just before using it, with a little solution of caustic potash, and then with clean water.

[2] $2\,H_2O + K_2 = 2\,K\,H\,O + H_2$.

the globule touched the water, causing a fresh burst of steam sufficient to propel portions of the globule to a considerable distance. It is well, therefore, to cover the substance with a funnel, as above directed, lest the eyes should suffer. After burning the other piece of potassium in a similar way, you may examine the solution in the dish. It will have acquired a sharp caustic taste, and the colour of a piece of reddened litmus-paper dipped into it will be changed to blue. If the liquid is evaporated to dryness a white residue of potassium hydrate will be left.

2. Place a piece of moist reddened litmus-paper in a porcelain dish, and hold over it a burning cedar match or wooden spill, so that the ashes may drop upon the paper. Pour over the ashes a few drops of water, and notice that the litmus-paper becomes blue, owing to the potassium carbonate present in the ash.

3. Place a small lump of potassium hydrate on a watch-glass, and leave it for a short time exposed to the air. Its surface will almost immediately become moist, and it will soon deliquesce, owing to absorption of moisture from the air, forming an oily liquid which turns reddened litmus-paper blue. Leave it exposed until the next day, and then add to it a drop or two of dilute hydrogen chloride. An effervescence will take place, owing to the escape of carbon dioxide. Potassium hydrate absorbs not only moisture but also carbon dioxide from the air[1].

4. Dissolve a small lump of potassium hydrate in about 5 c.c. of water, noticing the heat evolved (as in the case of hydrogen sulphate) during the solution. Add to the liquid about half its volume of oil, and heat the mixture to boiling, shaking it frequently. A viscid solution of soap will be produced by degrees, by the action of the organic acids in the oil upon the potassium hydrate, potassium oleate being the chief product.

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[1] You will remember that it was used in the preparation of carbon protoxide (p. 92), for the purpose of absorbing the carbon dioxide produced in the decomposition of hydrogen oxalate.

5. Dissolve about 2 grms. of potassium nitrate in 20 c.c. of water, and test portions of the solution as follows:—

(*a*) Dip a perfectly clean platinum wire into the solution, and hold it in the flame of a Bunsen's burner. The salt will impart a violet colour to the flame. If you look at the flame through a piece of deep blue glass (which can be easily procured from a glazier), it will appear crimson.

(*b*) Test a portion of the solution with solution of ammonium sulphide. No precipitate will be formed.

(*c*) Test another portion with solution of ammonium carbonate. In this case also no precipitate will be formed.

(*d*) Add to another portion a drop of dilute hydrogen chloride, and then two drops of solution of platinum perchloride. A yellow crystalline precipitate of potassium and platinum chloride will be formed, especially if the liquid is shaken. In order to prove the delicacy of this test you may modify the experiment as follows:—Place one or two drops of the solution of potassium nitrate in a test-tube, add 5 c.c. of water, then one drop of dilute hydrogen chloride, and lastly a drop of solution of platinum perchloride. No precipitate will now be produced, since the quantity of the potassium salt present is very small. Pour the liquid into a watch-glass and evaporate it to dryness, on the sand-bath, at a gentle heat. When the residue is cool, mix in a test-tube equal volumes of water and common alcohol, and pour some of the mixture over the residue. The greater part of it will dissolve, but a light yellow residue, consisting of the platinum and potassium salt will remain, since the latter is almost insoluble in alcohol.

(*e*) Add to another portion a few drops of solution of sodium and hydrogen tartrate, rub the sides of the test-tube with a glass rod (p. 240 *d*), and shake the mixture. A crystalline precipitate of potassium tartrate will be formed.

SODIUM.

1. Examine the properties of the metal sodium and its decomposition of water in precisely the same way as you examined the properties of potassium in the last exercise. You will find that it has many points of resemblance to potassium, but that it does not tarnish quite so readily in the air, and that when placed upon water it causes the evolution of hydrogen, but the action is not so violent as to inflame the gas. If, however, you modify the experiment by floating a piece of filtering-paper upon the water, and placing the pellet of sodium in the centre of it so as to confine it in one place and thus prevent the distribution of the heat, the gas evolved will catch fire, and burn with an intense yellow flame, the colour being due to the volatilisation of a portion of the sodium.

2. Dissolve 1.2 grms. of sodium chloride in 20 c.c. of water, and examine the solution in the same way as you examined the potassium salt in the last exercise.

(*a*) The salt will impart an intense yellow colour to the flame. This will, however, be quite invisible when looked at through the blue glass[1].

(*b, c, d, e*) In none of these cases will any precipitate be produced, since the sodium salts formed in the reactions are soluble in water.

When a solution of sodium salt is evaporated to dryness after addition of solution of platinum perchloride, a residue is obtained, but this residue dissolves completely in dilute alcohol.

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[1] If a crystal of potassium dichromate is looked at by the light of the sodium flame it appears quite colourless. A scarlet geranium, looked at in the same way, appears white, and a blue lobelia or larkspur appears perfectly black.

HYDROGEN.

(The preparation and properties of hydrogen gas have been given at p. 67, and those of hydrogen dioxide have also been mentioned under the head of barium dioxide, p. 235.)

The chief distinctive properties of hydrogen salts, which form the class of substances commonly called acids, have already been illustrated. Thus,—

1. They redden blue litmus-paper (pp. 99, 145), but have no action on turmeric-paper.

This property belongs to all the hydrogen salts which the student is likely to meet with in a course of elementary work, with the exception of hydrogen borate, which, as we have seen (p. 181), renders turmeric-paper brownish red, while it gives the usual acid reaction with blue litmus-paper.

2. They react on certain radicles, of which potassium is a typical specimen, to form substances in which their action on litmus-paper is lessened, and in some cases destroyed (see p. 145, Experiment 3).

3. Some of them, e.g. hydrogen sulphate (p. 69), when brought into contact with zinc in presence of water give off hydrogen in the form of gas.

We are seldom, however, able to pronounce with certainty that a given substance is a hydrogen salt, until we have proved the absence of other metallic radicles and the presence of a non-metallic radicle in it. If, having done this, we find that it reddens litmus-paper when in solution, and only loses this property after the addition of several drops of solution of potassium hydrate or ammonia, it may be safely inferred that it is a hydrogen salt, and the solution should be further examined to see if it possesses the known properties, such as volatility, of the hydrogen salt of the non-metallic radicle.

PART II.

QUALITATIVE ANALYSIS OF SINGLE SALTS.

SECTION I.

Explanation of the Analytical Course.

Every portion of matter, considered chemically, is either a single substance or an aggregate of two or more single substances. By a single substance, we mean a substance from which no part, having different properties from the rest, can be separated by any physical means, such as pounding and sifting, or the action of solvents, or distillation, or diffusion. Water, and salt, and hydrogen, and indeed all the various substances whose properties you have been studying, are single substances[1]. Other portions of matter, consisting of two or more single substances aggregated together, such as a mixture of salts, or a piece of rock, or any part of a plant or of an animal, are called mixtures.

According to this twofold division of natural objects, analysis is divided into the analysis of single substances, and the analysis of mixtures. Further, the analysis may in each case be either qualitative or quantitative, the former having for its object the identification of a single substance or of the ingredients of a mixture, the latter enquiring in what proportions the elements of a single substance, or the various single substances in a mixture, coexist.

Of the four divisions of analysis thus indicated, the present

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[1] The term 'chemical substance' is often used in this sense, but has the disadvantage of suggesting a peculiar class of substances instead of expressing the simple fact.

volume treats only of the first—the qualitative analysis of a single substance.

Two further limitations should be named in order exactly to define the scope of the analytical course which follows. First, you will have observed that the preceding exercises deal only with the commoner or more abundant elements. About half the elements at present known are so rare that it is found expedient to exclude them from consideration in framing the analytical course. Secondly, of the radicles formed from these elements some are continually met with among natural or artificial products, while others occur but seldom. Here, again, it has been necessary to make an arbitrary limitation, and to select from a host of such radicles those of a few of the commonest series of salts. The problem, then, which the following analytical course supplies the means of solving should be stated thus:—Given that x is a single substance consisting of one or more of the radicles specified in our list, to find out what it is.

The great majority of the substances which present themselves for analysis are what are commonly termed salts, and consist of a combination of a simple radicle, belonging to the class of metals, with a simple or compound radicle belonging wholly or chiefly to the non-metallic class; a few salts contain more than one radicle of either class. For example, sodium chloride, $NaCl$, consists of the simple metallic radicle Na and the simple non-metallic radicle Cl; silver nitrate, $AgNO_3$, of the simple metallic radicle Ag, and the compound non-metallic radicle NO_3; sodium and potassium tartrate $KNaC_4H_4O_6$, sodium ammonium and hydrogen phosphate $Na(H_4N)HPO_4$, magnesium phosphate and fluoride Mg_2PO_4F, exemplify the union of more than two radicles. Conformably with this fact as to the constitution of salts, the course of analysis consists of two principal parts, the one determining the metallic radicle or radicles, the other the non-metallic radicle or radicles of which a substance is formed.

The general principles on which the analysis of a substance is based, have been already explained in the introductory remarks to Part I, Sect. 5, p. 139, and the acquaintance you have gained with the properties of the different radicles and their salts furnishes you with the means of distinguishing any one from the rest and identifying it. But it remains to be seen in what manner this end can be most surely and quickly accomplished. It would be possible by trying in any order a sufficient number of the experiments described in the section upon the substance under examination, to discover at last its true nature; but much time would be lost upon experiments which yielded no information, and the analysis would not unfrequently end in inextricable confusion. We ought to start, then, with a clear perception of the relative value of the tests we are going to employ: we must select them on definite principles, and observe a definite order in applying them.

It will have been noticed that among the experiments which have been tried with each individual substance, some have brought to light properties which it possesses in common with certain other substances, while others have given results which are quite characteristic of the particular substance, and distinguish it sharply from all others. To illustrate this, the following experiments may be repeated:—

Place five test-tubes in the stand, and pour into them, respectively, 2 or 3 c.c. of solution of SODIUM SULPHATE, AMMONIUM OXALATE, AMMONIUM CHLORIDE, POTASSIUM IODIDE, and POTASSIUM NITRATE. Place a small label on each tube, to show what it contains; then pour a few drops of the solution of sodium sulphate into another tube; add about twice as much water, and then add a drop of solution of **barium chloride.** A white precipitate will be produced (p. 175). Place the test-tube in the stand, and test in a similar way portions of the four other solutions. You will find that barium chloride produces a precipitate in the solutions of sodium sulphate, and ammonium oxalate, but not in the solutions of ammonium chloride, of potassium iodide, or of potassium nitrate. Assuming what you may hereafter verify, that the sodium, ammonium, and potassium present have no influence in the production or prevention of a precipitate in this case, you have learnt that certain substances, such as sulphates and oxalates, give precipitates when their aqueous solutions are tested with solution of barium chloride, while certain others, such as chlorides, iodides, and nitrates, give no precipitate when tested with the same reagent.

I. The SULPHATES, OXALATES, &c. are therefore classed together in one group, as having the common property of being precipitated from aqueous solutions by **barium chloride.**

II. The CHLORIDES, IODIDES, NITRATES, &c. are also classed together in one group, as having the common property of not being precipitated from aqueous solutions by **barium chloride.**

Next, take fresh portions of the solutions of ammonium chloride, potassium iodide, and potassium nitrate, and test each with a drop of solution of **silver nitrate.** You will obtain precipitates in the case of the chloride and iodide, but not in the case of the nitrate.

Therefore, of those substances which are not precipitated by barium chloride:—

The CHLORIDES, IODIDES, and certain others, are classed together in one group, as having the common property of being precipitated from their aqueous solutions by **silver nitrate**, the nitrates and certain others are classed together in another group, as having the common property of not being precipitated from their aqueous solutions by **silver nitrate.**

Now, if you examined in a similar way solutions of salts of all the radicles treated of in pp. 143–183, you would find that they might all be arranged under one or other of the three groups above indicated, and you would be able to construct a table such as the following, showing their distribution into groups, and the distinctive property of each group.

GROUP I.

Substances which are precipitated from neutral aqueous solutions on addition of **barium chloride.**

CARBONATES.
TARTRATES (partially).

OXALATES.
FLUORIDES (partially).
SULPHITES.
SULPHATES.
PHOSPHATES.
BORATES (partially).
SILICATES.

GROUP II.

Substances which are not precipitated from neutral aqueous solutions by **barium chloride**, but are precipitated by **silver nitrate.**

CYANIDES.
CHLORIDES.
BROMIDES.
IODIDES.
SULPHIDES.
HYPOSULPHITES [1].
HYPOPHOSPHITES [1].
ACETATES (partially).

GROUP III.

Substances which are not precipitated from neutral aqueous solutions by either **barium chloride** or **silver nitrate.**

NITRATES.
CHLORATES.

The next step will be to try the action of other reagents on the members of each group, in order to be able to carry the principle of division still further, and to arrange them in smaller sub-groups. For instance,—

Take the portions of the solutions of ammonium chloride and potassium iodide which contain the precipitates produced by the addition of silver nitrate, add to each five or six drops of solution of **ammonia**, and shake the mixture. The precipitate produced in the solution of the chloride will be dissolved (p. 202), while that which was produced in the solution

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[1] Silver hyposulphite and silver hypophosphite are very unstable.

of the iodide will remain undissolved. And if the same experiment is repeated with all the members of Group II, it will be found that they may be arranged in two divisions.

A.

Substances, the silver salts of which are soluble in **ammonia.**

CYANIDES.
CHLORIDES.
BROMIDES (with difficulty).
HYPOSULPHITES.
HYPOPHOSPHITES.
ACETATES.

B.

Substances, the silver salts of which are insoluble in **ammonia.**

IODIDES.
SULPHIDES.

Finally, when we have thus divided the radicles into conveniently small groups, we proceed to apply tests which give characteristic reactions with the individual members of each group. For instance,—

Take a fresh portion of the solution of potassium iodide, add a few drops of **carbon disulphide,** and one drop of **chlorine water.** The solution will become yellow, and after agitation the carbon disulphide will collect at the bottom of the tube in a globule of a violet colour. This, as we have already seen (p. 168), is a reaction which distinguishes an iodide from all other substances.

In the examination, then, of a single salt of an alkali metal (potassium, sodium, or ammonium) for its other constituent, the first step will be to ascertain, by testing separate portions of the neutral aqueous solution with barium chloride, and with silver nitrate, to which group the radicle belongs. When this is known with certainty, the subsequent examination will be limited to a search for one of the substances included in that particular group. Other tests are then applied in order to discover the subdivision of the group to which the radicle belongs, and thus the search is limited to a still smaller number of substances. Finally, special tests are applied in order to discover which one, out of the few possible substances, is actually present. We might here rest content with having obtained one reaction which is characteristic of the presence of one particular substance. But it is highly expedient to accumulate evidence, so as to leave no possible room for doubt as to the constitution of the substance under examination; and other confirmatory tests should always be tried.

Thus, in the case of the solution of potassium iodide, a precipitate has been obtained with silver nitrate, proving that the radicle belongs to Group II: this precipitate is insoluble in ammonia, and the radicle therefore is included in division B; the yellow colour of the precipitate and reaction with chlorine water and carbon disulphide indicate the presence of an iodide. Assuming, then, that an iodide is present, we try the action of other tests which are known to give characteristic reactions with an iodide. For example,—

Take a fresh portion of the solution of potassium iodide, add to it a few drops of solution of **starch**, and then a drop of **chlorine water**. The solution will acquire an intense blue colour. This reaction is absolutely characteristic of an iodide, and confirms the previous results.

The general course, then, to be pursued in a qualitative analysis of an unknown substance for one radicle, is—

Firstly, to ascertain the group to which the radicle belongs.

Secondly, to ascertain the subdivision of the group to which it belongs.

Thirdly, to ascertain which member of the subdivision is present.

Lastly, to confirm the results, by applying tests which give characteristic reactions with the radicle, the presence of which has been indicated.

The above experiments will, it is hoped, be sufficient to illustrate the principles of the systematic course of analysis which should next be practised. Single salts should be taken, containing an alkali metal associated with one of the radicles of which the properties have been already examined, and should be analysed with a view to the detection of this radicle only.

The substances which are treated of in the latter part of Sect. **5**, p. 183 seq., are arranged in groups on a principle similar to that which has been already explained above, viz. their precipitation from solution by certain reagents. The following experiments will illustrate the formation of these groups.

Take solutions of the six following substances (made, if necessary, by dissolving about 1 grm. of each salt in 10 c.c. of water):—

1. SILVER NITRATE.
2. COPPER NITRATE: the salt obtained as a residue in the preparation of nitrogen dioxide[1].
3. ZINC SULPHATE: the salt obtained as a residue in the preparation of hydrogen (p. 76).
4. CALCIUM CHLORIDE: the salt obtained as a residue in the preparation of carbon dioxide.
5. MAGNESIUM SULPHATE: Epsom salts.
6. POTASSIUM NITRATE: the salt used in preparing hydrogen nitrate (p. 143).

I. Pour a few drops of the solution of the silver salt into another test-tube, add about twice its volume of water, and then five or six drops of dilute **hydrogen chloride**[2]. A white precipitate will be produced. Make similar experiments with portions of each of the other solutions. No precipitate will be formed in any case.

We place, then, in one group substances, such as SILVER salts, which are precipitated from solutions on the addition of **hydrogen chloride.**

II. Now take the solution of copper nitrate to which hydrogen chloride was added in the last experiment, and add to it a few drops of solution of **hydrogen sulphide.** A black precipitate

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[1] Copper sulphate will answer equally well.

[2] This, and the other reagents used, are so selected that the presence of the other radicle (sulphate, nitrate, &c.) has no influence on the formation of a precipitate.

will be produced. Test in a similar way the four other solutions containing hydrogen chloride. No precipitate will be formed in any of them.

We associate in another group substances, such as COPPER salts, which are precipitated from solutions containing hydrogen chloride by **hydrogen sulphide.**

III. In the next place take a fresh portion of the solution of zinc sulphate and test it with a drop or two of solution of **ammonium sulphide.** A white precipitate will be produced. Make a similar experiment with portions of the other three solutions. No precipitate will be produced in any case.

We form, therefore, into a group substances, such as ZINC salts, which are not precipitated from solutions containing free acid by **hydrogen sulphide,** but are precipitated from solutions by **ammonium sulphide.**

IV. Next, take a fresh portion of the solution of calcium chloride, add half its volume of solution of ammonium chloride, and test it with a drop or two of solution of **ammonium carbonate.** A white precipitate will be produced. Make a similar experiment with the solutions of magnesium sulphate, and potassium nitrate. No precipitate will be produced in either case.

We constitute, therefore, another group of those substances, such as CALCIUM salts, which are not precipitated from their solutions by either **hydrogen sulphide** or **ammonium sulphide,** but which are precipitated by **ammonium carbonate** from solutions containing ammonium chloride.

V. Finally, take a fresh portion of the solution of magnesium sulphate, add half its volume of solution of ammonium chloride, and test it with a drop of solution of **sodium phosphate.** A white crystalline precipitate will be formed. Make a similar experiment with the solution of potassium nitrate. No precipitate will be produced.

We place, then, in a group substances, of which MAGNESIUM salts are the only known examples, which are not precipitated from solutions by **hydrogen sulphide, ammonium sulphide** or **ammonium carbonate** (if the solutions already contain ammonium salts), but which are precipitated by **sodium phosphate** from solutions containing ammonium chloride.

VI. Our last group consists of those substances, such as POTASSIUM, which are not precipitated from solutions by any of the reagents above mentioned, and which remain as a residue, in the course of an analysis, when all the other substances have been removed from the solution under examination.

By repeating the above experiments with each of the metallic radicles treated of in p. 183 seq., we should be enabled to place it in some one of the above six groups, and we might construct a Table similar to that which is given in p. 259.

The explanation of the system of analysis (p. 252) applies, of course, equally to the method to be followed in order to detect the metal present in a single salt. We have first to ascertain the group to which it belongs, next the subdivision of the group, then the individual metal, and lastly we apply confirmatory tests. In practice it is found advisable to examine a substance first for the metallic radicle it contains, chiefly for the reason that there are more cases in which ignorance of the metal present would interfere with the examination for the non-metallic radicle, than *vice versâ.*

It cannot be too strongly urged that a full account of every experiment made with the view of ascertaining the constituents of a substance, should be written out, not more for the sake of the teacher or examiner than for the advantage of the student himself. The former has, in general, no way of ascertaining what has been done except from the contents of the note-book; the latter will find no means of gaining accuracy of thought, clearness of expression, and insight into analytical methods, more effectual than constant practice in writing out accounts of work done.

Excellent models for such descriptions may be found in the original papers on substances either newly discovered or examined for the first time, contributed by our best chemists to scientific magazines. The object of every such paper is to make clear to others the views of the author on the nature of the substance he has examined, and his reasons for adopting these views; to give the experiments which have been tried, and the inferences drawn from them. This is no less the object of the student commencing analysis. Every substance presents to him an original problem, which should be approached in the same spirit, and worked out with the same care as the most elaborate research of a chemist like Faraday or Dumas.

A meagre, tabular view of work done, full of chemical symbols introduced without reference to their quantitative chemical meaning, but solely in order to save a few seconds of time, is not sufficient. The essential points in each experiment, and the inferences deducible from it should be put down in plain logical English as soon as possible after the experiment has been made. Many moments of spare time, while a precipitate is being washed or an evaporation is going on, will be found available for writing; while, if all description is deferred until the analysis is completed, there is great danger that some steps will escape the memory.

An attempt is made at the end of this Part to give an example of the way in which an account of the analysis of a single salt should be written out, and to this the student should refer before he begins his analytical work.

The authors have intentionally omitted to give any so-called 'Analytical Tables' (with the exception of those which show the distribution of the radicles into groups); not because they consider such Tables of no value, but from a conviction that the Tables will be best made by the student himself, in the same way as a summary of the contents of a book, or a *prècis* of a document, rather as a result of intelligent work than as an introduction to it.

Comparatively few single substances are met with in a state of absolute chemical purity, and reactions which are due to admixture of traces of other substances must be carefully distinguished from those which are due to the principal salt. In cases where the subject of examination is a solid, it will be easy to infer roughly what amount of a precipitate should be obtained from the known quantity of it which has been taken to form the solution; and when a liquid is being examined, the amount of residue obtained by evaporating a known volume of it will be an indication of its strength. If, then, the addition of a reagent merely causes a turbidity in a solution which is known to be strong, it would be inexcusable thoughtlessness to take such a result as indicating the general constitution of the substance.

For instance, ordinary sodium carbonate invariably contains a little sulphate, and will, therefore, when tested with barium chloride, give a white precipitate which is not wholly soluble in hydrogen chloride. But the amount of the insoluble residue will be obviously disproportionate to the amount of the salt known to be present in the solution, and hence, while the fact is observed and recorded, the examination for another radicle is to be proceeded with.

TABLE

SHOWING THE DISTRIBUTION OF THE MORE IMPORTANT METALS INTO GROUPS FOR THE PURPOSES OF ANALYSIS.

These Metals are divided into

I. Metals which are separated from solutions by
Hydrogen Chloride.

SILVER
MERCURY (monatomic)
LEAD (partially)

Metals which are not separated from solutions by
Hydrogen Chloride.

II. Metals which are separated from solutions containing hydrogen chloride by
Hydrogen Sulphide.

LEAD
MERCURY (diatomic)
BISMUTH
COPPER
CADMIUM
ARSENIC
ANTIMONY
TIN
GOLD
PLATINUM

Metals which are not separated from solutions containing hydrogen chloride by
Hydrogen Sulphide.

III. Metals which are separated from solutions by
Ammonium Sulphide.

ZINC
MANGANESE
NICKEL
COBALT
IRON
CHROMIUM
ALUMINIUM

Metals which are not separated from solutions by
Ammonium Sulphide.

IV. Metals which are separated from solutions by
Ammonium Carbonate.

BARIUM
STRONTIUM
CALCIUM

Metals which are not separated from solutions by
Ammonium Carbonate.

V. Metal which is separated from solutions by
Ammonium Phosphate.

MAGNESIUM.

VI. Metals which are not separated from solutions by
Ammonium Phosphate.

POTASSIUM
SODIUM
AMMONIUM
HYDROGEN.

SECTION II.

PRELIMINARY EXAMINATION OF THE SUBSTANCE.

Before proceeding to the regular examination, in which solutions are chiefly employed, it will be expedient to try a few experiments of a general kind on the substance, in order to obtain some indication of its prominent characteristics, and of the best mode of attacking it. This part of the analysis should never be neglected. Many substances, e. g. mercury oxide (p. 139), may be analysed entirely and satisfactorily by its means, and there are comparatively few substances which will not give important indications of their character to a careful observer of their behaviour when heated with and without the assistance of the blowpipe.

A. *The Substance for Examination is a Solid.*

1.

Physical characteristics of the substance.

Examine and note down the physical characteristics of the substance; its colour, shape, hardness, metallic or non-metallic appearance, &c. If it is brittle and in a single lump, break it into small fragments in a mortar, or by wrapping it in a fold of clean paper and striking it with a hammer on an anvil. Reduce the greater part of these fragments to a very fine powder in the mortar, which should not be of glass unless the substance is a friable salt[1]. The powdered substance may be conveniently kept in a small dry test-tube, fitted with a cork.

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[1] For pulverising minerals, an agate mortar should, if possible, be used.

1. Solution of a Substance which is not a Metal.

2. **Solution in water.** Place about 0.5 grm. of the powder (as much as will lie on the broad end of a spatula) in a test-tube, pour on it about 10 c.c. of **water**, and heat the liquid to boiling, shaking it occasionally. The tube may be supported on the sand-bath resting in a ring of the retort-stand, and while it is being gradually heated, you may proceed to other experiments (p. 264).

(**A**) *The substance is entirely dissolved.*

Filter the solution, if necessary, from any suspended particles of dirt, and examine it according to **23** (p. 270).

(**B**) *The substance is not dissolved, or at least not wholly.*

Allow the particles to subside, decant the liquid carefully, and boil the residue with a fresh quantity of **water.** If it *dissolves*, add this solution to the first portion, and proceed to **23**. If it *does not dissolve*, pass on to the next paragraph.

3. **Solution in dilute hydrogen chloride.** Add to the liquid in which the substance insoluble in water is suspended, about eight or ten drops of concentrated **hydrogen chloride**, and if it does not dissolve, heat the mixture to boiling. If there is an *evolution of gas* it may be due to the presence of—

(α) A CARBONATE. In this case the gas will be nearly inodorous, and a drop of lime water on a glass rod held in the tube will be rendered turbid (p. 152).

(β) A SULPHIDE. If so, the gas will have the offensive smell of hydrogen sulphide, and a piece of paper moistened with solution of lead acetate and held within the tube will be blackened (p. 112).

(γ) A CHROMATE or PEROXIDE. The gas will have the smell of chlorine, and will bleach litmus-paper held in the tube.

(δ) A CYANIDE. The gas will have the characteristic smell of hydrogen cyanide.

(ε) A SULPHITE, or HYPOSULPHITE. If so, the gas will have the suffocating smell of sulphur dioxide. If the substance is a hyposulphite, sulphur will be deposited (**4 B**, *a*).

(**A**) *The substance is dissolved by the hydrogen chloride.* Examine the solution as directed in **34**.

(**B**) *The substance is not dissolved by the hydrogen chloride.* Pass on to the next paragraph.

4.

Solution in concentrated hydrogen chloride.

Take a fresh portion of the substance, add 6 or 7 c.c. of concentrated **hydrogen chloride**, and heat it to boiling[1].

(**A**) *The substance is dissolved.*

Dilute the solution with twice its bulk of water, and examine it as directed in **34**. If any precipitate is produced on dilution, add strong **hydrogen chloride**, drop by drop, until it disappears[2].

(**B**) *The substance appears to be acted upon, but there is a residue left, different in appearance to the original salt.*

Allow the residue to subside, pour off the solution and examine it after dilution (**4 A**) as directed in **34**. If the residue does not subside quickly, add to the solution about one-third its volume of water (that it may not act on the filter), filter it, and proceed to **34**.

The residue may consist of—

(*a*) SULPHUR. If so, it will be yellowish white, and remain long in suspension, being more easily filtered off after long boiling. It indicates the presence of a PERSULPHIDE, in which case hydrogen sulphide will be evolved (see **3** β); or of a HYPOSULPHITE, when sulphur dioxide will be evolved (**3** ε).

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[1] Concentrated hydrogen chloride will often, in the case of sulphides, chromates, and cyanides, produce an evolution of gas (**3**), where the dilute acid has failed to do so.

[2] It may be due to the presence of BISMUTH or ANTIMONY, since their chlorides are decomposed by water; possibly to SILVER CHLORIDE, since this substance is soluble in concentrated hydrogen chloride, but not in the dilute acid; possibly also to LEAD, since lead chloride is much less soluble in cold water than in hot, and the addition of water will have cooled the solution. In this last case the precipitate will redissolve when the liquid is warmed, and will be reprecipitated in needle-like crystals as it again becomes cool.

(β) SILICON HYDRATE, which is a white gelatinous substance, and will have been formed from the decomposition of a silicate. Wash the residue on a filter, dry it at a gentle heat, and heat some of it in a bead of microcosmic salt, as directed, p. 182. If it is silica it will float undissolved in the bead[1].

5. **Solution in aqua regia.**

(C) *The substance is unacted upon by concentrated hydrogen chloride.*

Add three or four drops of strong **hydrogen nitrate** to the solution containing hydrogen chloride, and boil the liquid[2].

(A) *The substance is dissolved.*

Boil the liquid until no more chlorine is evolved, dilute with water, and examine the solution as directed in **32.**

(B) *The substance is not dissolved.*

6. **Solution in hydrogen nitrate.**

Boil a fresh portion of it with strong **hydrogen nitrate.** If this *dissolves* it, dilute the solution and test it as directed in **32.** If it *does not dissolve* it, the substance must be examined as directed in Sect. V.

2. Solution of a Metal.

7. **Solution of a metal in hydrogen nitrate.**

Place a few small fragments in a test-tube, add about 6 or 7 c.c. of concentrated **hydrogen nitrate,** and heat it to boiling.

(A) *The substance dissolves.*

Dilute the solution with three or four times its volume of water, and examine it as directed in **32.**

(B) *The substance does not dissolve,* but is converted into a white powder. It is either ANTIMONY or TIN: the former if it is brittle and crystalline, the latter if it is malleable. It will dissolve in strong **hydrogen chloride** to which one drop of **hydrogen nitrate** has been added. Examine the solution as directed in **32.**

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[1] The residue may also possibly consist of silver chloride, formed by the decomposition of a silver salt by the hydrogen chloride. If so, it will be white and curdy, and become grey when exposed to the light.

[2] This should be done in a draught-cupboard, since chlorine is evolved.

8. **In aqua regia.** (C) *The substance is unacted upon.* It is probably either GOLD or PLATINUM; gold if it is yellow, platinum if it is white. It will dissolve in aqua regia, and the solution should be examined as directed in **32.**

3. Blowpipe Examination.

[This should be begun while the solution of the substance is being made, in order to economise time. If no characteristic results are obtained at once, and if the substance has entered readily into solution, the blowpipe examination may be continued at intervals during the testing of the solution, e. g. while a filtration is going on, or a precipitate is subsiding, and the results will be applicable as confirmatory evidence.]

9. **Substance heated in tube.** Place a small quantity of the substance in a tube of hard glass, sealed at one end (Fig. 13, p. 11), and heat it at first very gently over the Bunsen's burner, and afterwards before the blowpipe to a temperature as high as the tube will bear.

(A) *The substance remains unaltered, even at a high temperature.* Pass on to **16.**

(B) *The substance does not fuse, even when heated to redness, and gives off no gas or vapour, but changes colour.*

(*a*) *From white to yellow, becoming white on cooling.*

It is ZINC OXIDE, TIN DIOXIDE, or BISMUTH TRIOXIDE (probably the last, if it fuses at a strong red heat).

(*β*) *From red to black, turning red again on cooling.* It is probably IRON PEROXIDE.

(C) *The substance fuses at a very moderate heat, and possibly gives off water (of crystallisation).*

10. Wipe the inside of the tube perfectly dry with twisted slips of blotting-paper, and when no further moisture is condensed, raise the temperature.

(D) *The substance volatilises entirely.* It is a salt of AMMONIUM, ARSENIC, ANTIMONY, or MERCURY; or possibly HYDROGEN OXALATE, or SULPHUR. Observe the character of the sublimate in the tube.

(α) *It is white and amorphous.* The substance is an AMMONIUM salt, or a MERCURY salt (not, however, mercury iodide, sulphide or oxide). Pass on to **21.**

(β) *It is white and crystalline.* The substance is probably ARSENIC TRIOXIDE. Pass on to **21.**

(γ) *It is yellow, the original substance being red, and fusing to a nearly black liquid before volatilising. The sublimate becomes red when touched with a wire or glass rod.* It is MERCURY IODIDE[1].

(δ) *It is yellow, and amorphous, the original substance being also yellow.* It is ARSENIC SULPHIDE. Pass on to **32.**

(ε) *It forms brownish yellow, transparent drops, which do not solidify for some time.* It is SULPHUR. Persulphides, e. g. iron pyrites, give a sublimate of sulphur, while a residue of protosulphide remains behind.

11. To make quite sure that the sublimate consists of sulphur, cut off the closed end of the tube, and heat the sublimate gently, holding the tube in a slanting position. The sulphur will be oxidised in the current of air, and sulphur dioxide will be formed, which may be recognised by its smell and by its acid reaction on litmus-paper held at the upper end of the tube.

(E) *The substance becomes charred,* i. e. *turns black, and leaves a carbonaceous residue, while vapours are evolved which have a strong smell like burnt paper.* It is an ACETATE, or TARTRATE, or other organic salt[2].

12. As a further proof that the substance is organic, heat a little of it in a test-tube with some strong hydrogen sulphate. Tartrates, and most other

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[1] This reaction is so characteristic of mercury iodide that the substance may be at once examined for mercury and iodine. For this purpose, a little of the substance should be decomposed by boiling it with some solution of potassium hydrate. A solution containing potassium and mercury iodide will be obtained, which should be acidified with hydrogen sulphate and tested for iodine, as directed in p. 168, Expt. 9. The residue, which consists of mercury protoxide, should be washed by decantation, dissolved in dilute hydrogen chloride, and tested for mercury with tin protochloride (p. 208, Expt. *d*).

[2] Some other substances, e. g. COPPER CARBONATE, MANGANESE CARBONATE, turn black when heated, but do not evolve strong-smelling vapours.

organic substances (not, however, acetates) turn black when thus treated.

(**F**) *The substance does not become charred, but evolves a gas, with or without previous fusion.* This gas is—

(*a*) *Colourless, and has the peculiar suffocating smell of sulphur dioxide, and reddens a piece of blue litmus-paper held in the mouth of the tube.* This indicates the presence of a SULPHATE decomposable by heat.

(*β*) *Colourless, and has the peculiar smell of cyanogen, resembling bitter almonds.*

13. Hold a lighted match to the mouth of the tube. If the gas burns with a pink flame, a CYANIDE is present.

(*γ*) *Colourless and odourless.*

14. Place a single drop of lime water on a watch-glass, and hold it close to the mouth of the tube (which should be as far as possible from the lamp-flame). If the drop becomes turbid, the gas is CARBON DIOXIDE, and proceeds from the decomposition of a CARBONATE or an OXALATE.

15. If carbon dioxide is not detected, drop a small splinter of charcoal from the charred end of a match into the tube, and again heat the end of the tube. If the charcoal burns vividly, the gas is OXYGEN and the substance is a CHLORATE, NITRATE, CHROMATE, or PEROXIDE[1].

(*δ*) *Orange-coloured,* and reddens litmus-paper. The substance is probably a NITRATE; if so, a splinter of charcoal dropped into the tube will deflagrate as in the case of chlorates.

Some few bromides and iodides are decomposed by heat alone, and give off orange vapours of bromine, which condense to an orange liquid, or violet vapours of iodine, which condense in the form of steel-grey flakes.

[Hypophosphites, when heated, give off hydrogen phosphide, which inflames spontaneously at the mouth of the tube.]

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[1] AMMONIUM CHROMATE, however, gives off, not oxygen, but water and nitrogen, and a light, bulky, olive-green residue of chromium oxide is left.

(G) *The substance fuses to a clear transparent liquid, when heated to redness.* It is probably a SALT OF AN ALKALI METAL, or BARIUM STRONTIUM or CALCIUM CHLORIDE.

The course next to be followed will depend upon the appearance of the substance and the results obtained by heating it in a tube.

(A) *The substance is coloured.* If so, it is extremely probable that it will impart colour to a borax bead.

16.
Examination in borax bead.

Fuse some **borax** in a loop of platinum wire, as directed in p. 135, and when a clear transparent bead has been obtained, bring into it a minute portion of the powdered substance and heat it first at the tip of the blowpipe flame, then in the oxidising flame, and lastly in the reducing flame. Observe the colour, if any, which is imparted to the bead.

(α) *It is blue, in both oxidising and reducing flames.* The substance is a COBALT salt.

(β) *It is greenish blue in the oxidising flame, and becomes almost colourless in the reducing flame. If much of the substance has been added, the bead becomes red and opaque in the reducing flame.* The substance is a COPPER salt.

(γ) *It is green, both in the oxidising and reducing flame.* The substance is a CHROMIUM salt.

(δ) *It is orange-red in the oxidising flame, becoming light yellow as it cools. In the reducing flame it is orange while hot, and dull bottle-green when cold.* The substance is an IRON salt.

(ε) *It is amethyst-red in the oxidising flame, and becomes quite colourless in the reducing flame.* The substance is a MANGANESE salt.

(ζ) *It is brownish red in the oxidising flame, and becomes grey and turbid in the reducing flame.* The substance is a NICKEL salt.

(η) *It is light yellow in the oxidising flame, and becomes colourless in the reducing flame.* The substance is a BISMUTH or SILVER salt.

(B) *The substance is colourless, or if coloured has given no reaction with a borax bead, and is infusible, or difficultly fusible.*

17.
Reduction on charcoal.

Mix a small portion of it with an equal quantity of **sodium carbonate**, and heat it on charcoal, as directed in p. 137 [1].

(*a*) *An infusible white residue is left on the charcoal.* If this residue is *strongly luminous* while held in the blowpipe-flame, MAGNESIUM, STRONTIUM, or CALCIUM is probably present.

18.

Moisten the residue with one drop of solution of **cobalt nitrate**, and heat it again strongly for a few seconds. If on cooling the residue has a *blue colour*, ALUMINIUM is present; if it has a *faint pink tinge*, MAGNESIUM is present; if it is *green*, ZINC is present.

19.
Colour imparted to flame.

If no characteristic result is obtained, moisten the end of a perfectly clean platinum wire with solution of hydrogen chloride, dip the wire into the powdered substance, and hold it at the edge of the flame of a Bunsen's burner. Observe the colour, if any, imparted to the flame.

(*aa*)	It is	*yellow.*	The substance is a	SODIUM	salt.
(*ββ*)	„	*violet.*	„ „	POTASSIUM	„
(*γγ*)	„	*green.*	„ „	BARIUM	„
(*δδ*)	„	*crimson.*	„ „	STRONTIUM	„
(*εε*)	„	*orange-red.*	„ „	CALCIUM	„

[It must be borne in mind that traces of sodium are almost invariably present in a substance, and that the intense yellow flame due to its presence will often mask other colours. This difficulty is especially felt in the case of potassium salts; the barium, strontium, and calcium salts are so much less volatile that they remain long after the traces of sodium have volatilised. A transient yellow tinge will almost always be imparted to the flame when the salt is first introduced into it; and when this has passed away, the other characteristic colours should be

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[1] The substance should be held just within the tip of the blowpipe-flame, and the blast should be pretty strong. As we have such powerful reducing agents present as sodium carbonate and charcoal, we can afford to sacrifice some of the reducing power of the blowpipe-flame itself, for the sake of getting as high a temperature as possible.

looked for. In order to detect potassium the flame should be looked at through a piece of deep blue glass. If sodium alone is present, the flame will be invisible or very pale blue, the yellow rays being absorbed by the glass. If potassium is present, the flame when thus looked at will appear deep crimson.]

(β) *Bright metallic globules are obtained.*

20. Continue the heat, with addition, if necessary, of a little more **sodium carbonate** or **potassium cyanide**, until the small globules have run together into one large one, and the flux has disappeared. When the metal is cool[1], detach it from the charcoal with a knife, and try its malleability, as directed in p. 137, Expt. 8, noting also if any incrustation has formed on the charcoal in or around the cavity[2].

(αα) *The metallic globule is malleable, with brilliant lustre, and there is no incrustation on the charcoal.* The substance is a SILVER salt.

(ββ) *The globule is malleable, and a slight white incrustation is formed on the sides of the cavity.* The substance is a TIN salt.

(γγ) *The globule is malleable and soft, and a yellow incrustation is formed on the charcoal.* The substance is a LEAD salt.

(δδ) *The globule is brittle, and white fumes ascend from it while hot; a white incrustation is formed on the charcoal.* The substance is an ANTIMONY salt.

(εε) *The globule is brittle, and a yellow incrustation is formed.* The substance is a BISMUTH salt[3].

(C) *The substance has volatilised entirely in the glass tube, and*

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[1] The cooling may be hastened by very cautiously dropping water upon the charcoal at a little distance from the cavity, not into the cavity itself.

[2] Care must be taken not to mistake the white ash, which charcoal always leaves when burnt, for an incrustation of metallic oxide. The former is light, almost downy, in appearance; the latter is more compact, and generally has a definite border.

[3] The globule of bismuth, although in reality brittle, does not always crumble to pieces under the pestle or hammer; the particles may remain aggregated so as to appear not unlike a flattened plate of malleable metal. But the edges of the plate are always jagged, as may be readily seen with a magnifier, and a blow or two of the pestle is generally sufficient to break it into several pieces.

the character of the sublimate has led to the inference that a mercury or arsenic salt is present.

21.
Reduction in tube.

Mix a small quantity of the original substance with an equal quantity of **sodium carbonate,** introduce into a sealed tube enough of the mixture to nearly fill the bulb, and heat it slowly over the lamp. If any moisture condenses, wipe the tube dry with slips of blotting-paper.

(*a*) *A bright metallic sublimate is formed, which may be seen with a magnifier to consist of liquid metallic globules.* The substance is a MERCURY salt.

To make quite sure of the character of the sublimate, pass down into the tube a thin slip of wood, such as a match, and scrape together the sublimed metal. If it is mercury, the particles will run together into one large globule, which may be allowed to drop into a watch-glass.

(*β*) *It is a steel-grey lustrous mirror.* The substance is an ARSENIC salt.

Break off the tube close to the bulb, hold it horizontally, and heat the part containing the sublimate over a spirit lamp with a very small flame. If the deposit is arsenic, it will readily volatilise and become oxidised, condensing in the cool parts of the tube in sparkling transparent crystals of arsenic trioxide.

B. *The Substance for Examination is a Liquid*[1].

Observe and note down the colour and smell of the liquid.

22.

Place about 2 or 3 c.c. of the liquid in a watch-glass and set it on the sand-bath to evaporate to dryness, while other experiments with the solution are proceeded with. The residue, if any, should be examined

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[1] It should be noticed that the liquids with which the student will have to deal are in all cases solutions of substances in water, even where an acid has been used to dissolve them; the reaction then consisting in the formation of a soluble compound and its solution in water.

before the blowpipe according to the directions already given, and if the nature of the liquid has been discovered before the residue is obtained, the results of the blowpipe examination will serve as confirmatory evidence.

23. Dip a clean platinum wire into the solution, and hold it at the border of the flame of a Bunsen's burner. If any characteristic colour is imparted to the flame, refer to **19**.

24. Reaction on litmus-paper. Take out a drop on a glass rod and place it upon a piece of **blue litmus-paper.** If no effect is produced, place another drop upon a piece of **reddened litmus-paper.**

(**A**) *The liquid reddens blue litmus-paper.*

This may be due to the presence either of a HYDROGEN salt or of a double salt of HYDROGEN and a METAL, or lastly of certain metallic salts, e. g. COPPER or MERCURY salts, which, although containing no basic hydrogen, have an acid reaction.

25. To distinguish between these, place a little of the liquid in another test-tube, add a trace of solution of sodium carbonate on the end of a glass rod, and stir the mixture. If no precipitate is produced, or the precipitate redissolves immediately, a hydrogen salt is present. If a permanent turbidity is produced, the acid reaction is due to the presence of a metallic salt, soluble in water.

(**B**) *The liquid turns reddened litmus-paper blue.*

This may be due to the presence of a BORATE, SILICATE, HYDRATE, CARBONATE, SULPHIDE, or CYANIDE of a member of Group VI, or of a HYDRATE or SULPHIDE of a member of Group IV or V.

26. Action of hydrogen chloride on alkaline solutions. Pour about one-half of the solution into a test-tube and add dilute **hydrogen nitrate,** drop by drop, until the liquid is strongly acid.

(*a*) *No precipitate or evolution of gas occurs.* The substance is a HYDRATE, ARSENITE, or ARSENATE, of a member of Group IV, V, or VI. Pass on to **32**.

(*β*) *No gas is evolved, and any precipitate which is produced* *redissolves in the excess of hydrogen chloride.* The substance contains one of the metals which form soluble double oxides or hydrates with an alkali metal[1].

Pass on to **32**, bearing in mind that an alkali metal must be sought for, as well as one of the others above mentioned.

(γ) *No gas is evolved, but a gelatinous precipitate is formed which does not dissolve in the excess of acid.* The substance is a SILICATE.

27. Evaporate the solution, containing excess of hydrogen nitrate, to complete dryness, digest the residue with a little strong hydrogen chloride, stirring it with a glass rod. After adding a little water, filter the solution and examine the filtrate for an alkali metal (**50**). To be quite sure that the residue is silicon oxide, test it in a bead of microcosmic salt, as directed in p. 182.

(δ) *A gas is evolved, which is inodorous and renders lime water turbid.* The substance is an ALKALI-METAL CARBONATE.

28. Boil until the carbon dioxide is expelled, and test the solution as directed in **50**.

(ϵ) *A gas is evolved, which possesses the peculiar odour of hydrogen cyanide.* An ALKALI-METAL CYANIDE is present. If a *permanent precipitate* is formed, the substance contains one of the metals which form soluble double cyanides with an alkali metal.

29. Add a few drops of strong hydrogen chloride, boil to get rid of hydrogen cyanide, and examine the solution as directed in **34**, remembering that POTASSIUM or SODIUM is present, as well as the other metal. If a residue remains undissolved even after boiling with hydrogen chloride, examine it as directed in Sect. V. To make quite sure that the substance is a cyanide, examine a fresh portion, as directed in p. 157, Expt. 2.

(ζ) *A gas is evolved, which may be recognised, by its odour and its action on lead acetate, to be hydrogen sulphide.*

(*aa*) *No precipitate, or only a white milky precipitate of sulphur, is produced.*

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[1] A list of these metals is given at the foot of the Table of Solubilities, Appendix C.

The substance is a SULPHIDE of a metal belonging to Groups IV, V, or VI.

30. Boil the solution in a dish for a few minutes, filter if necessary, and examine the filtrate as directed in **47**.

($\beta\beta$) *A coloured precipitate is produced.* It may consist of TIN, ARSENIC, or ANTIMONY SULPHIDE, formed by the decomposition of a double sulphide.

31. Filter the solution, and test the filtrate for an alkali metal; wash the precipitate, dissolve it in strong hydrogen chloride, to which one drop of hydrogen nitrate has been added, and test the solution as directed in **34**.

SECTION III.

EXAMINATION OF THE SOLUTION OF A SINGLE SALT FOR A METAL.

1. Examination for the Metals included in Group I.

SILVER, MERCURY (protosalts), LEAD.

A. *The Substance was originally given in Solution, or has been dissolved in Water or Hydrogen Nitrate.*

32. **Hydrogen chloride test.** Add **dilute hydrogen chloride**, drop by drop, to a portion of the cold solution. If a precipitate is obtained, add six or eight more drops of the acid, in order to ascertain if the precipitate is soluble in excess.

(A) *No precipitate, or a precipitate which dissolves in excess of the acid, is obtained*[1].

SILVER salts, MERCURY protosalts, and also LEAD salts, unless the solution is very dilute, are absent. Pass on to **34.**

(B) *The fluid gradually becomes milky, and evolves sulphur dioxide* (recognisable by its odour).

This is due to the presence of a HYPOSULPHITE, of which

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[1] Such a precipitate will be probably due to the presence of a BISMUTH or ANTIMONY salts, since bismuth and antimony chlorides (which will be formed on addition of hydrogen chloride) are more easily decomposed by water than their other salts. If the solution is yellow and turns red on addition of hydrogen chloride, a CHROMATE is present.

the reaction with **silver nitrate** (p. 174) will afford conclusive proof.

The solution, after boiling, should be filtered from the sulphur, and tested as directed in **34.**

(C) *A permanent white precipitate is formed.*

A metal belonging to Group I is present.

33. Add to the fluid in which the precipitate is suspended, **ammonia** in excess.

(α) *The precipitate is redissolved.*

SILVER is present. This may be also inferred if the precipitate is curdy, and becomes grey on exposure to light. As a confirmatory test, add to another portion of the original solution (which, if acid, must be neutralised by ammonia) a drop of solution of **potassium chromate**, which will cause a crimson precipitate, if silver is present.

(β) *The precipitate turns black.*

A MERCURY protosalt is present. To confirm this, place a clean strip of **copper** in another portion of the original solution, acidified, if necessary, with dilute hydrogen chloride. If the copper is coated with a grey deposit, which becomes silvery-bright when rubbed with a cloth, and disappears when heated over a lamp, the presence of mercury may be considered certain.

(γ) *The precipitate remains unaltered in appearance.*

LEAD is present. To confirm this, test another portion of the original solution with **dilute hydrogen sulphate**: a white precipitate will be produced if lead is present.

B. *The Substance was dissolved in Hydrogen Chloride.*

In this case neither silver salts nor mercury protosalts can be present. Lead may be present in small quantity, since lead chloride is not quite insoluble, and it should be looked for in the next part of the examination.

2. Examination for the Metals included in Group II.

MERCURY (persalts), LEAD, COPPER, BISMUTH, CADMIUM, TIN, ARSENIC, ANTIMONY, GOLD, PLATINUM.

34. **Hydrogen sulphide test.** Add to the portion of the solution which has been acidified with hydrogen chloride, solution of **hydrogen sulphide** in excess. Observe whether the addition of a single drop of the reagent produces a precipitate, and if so, whether the precipitate is altered in appearance when more of the reagent is added[1]. If no precipitate is produced at once, warm the solution.

(A) *No precipitate is formed.*

None of the metals included in Group II are present. Pass on to **40**.

(B) *A perfectly white, milky precipitate is produced.* This consists solely of SULPHUR, and is due to the decomposition of the hydrogen sulphide by some oxidising substance present in the solution, probably a CHROMATE or an IRON PERSALT. If the solution is red or yellow, and turns light green simultaneously with the precipitation of sulphur, one of the above two substances is certainly present; they will, however, be detected in the course of examination for Group III (**40**), to which you should pass on.

The decomposition of the hydrogen sulphide may also be due to the presence of CHLORINE (if aqua regia has been used to dissolve the substance), a HYPOCHLORITE, a CHLORATE, a NITRITE, or a SULPHITE. These will, even if not detected in the preliminary examination, be examined for in Sect. IV. Bear in mind the result, therefore, but proceed with the examination as if the precipitate had not occurred.

(C) *The precipitate is light yellow.*

CADMIUM, ARSENIC, or TIN (as persalt) is present.

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[1] The precipitate produced in solutions of mercury persalts by the first drop of the solution of hydrogen sulphide is white, and quickly changes to yellow, brown, and finally black, on further addition of the reagent.

35. Add to the fluid in which the precipitate is suspended, **ammonia** in slight excess, then three or four drops of solution of **ammonium sulphide**, and warm the mixture.

(*a*) *The precipitate dissolves.*

TIN or ARSENIC is present. To distinguish between them, add a few drops of **ammonia** to a portion of the original solution. If *a white precipitate is produced*, TIN is present; the reduction of the salt before the blowpipe will afford confirmatory evidence. If *no precipitate is obtained*, ARSENIC is present; the production of a mirror-like sublimate when the substance is heated in a bulb-tube with **sodium carbonate** (p. 185) will remove any doubt.

If arsenic is found, the substance may be an ARSENITE or ARSENATE; if the latter, the precipitate caused by hydrogen sulphide will have formed only on boiling the liquid. Refer to p. 184, Expts. *b*, *d*, and p. 190, Expts. *b*, *d*, for other means of distinguishing between them.

(*β*) *The precipitate does not dissolve.*

CADMIUM is present. Confirm this inference by applying the blowpipe test (p. 214, Expt. 1).

(D) *The precipitate is orange-coloured.*

ANTIMONY is present. Confirm this by repeating Expt. 5, p. 192, with a portion of the original solution.

(E) *The precipitate is dark brown or black.*

MERCURY, LEAD, COPPER, BISMUTH, TIN (protosalt), GOLD, or PLATINUM is present[1].

36. (*a*) Add to the fluid in which the precipitate is suspended, a slight excess of **ammonia**, then (disregarding any additional precipitate which may possibly be caused by the ammonia) add five or six drops of solution of **ammonium sulphide**, and heat the mixture. If *the precipitate is dissolved*, TIN is present (or possibly GOLD or PLATINUM). In confirmation, (1) add to the solution thus obtained some **dilute hydrogen chloride**, which will, if tin is present,

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[1] If the solution has a blue or green colour, copper may be at once tested for as directed in **39**.

produce a light yellow precipitate of tin persulphide; (2) add a drop of the original solution to some solution of **mercury perchloride**, which latter will be reduced to protochloride and form a white precipitate[1].

37. (β) Test a portion of the original solution with dilute hydrogen sulphate. If *a white precipitate is produced,* LEAD is present. The reduction of the substance on charcoal before the blowpipe (p. 209) will serve as a confirmatory test.

38. (γ) Place a clean slip of **copper** in a portion of the original solution, acidified, if necessary, with dilute hydrogen chloride. The formation of *a grey metallic film* on the copper proves that MERCURY is present (see **33** β). The changes in colour of the precipitate produced by addition of successive portions of hydrogen sulphide to the original solution (see p. 276, note), will have also indicated the presence of mercury, and the result of heating the dry substance with **sodium carbonate** in a tube (**21**) will afford absolute certainty.

39. (δ) Add to a portion of the original solution, **ammonia** in excess.

If a precipitate is at first formed which readily dissolves in excess of ammonia, forming a deep blue solution, COPPER is present. To remove any doubt, test another portion of the original solution with solution of **potassium ferrocyanide**, which will give a reddish brown precipitate of copper ferrocyanide.

If a white precipitate is produced which does not dissolve in excess of the ammonia, BISMUTH is present. Add to the liquid **dilute hydrogen chloride**, drop by drop, until the precipitate is just redissolved, then fill up the test-tube with water. *If the fluid becomes milky*, the presence of BISMUTH is confirmed. As a further proof, a portion of the original substance may be mixed with some **sodium carbonate** and reduced on charcoal before the blowpipe (see p. 138).

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[1] It is scarcely probable that gold and platinum will be met with; but if tin is not detected by the above tests, separate portions of the original solution should be tested (*a*) for GOLD, with solution of **iron protosulphate** (p. 197), (*b*) for PLATINUM, with solution of **ammonium chloride** (p. 198).

3. Examination for the Metals included in Group III.

ALUMINIUM, IRON, CHROMIUM, MANGANESE, NICKEL, COBALT, ZINC.

40. **Ammonium sulphide test.** To the portion of the solution to which hydrogen sulphide has been added without producing a precipitate (other than sulphur), add one-fourth its volume of solution of **ammonium chloride**, then solution of **ammonia** until the liquid smells of it strongly, and lastly, disregarding any precipitate which may have been formed by the ammonia, add three or four drops of solution of **ammonium sulphide**, and warm the mixture.

(A) *No precipitate is produced.*

None of the metals included in Group III are present. Pass on to 47.

(B) *A black precipitate is produced.*

IRON, NICKEL, or COBALT is present.

41. Add to a portion of the original solution some solution of **potassium hydrate**.

(*a*) *A dull green precipitate is formed, which becomes nearly black, and finally reddish brown when shaken up for a short time in the tube, in order to expose it to the air.*

An IRON protosalt is present. In confirmation, test another portion of the original solution with solution of **potassium ferricyanide**, which should produce a deep blue precipitate (p. 219, Expt. *e*).

(β) *A light green precipitate is produced, which does not alter in colour when exposed to the air or when the solution is boiled.*

NICKEL is present. The colour imparted to a borax bead by a portion of the substance will be the best confirmatory test (16 ζ).

(γ) *A light blue precipitate is formed, which slowly turns* *green in the air, and becomes reddish brown when the solution is boiled.*

COBALT is present. The blue colour imparted to a borax bead by a little of the substance will afford absolute certainty (**16** *a*).

(*δ*) *A reddish brown precipitate is formed.*

An IRON persalt is present. If so, hydrogen sulphide will have produced a precipitate of sulphur (**34 B**). Confirm this by testing another portion of the original solution with solution of **potassium ferrocyanide**, which will produce a deep blue precipitate of Prussian blue.

(**C**) *A dull pink precipitate is formed.*

MANGANESE is present. The colour imparted to a bead of borax or sodium carbonate will afford confirmatory evidence (**16** *ε*).

(**D**) *A light bluish green flocculent precipitate is formed.*

CHROMIUM is present. The colour imparted to a borax bead will confirm this (**16** *γ*).

42. It remains, however, to be decided whether the chromium was present as a chromate or as a chromium salt. If the original solution was red (or yellow, becoming red on addition of hydrogen chloride), and if when mixed with hydrogen sulphide it became green, with simultaneous precipitation of sulphur, the substance is a chromate, and another metal has yet to be examined for, which may be included in Group III, IV, V, or VI.

If the salt is soluble in water, it will be STRONTIUM, CALCIUM, MAGNESIUM, POTASSIUM SODIUM, or AMMONIUM chromate. Proceed with the examination, as directed in **47**, since the presence of chromium will not interfere with the tests.

If the salt is insoluble in water, it may be ZINC or BARIUM chromate. (Other insoluble chromates in these groups are not likely to occur.)

Add to a portion of the original solution, excess of solution of **potassium hydrate**.

(*a*) *A precipitate is produced which does not redissolve in excess of potassium hydrate.*

BARIUM is present. This should be confirmed by testing a portion of the original solution with solution of **calcium sulphate** (48 *a*).

(β) *A precipitate is produced which dissolves in excess of potassium hydrate.*

ZINC is present. To confirm this, add to the same solution (containing excess of potassium hydrate) some solution of **hydrogen sulphide**, which, if zinc is present, will produce a white precipitate of zinc sulphide.

(E) *A white precipitate is formed*[1].

The next step must depend upon whether the original substance was soluble in water, or insoluble in water, but soluble in hydrogen chloride; since in the latter case certain salts of metals belonging to Groups IV and V, which are soluble in dilute hydrogen chloride, but are reprecipitated when the solution is neutralised, may be present.

1. The salt was soluble in water.

ZINC, ALUMINIUM, or SILICON (a silicate) is present.

43. Add to a portion of the original solution one drop of solution of **potassium hydrate**. If this produces a precipitate, add more potassium hydrate, until the precipitate is redissolved.

(*a*) *A precipitate is produced, soluble in excess of potassium hydrate.*

ZINC or ALUMINIUM is present.

44. Test a portion of the solution (containing excess of potassium hydrate) with **hydrogen sulphide**. If this *produces a white precipitate*, ZINC is present. This should be confirmed by the application of the blowpipe test (p. 232, Expt. 3). If *no precipitate is produced by the hydrogen sulphide*, ALUMINIUM is present. To make certain of its presence add to another portion of the solution about half its volume of solution of **ammonium chloride**, and apply heat. The

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[1] It must be remembered that if the ammonium sulphide is yellow, and if enough has been added to colour the solution, a white precipitate will appear light yellow when looked at through the yellow solution.

formation of a gelatinous precipitate will confirm the presence of aluminium.

(β) *No precipitate is formed.*

SILICON is present, and the salt is in all probability potassium or sodium silicate, since the other silicates are insoluble in water.

Examine another portion of the original solution as directed in **27**, if this has not been already done.

2. The salt was insoluble in water, and soluble in hydrogen chloride.

45. Add to another portion of the original solution about one-third its volume of solution of **sodium and hydrogen tartrate**, and then add a slight excess of **ammonia.**

(**A**) *No precipitate is produced.*

ZINC or ALUMINIUM is present, and should be tested for as directed in **43**[1].

(**B**) *A permanent white precipitate is formed.*

The substance is an OXALATE, a PHOSPHATE, or a FLUORIDE, the other radicle being BARIUM, STRONTIUM, CALCIUM, or MAGNESIUM. The colour imparted by the substance to flame (**19**) will, if one of the first three is present, have already given evidence of it.

46. Add to another portion of the original solution an excess of solution of sodium carbonate, and boil the mixture for five or six minutes. This will (partially or wholly) decompose the salt, the metal being precipitated as an insoluble carbonate, while the other radicle remains in solution as a sodium salt. While this is going on you may examine other portions of the original solution for an oxalate, a phosphate, and a fluoride, as directed in **57** and **58**[2].

Filter the solution containing excess of sodium carbonate,

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[1] Aluminium phosphate much resembles aluminium hydrate in properties. If the presence of aluminium is ascertained, test another portion of the solution for a phosphate, as directed in p. 179 *c*.

[2] If a fluoride is found, it will be best to test for the metal in the residue left in the platinum vessel (p. 297, note 2), since fluorides are with difficulty decomposed by sodium carbonate.

wash the precipitate on the filter several times with warm water, then pour over it a little **dilute hydrogen chloride**, and pour the fluid which runs through again on the filter, until the whole of the precipitate is dissolved. The solution, which contains the metal as chloride, should be mixed with a slight excess of ammonia, filtered from any precipitate formed, and tested as directed in **47**.

4. Examination for the Metals included in Group IV.

BARIUM, STRONTIUM, CALCIUM.

47. **Ammonium carbonate test.** To the portion of the solution in which ammonium sulphide has produced no precipitate, and which also contains ammonium chloride and ammonia, add a few drops of solution of **ammonium carbonate**, and apply heat.

(A) *No precipitate is formed.*

None of the metals included in Group IV are present. Pass on to **49**.

(B) *A white precipitate is formed.*

BARIUM, STRONTIUM, or CALCIUM is present.

48. Add to a fresh portion of the original solution an equal volume of solution of **calcium sulphate.**

(α) *A white precipitate is produced immediately.*

BARIUM is present. As a confirmatory test, add to another portion of the solution some **hydrogen and silicon fluoride,** which will produce a crystalline precipitate if barium is present. Observe also the colour imparted by the original substance to flame (**19**).

(β) *No immediate precipitate is formed.*

STRONTIUM or CALCIUM is present.

Allow the solution to stand for four or five minutes, and then, if no turbidity appears, warm it.

(γ) *A precipitate is formed after the lapse of some time.*

STRONTIUM is present. The carmine-red colour imparted to flame will be a conclusive proof.

(δ) *No precipitate is formed, even after the solution has been allowed to stand and warmed.*

CALCIUM is present. In confirmation, add to a fresh portion of the original solution enough **ammonia** to render it alkaline, and then a drop of solution of **ammonium oxalate**, which should produce a white precipitate [1]. Observe also if the substance imparts a brick-red colour to flame.

5. Examination for the Metal included in Group V.

MAGNESIUM.

49. **Sodium phosphate test.** To the portion of the solution in which ammonium carbonate produced no precipitate, and which also contains other ammonium salts, add a drop of solution of **sodium phosphate**, and, if no precipitate is produced, rub the sides of the tube gently with a glass rod.

(A) *No precipitate is formed.*

MAGNESIUM is absent. Pass on to **50.**

(B) *A crystalline precipitate is formed.*

MAGNESIUM is present. In confirmation, apply the blowpipe test (p. 240 *e*).

6. Examination for the Metals included in Group VI.

POTASSIUM, SODIUM, AMMONIUM, HYDROGEN.

50. To a fresh portion of the original solution placed in a small beaker, add a little **calcium hydrate** or **oxide**, cover the beaker with a watch-glass on which a slip of **reddened litmus-paper** has been placed (p. 150 *b*), and warm the mixture very gently for half a minute.

(A) *The litmus-paper is turned blue.*

AMMONIUM is present. In confirmation, observe the odour

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[1] It must be borne in mind that this result is only characteristic of the presence of a calcium salt under the circumstances here mentioned; since a barium or strontium salt would, if present, yield a similar precipitate.

of the gas, and see whether white fumes are formed when a glass rod dipped in **hydrogen chloride** (or **acetate**) is held within the beaker. The original substance will also have been found wholly volatile.

(B) *The litmus-paper is unaltered.*

POTASSIUM, SODIUM, or HYDROGEN is present.

51. Add to another portion of the original solution, rendered slightly acid, if necessary, by hydrogen chloride, a drop or two of solution of **platinum perchloride,** and, if no precipitate is formed, evaporate the solution to dryness in a watch-glass and treat the residue with dilute alcohol (p. 243 *d*).

(A) *A yellow crystalline precipitate, or insoluble residue, is obtained.*

POTASSIUM is present. To remove any doubt, test another portion of the solution (concentrated, if necessary) with solution of **sodium and hydrogen tartrate** (p. 243 *e*), and also observe if the substance imparts a violet colour to flame (p. 243 *a*).

(B) *No precipitate or insoluble residue is obtained.*

SODIUM or HYDROGEN is present.

52. To test for the former metal, place a little of the original substance in a loop of platinum wire, and hold it in the flame of a Bunsen's burner. If an intense yellow colour, lasting for some time, is imparted to the flame, sodium is present.

If sodium is not found, and no other metal has been detected in the previous stages of the analysis, the basic radicle is probably HYDROGEN.

Refer to p. 245 for other means of ascertaining this, and proceed with the examination for the other radicle (Sect. IV).

SECTION IV.

EXAMINATION OF THE SOLUTION OF A SINGLE SALT FOR A NON-METALLIC RADICLE.

53. The first point to consider is, whether the substance has been found to be soluble in water or in acids.

If it is soluble in **water**, the radicle sought must be one of those which form compounds soluble in water with the metal which is known to be present. If it is insoluble in water, but soluble in **acids**, the radicle must be one of those which form compounds soluble in acids only, with the metal which is known to be present.

The Table in Appendix C will show what radicles may be inferred to be present from the solubility or insolubility of a substance.

Much information will have been gained from the preliminary examination (Sect. II); and the following radicles will, if present, have been certainly detected already in the course of it:—

NITRATE (15), CARBONATE (14), CYANIDE (13), SULPHIDE (3), CHLORATE (15), SILICATE (4), TARTRATE or ACETATE (11), HYPOSULPHITE (4 B *a*).

However, even if the presence of one of the above radicles has been indicated, it will still be advisable to confirm the result by the application of reagents in solution, as follows.

A. *The Salt is Soluble in Water.*

1. Examination for the Radicles included in Group I.

CARBONATE, (TARTRATE), OXALATE, (FLUORIDE), SULPHITE, SULPHATE, PHOSPHATE, (BORATE), SILICATE[1].

54. **Barium chloride test.** Add to a portion of the concentrated aqueous solution (which, if acid, must be made neutral or slightly alkaline by ammonia), a few drops of solution of **barium chloride.**

If lead, silver, or mercury as protosalt, is present, **barium nitrate** must be used instead of barium chloride, since chlorine, as we have seen, forms insoluble compounds with these metals[2].

(A) *No precipitate is produced.*

The salt is not a SULPHATE, SULPHITE, CARBONATE, PHOSPHATE, or OXALATE, and probably not a TARTRATE, FLUORIDE, BORATE, or SILICATE.

It will be advisable to test for the last four radicles, even though no precipitate is obtained with barium chloride, since barium borate and tartrate are decidedly soluble in water, and the precipitate of barium silicate or fluoride is transparent and gelatinous, and might be overlooked. If these radicles are not found to be present, pass on to **59.**

(B) *A white precipitate is produced.*

55. Add an excess of **dilute hydrogen nitrate** to the liquid in which the precipitate is suspended.

(*a*) *The precipitate remains undissolved.*

The salt is a SULPHATE. To confirm this, add to a fresh

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[1] The radicles which are included in brackets are only partially precipitated from solutions by barium chloride.

[2] A little barium nitrate for this purpose may be readily prepared by dissolving a small quantity of barium oxide, hydrate, or carbonate, in dilute hydrogen nitrate.

portion of the original solution, a drop of solution of LEAD ACETATE, which will produce a white precipitate if a sulphate is present. The blowpipe test (p. 138) may also be tried.

(β) *The precipitate dissolves.*

Pass on to the next paragraph.

56. Add to another portion of the original solution (rendered slightly alkaline), a few drops of solution of **ammonium chloride**, and then some quantity of solution of **calcium sulphate** or of **calcium chloride**[1].

(**A**) *No precipitate is produced.*

The salt is a BORATE or TARTRATE.

(α) If *the salt, when heated in a glass tube* (**11**), *blackened and gave off vapours smelling like burnt sugar*, it is a TARTRATE. To confirm this, add to a portion of the original solution (neutralised if necessary) some solution of silver nitrate, and boil it. If a tartrate is present, the solution will become black and turbid, owing to reduction of the silver salt (p. 154, 3 *c*).

(β) If *the salt did not blacken when heated*, it is probably a BORATE. Place a little of the original solution in a watch-glass, acidify it with hydrogen chloride, dip into the liquid a piece of turmeric-paper, and dry it at a gentle heat. If the dipped portion becomes red, a borate is present. The colour imparted by the salt to the flame of alcohol (p. 181, 3) may be also tried, unless the metal present is copper or barium.

(**B**) *A white precipitate is formed.*

The salt is an OXALATE, FLUORIDE, PHOSPHATE, SILICATE, or CARBONATE (the two last radicles will, if present, have been detected).

57. Add to the solution containing the precipitate in suspension, a few drops of **hydrogen acetate.**

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[1] **Calcium sulphate** must not be used if the substance is a LEAD, BARIUM, or STRONTIUM salt.

Calcium chloride must not be used if the substance is a SILVER or LEAD salt, or a MERCURY protosalt.

Solution of **calcium nitrate** is a convenient reagent, as it may be used in all cases. It may be easily made by adding excess of pure chalk, or white marble, little by little, to some dilute hydrogen nitrate in a beaker. The solution should be boiled to drive off all the carbon dioxide, and filtered.

(*a*) *The precipitate dissolves.*

The substance is a PHOSPHATE (SILICATE or CARBONATE[1]). To make sure of the presence of a phosphate, add a few drops of the original solution to a little solution of ammonium molybdate, and warm the mixture. If a yellow precipitate is formed, a PHOSPHATE is present.

(*β*) *The precipitate remains undissolved.*

The salt is an OXALATE or FLUORIDE.

58. Pour some of the original solution upon a little **manganese dioxide** in a test-tube, and add a few drops of **strong hydrogen sulphate.** If *carbon dioxide is given off with effervescence* (p. 155, Expt. 4), an OXALATE is present.

If *no result is obtained*, test for a FLUORIDE, as directed in p. 169.

2. Examination for the Radicles included in Group II.

CYANIDE, CHLORIDE, BROMIDE, IODIDE, SULPHIDE, HYPOSULPHITE, HYPOPHOSPHITE, (ACETATE)[2].

59. **Silver nitrate test.** Add to a portion of the original solution (which, if alkaline, must be neutralised by addition of hydrogen nitrate), several drops of solution of **silver nitrate.**

(A) *No precipitate is formed.*

No radicle belonging to Group II is present[3]. Pass on to **60.**

(B) *A white or yellowish white precipitate is formed, which, after being washed, dissolves readily in ammonia.*

The salt is a CHLORIDE, CYANIDE, BROMIDE, or ACETATE.

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[1] These will have been already detected (**14, 4**).

[2] Silver acetate is not quite insoluble in water, and therefore addition of silver nitrate causes a precipitate in solutions of acetates only if they are concentrated.

[3] Mercury cyanide, however, is not precipitated by silver nitrate. The presence of cyanogen will have been shown by the preliminary examination.

A CYANIDE will, if present, have been already detected (**13**). As a confirmatory test, the reaction with **iron protosulphate** (Expt. 2, p. 157) may be tried with a fresh portion of the original solution.

[A FERROCYANIDE will also evolve cyanogen when heated in a tube, but it may be distinguished from a cyanide by giving a *deep blue precipitate* when the acidified solution is tested with a drop of **iron perchloride.**]

If there is *a tinge of yellow in the colour of the precipitate*, a BROMIDE is probably present. The reaction with **carbon disulphide** and solution of **chlorine** (p. 162) will afford decisive proof.

If *the precipitate is crystalline*, and if the original salt blackened when heated in a glass tube (**9**), an ACETATE should be tested for, as directed in p. 153, Expt. 3.

If none of the above radicles are present, the salt is a CHLORIDE. If so, chlorine gas will be evolved when a little of the original solution is heated with **manganese dioxide** and **strong hydrogen sulphate** (p. 158, Expt. 3).

(**C**) *A light yellow precipitate is formed.*

The salt is an IODIDE.

The reaction with **potassium nitrite and starch** (p. 168) will afford conclusive evidence.

(**D**) *A precipitate is formed which is at the first moment white, but rapidly turns yellow, brown, and finally black.*

The salt is a HYPOSULPHITE. The precipitation of sulphur and evolution of sulphur dioxide, on addition of an acid to the neutral solution, will have been already noticed[1].

(**E**) *A black precipitate is produced at once.*

The salt is a SULPHIDE.

The results of the preliminary examination (**3** β) will serve to confirm this.

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[1] Hypophosphites give a white precipitate of silver hypophosphite, which rapidly darkens, owing to its reduction to metallic silver. The evolution of spontaneously inflammable hydrogen phosphide, when the original salt is heated in a tube (p. 178, Expt. 1), will be a conclusive proof of the presence of a hypophosphite.

3. Examination for the Radicles included in Group III.

NITRATE, CHLORATE, ACETATE.

60. Acidify a portion of the original solution with **hydrogen sulphate** (if lead, barium, or strontium is present, a precipitate will, of course, be formed, which must be filtered off), add one drop of dilute solution of **indigo sulphate**, and heat the liquid.

(**A**) *The blue colour of the indigo disappears.*

The salt is a NITRATE or a CHLORATE [1].

61. Place a portion of the solid salt, or of the residue obtained on evaporation of the solution (**22**) in a test-tube, and add a little **strong hydrogen sulphate.**

(*a*) *The substance turns yellow, and a yellow gas is given off, possessing the odour of a chlorine oxide.*

The substance is a CHLORATE.

The reaction given at p. 161, Expt. 7 *c*, will afford confirmatory evidence.

(*β*) *The substance dissolves in the acid without turning yellow, or evolving gas.*

It is a NITRATE.

To confirm this, allow the solution to become quite cold, then pour down the side of the tube a little solution of **iron protosulphate.** If a dark brown ring is formed (either immediately or in a minute or two) at the junction of the fluids, a NITRATE is present (p. 147, Expt. 7).

(**B**) *The blue colour of the indigo remains unchanged.*

The salt is an ACETATE.

If so, the original substance will have become charred when heated in a glass tube (**9**). The reaction with **hydrogen sulphate** and **alcohol** (p. 153) should be tried as a confirmatory test.

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[1] This will have been already inferred from the results obtained in **15.**

62. If no other radicle can be discovered, the substance is probably a HYDRATE. The alkaline reaction of the solution, and the resemblance of the substance to one of the hydrates which are soluble in water, viz. BARIUM, POTASSIUM, SODIUM, and AMMONIUM HYDRATES, will confirm this.

B. *The Substance is insoluble in Water, but dissolves in Hydrogen Chloride or Aqua Regia; or, if it is in solution, the solution has a strongly acid reaction (24).*

63. If the substance is a metal, which may be inferred with certainty from its possessing the known properties, e.g. lustre, malleability, fusibility, &c. of the metallic radicle which has been already detected, no other radicle need be examined for[1].

64. Refer to the Table of Solubilities, Appendix C, to ascertain what radicles form salts insoluble in water but soluble in acids with the metal which has been found. These radicles are all which, as a rule, need be regarded as possibly present; but as there are many different degrees of insolubility, it will be safest to consider the Table as indicating what radicles ought at all events to be examined for, without absolutely excluding others.

65. If the preliminary examination has shown that *an organic acid is present*, pass on to **67**.

If *an organic acid is not present*, test separate portions of the acid solution,

(A) With solution of **barium chloride** (**54**) for a SULPHATE.

(B) With solution of **ammonium molybdate** (**57** *a*) for a PHOSPHATE.

(C) With **turmeric-paper** (**56** *β*) for a BORATE.

(D) With a drop of **chlorine** water and a little **carbon disulphide** (pp. 162, 168) for a BROMIDE and IODIDE.

---

[1] Some few minerals, such as galena, pyrites, graphite, possess metallic lustre, but not the other characteristics of metals.

(E) By evaporation to dryness and treatment of the residue with **hydrogen chloride** (27) for a SILICATE.

66. Test separate portions of the solid substance (or of the residue obtained by evaporation),

(A) With solution of **iron sulphate** (61) for a NITRATE.

(B) With **manganese dioxide** and **hydrogen sulphate** (58) for an OXALATE.

The same mixture will, on the application of heat, give off chlorine, if a CHLORIDE is present.

(C) With strong **hydrogen sulphate**, in a platinum vessel (p. 169), for a FLUORIDE.

(D) For a CYANIDE, in the following way:—

Take two watch-glasses of equal size; place in one a little of the original substance, and add six or eight drops of slightly diluted **hydrogen sulphate**; place in the other watch-glass a single drop of **ammonium sulphide**, and invert the glass quickly over the other one, so that the drop of ammonium sulphide may be retained in the centre of the concavity. Place the two watch-glasses, with their edges in contact, on a sand-bath, and apply a gentle heat. The cyanide will be decomposed, and the hydrogen cyanide evolved will be absorbed by the solution of ammonium sulphide with formation of ammonium sulphocyanide (p. 157). After the lapse of two or three minutes, take off the uppermost watch-glass, and evaporate the drop of liquid carefully to dryness; pour a drop or two of dilute **hydrogen chloride** on the residue, and add one drop, or less, of solution of **iron perchloride.** If *a deep blood-red solution is formed,* a CYANIDE is present.

67. If *an organic salt is present,* boil a portion of the substance with solution of **sodium carbonate**, filter it from any insoluble residue, and, when the solution is nearly cool, add one or two drops of solution of silver nitrate (which will probably cause a white precipitate), and warm the liquid gently.

(A) *The mixture turns black.*

The substance is a TARTRATE. The peculiar odour emitted during its carbonisation in a tube (11) will confirm this.

(**B**) *There is no reduction of the silver salt.*

The substance is an ACETATE. The reaction with hydrogen sulphate and alcohol (p. 153) will serve as a confirmatory test.

68.
Separation of the metal.

If the above tests have given no satisfactory results, it will be advisable to separate the metal and to obtain the other radicle in combination with sodium or ammonium.

The means to be employed for effecting this will depend upon the metal present.

(**A**) The metallic radicle belongs to Group I, II, or III.

Boil some of the substance with 5 or 6 c.c. of solution of **ammonium sulphide** for a few minutes: allow any residue to subside, decant the clear solution into a porcelain dish, and evaporate it to dryness. Warm the residue with some water, filter the solution, which (unless the original substance was an ARSENIC, ANTIMONY, or TIN salt) will contain only the non-metallic radicle associated with ammonium, and examine it as directed in Sect. IV **A**.

If ARSENIC, ANTIMONY, or TIN have been found to be present the solution will contain ammonium sulpharsenate, sulphantimonate, or sulphostannate. In this case, add to the solution a slight excess of dilute **hydrogen nitrate**, which will throw down the metal as sulphide, filter the solution, and examine it as directed in Sect. IV **A**, remembering that a NITRATE is now present.

(**B**) The metallic radicle belongs to Group IV or V.

Boil some of the substance with solution of **sodium carbonate** for a few minutes, filter the solution, and test it as directed in Sect. IV **A**, decomposing the excess of sodium carbonate with dilute hydrogen chloride or nitrate, as required.

SECTION V.

ANALYSIS OF A SINGLE SALT WHICH IS INSOLUBLE IN WATER AND ACIDS.

69. It will be seen, by a reference to the Table of Solubilities (Appendix C), that the salt must be one of a very limited number of substances. The following list includes all the insoluble salts which are commonly met with.

CARBON, SULPHUR, SILVER CHLORIDE, SILVER BROMIDE, SILVER IODIDE, LEAD SULPHATE, TIN DIOXIDE (tin-stone, putty powder), ANTIMONY PENTOXIDE, IRON AND CHROMIUM OXIDE (chrome iron-stone), CHROMIUM PEROXIDE[1], BARIUM SULPHATE, STRONTIUM SULPHATE, CALCIUM FLUORIDE, SILICON DIOXIDE, and several SILICATES.

The preliminary examination will here be especially useful, and will have led to the detection of the metallic radicle, at all events, in most of the above substances.

Refer to the results obtained by heating the substance in a tube (**9**).

(**A**) *The substance is dark-coloured, and infusible.*

It may be CARBON, CHROME IRON-STONE, or one of certain SILICATES.

70. Heat it strongly on a piece of platinum foil, with free access of air, directing the blowpipe-flame on the under surface of the foil.

(*a*) *It glows and burns away more or less quickly, leaving a slight residue of white ash.*

---

[1] Iron peroxide (rouge) and aluminium peroxide, after ignition, dissolve only with great difficulty in acids.

It is CARBON. Confirm this by heating a little potassium nitrate in a tube until it begins to decompose, and dropping into it a small fragment of the substance. If it is carbon, it will deflagrate strongly, with formation of a carbonate, which latter may be recognised in the usual way (p. 152)[1].

(β) *It is unaltered in appearance.*

71. Make a bead of **microcosmic salt**, introduce into it a very small quantity of the finely powdered substance, and heat it in the blowpipe-flame.

If it *dissolves entirely, and colours the bead deep green in both oxidising and reducing flames*, it is CHROMIUM SESQUIOXIDE, or CHROME IRON-STONE. It must be decomposed by fusion with **sodium carbonate** (**74**).

If *a residue remains floating undissolved in the melted bead*, the substance is a SILICATE and must be decomposed by fusion with **sodium carbonate** (**74**).

(B) *The substance is yellow, and volatilised entirely in the tube, condensing in brown drops.*

It is SULPHUR. If so, it will deflagrate when dropped into melted **nitre** (**70**), and the fused mass will contain a SULPHATE, which should be tested for with barium chloride[2].

(C) *The substance is white or light yellow, and fused below a red heat.*

It is SILVER CHLORIDE, BROMIDE, or IODIDE.

72. Fuse a portion of the substance with about three times its weight of **sodium carbonate** (or of a mixture of sodium and potassium carbonates) in a porcelain crucible, or on a piece of a broken porcelain dish.

Boil the fused mass with several successive portions of water, until everything that is soluble is dissolved; then examine the solution, filtered, if necessary, for a CHLORIDE, BROMIDE, and IODIDE (**59**). The residue will consist of SILVER, and should be dissolved in rather dilute **hydrogen nitrate**, and the solution tested for silver (**32**).

---

[1] Graphite, however, is not acted on by melted nitre.

[2] It must be borne in mind that nitre often contains a trace of sulphate.

(D) *The substance is white, or yellowish white, and did not fuse when heated in the tube, but turned yellowish brown, becoming light again on cooling.*

It is TIN DIOXIDE, or ANTIMONY PENTOXIDE.

The reduction on charcoal (17) will enable you to distinguish between these two substances, and as a further confirmation, the globule of reduced metal may be dissolved in strong **hydrogen chloride** with addition of a drop of **hydrogen nitrate**, and the solution tested for tin and antimony (34).

(E) *The substance is white, and remained unaltered on being heated strongly in the tube.*

(*a*) *No evidence of the presence of a silicate has been obtained in* 71.

The substance may be LEAD SULPHATE[1], BARIUM SULPHATE, STRONTIUM SULPHATE, or CALCIUM FLUORIDE (possibly also ALUMINIUM AND SODIUM FLUORIDE, cryolite). The two last substances fuse, however, at a strong red heat.

73. Boil a portion of the substance with some strong solution of **sodium carbonate** for two or three minutes[2]; filter the solution and test it (after decomposing any excess of sodium carbonate by adding a few drops of **hydrogen chloride** and heating) for a SULPHATE (54). Wash the residue (which may consist of LEAD OXIDE, BARIUM CARBONATE, or STRONTIUM CARBONATE), transfer it to a tube, dissolve it in dilute **hydrogen nitrate**, and test the solution for LEAD (34), BARIUM, and STRONTIUM (48).

(*β*) *A silicate has been found to be present.*

---

[1] If so, when a small portion is moistened with a drop of solution of ammonium sulphide, it will turn black.

[2] While this is going on, a fluoride may be tested for by heating a little of the substance (in a platinum vessel) with strong hydrogen sulphate, and observing whether vapours are evolved which etch glass (p. 169). If a fluoride is found to be present, continue heating the substance with hydrogen sulphate for five or six minutes, then allow the mass to cool, add a little water, mixing it thoroughly with the substance, and boil the mixture in a test-tube. Test the filtered solution for CALCIUM (48) and ALUMINIUM (43).

74. **Decomposition of insoluble silicates.** In this case, and also if CHROMIUM has been detected (**71**), the substance must be decomposed by fusion with sodium carbonate, in the following way:—

Reduce some of the substance to an impalpable powder (using an agate mortar, if possible), and mix it with about four times its weight of **sodium carbonate** (or of a mixture of sodium and potassium carbonates, which fuses at a much lower temperature than sodium carbonate alone). Place the mixture in a platinum capsule or on a piece of platinum foil turned up at the edges, and heat it strongly over the gas blowpipe, adding a small quantity of **potassium nitrate**, until all action is over and the mass is in a state of calm fusion. After allowing it to cool, detach it from the capsule, and heat it in a porcelain dish with some dilute **hydrogen chloride**, evaporating it, finally, to complete dryness, in order to separate the silica, if present. Warm the residue with a little strong **hydrogen chloride**, then add some water, and, after filtering the solution, examine it, as directed in Sect. III, for a metallic radicle[1].

The residue on the filter, if any, will consist of SILICA, or possibly of some of the original substance which has escaped decomposition. It should be examined in a bead of microcosmic salt to see if it really consists of silica.

---

[1] If CHROMIUM is detected, IRON should also be examined for.

Silicates are so often complex in their structure, consisting of silicon associated with several metallic radicles, that it is always safest to examine the solution by a method which is applicable to mixtures of salts.

SECTION VI.

EXAMPLE OF THE ANALYSIS OF A SINGLE SALT.

(The salt taken was manganese chloride.)

The single salt for examination was a light pink amorphous substance, which soon became moist when exposed to the air, and was readily powdered in a mortar.

A. Preliminary Examination.

1. A portion of the substance, heated in a test-tube with some water, readily dissolved, forming a nearly colourless solution which was neutral to test-paper.

2. A little of the solid substance was heated gently in a bulb-tube. It gave off traces of water, and when heated nearly to redness it fused into a brownish liquid which solidified, on cooling, into a pink crystalline mass. No vapours were given off, and no charring of the substance was observed. Hence no organic radicle is present.

3. Since the substance was coloured, a little of it was heated in a borax bead before the blowpipe. It gave a transparent bead which was amethyst-red in the oxidising flame and colourless in the reducing flame, indicating the presence of manganese.

B. Examination of the Solution for a Metal.

1. A portion of the solution was tested with a few drops of dilute hydrogen chloride. No precipitate was formed, and therefore silver, mercury (as protosalt), and probably lead, are absent.

2. To the same portion of the solution (containing hydrogen

chloride) was added an excess of solution of hydrogen sulphide, and the mixture was warmed. No precipitate was obtained, and therefore no metal belonging to Group II is present.

3. The same portion of the solution was mixed with some solution of ammonium chloride, rendered alkaline with ammonia (which caused a precipitate), and tested with a drop of solution of ammonium sulphide. A dull pink precipitate was produced, indicating the presence of manganese. This result was sufficiently confirmed by the colour imparted to a borax bead.

The metal, therefore, is Manganese.

C. Examination for the non-metallic Radicle.

Since the substance is a manganese salt readily soluble in water, the following radicles may be present :—Nitrate, Acetate, Chloride, Iodide, Bromide, Sulphate, Chromate.

The results of the preliminary examination have shown that the salt is not a nitrate, acetate, or chromate.

1. A portion of the original solution was tested with a drop of solution of barium chloride. A slight turbidity was produced, which did not disappear when excess of hydrogen nitrate was added; but as the precipitate was quite disproportionate to the quantity of salt known to be in solution, it proved that a sulphate was present only as an impurity.

2. Another portion of the solution was tested with solution of silver nitrate. A white flocculent precipitate was formed, which was found to be insoluble in hydrogen nitrate, but readily soluble in ammonia, indicating the presence of a chloride.

As a confirmatory test, a little of the solid substance was mixed with some manganese dioxide, and warmed in a test-tube with some strong hydrogen sulphate. A gas was given off which was proved to be chlorine by its odour, its yellowish green colour, and its bleaching action on a piece of litmus-paper held in the tube.

The non-metallic radicle is, therefore, Chlorine;
and the substance is Manganese Chloride.

APPENDIX A.

SUGGESTIONS FOR THE CONSTRUCTION OF CHEMICAL APPARATUS.

The set of apparatus described at the beginning of this book is, necessarily, a rather expensive one; and although it includes nothing which would not be found in a regular laboratory, yet a student who is working by himself may not find it possible or advisable to spend so much on the materials for his experiments.

The object of the present chapter is to supply a few hints for the construction of simpler and cheaper forms of apparatus, such as may be made by any one possessed of a little mechanical skill and a few carpenter's tools. The student should never rest satisfied with clumsy makeshifts; he should endeavour to do the best he can with the materials he has at hand.

1. Pneumatic Troughs. An earthenware pan, a large biscuit tin, or even a wooden box (covered with three or four coats of good paint) will answer the purpose. The shelf may be made of a sheet of tin plate or zinc, about 14 or 15 cm. wide, and sufficiently long to reach across the trough. A portion on each side about 1 cm. broad should be bent down, as shown in Fig. 51, to give stiffness to the shelf, and a hole about 3 cm. in diameter should be cut in the centre. The size of the hole should first be traced out with a pair of compasses and the metal may then be cut with a chisel or with the blade of a strong knife struck with a hammer, the plate being laid on a piece of board. To suspend this shelf in

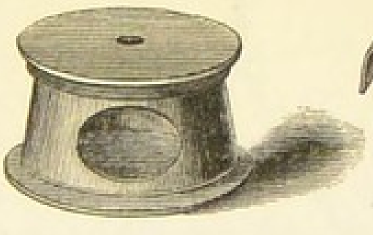

Fig. 52.

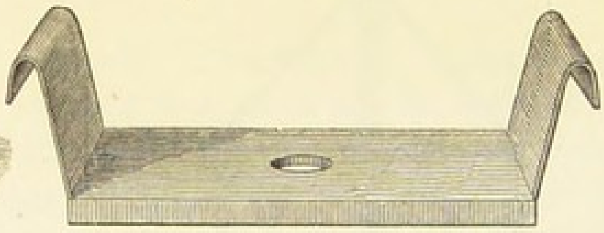

Fig. 51.

its place in the trough the ends of the strip of tin should be turned up and bent over the rim of the trough. Or, four pieces of strong brass wire may be passed through holes made at the four corners of the shelf, and bent up over the rim. Instead of tin plate the shelf may be made of wood (well painted), having one or two pieces of lead nailed on its under side to sink it, and suspended by wire hooks.

Instead of a shelf reaching across the trough, a 'bee-hive' shelf, Fig. 52, may be used. This is intended to stand on the bottom of the trough, the delivery tube being introduced into the side-opening, and the gas jar being placed over the central aperture. Such a shelf may be purchased for a shilling, or less; but it may be easily made out of a common gallipot, or flower-pot saucer. The hole in the centre may be pierced by gradually chipping away the material with the point of an old three-square file, care being taken that the blows of the hammer are light. The notch at the side may be cut out by chipping or crumbling away the earthenware with a pair of pliers. There is, of course, some chance of breaking the pot, but if it is placed on a folded cloth, and if the chipping is carefully and patiently done, the risk is slight.

Finally, an extempore shelf may be made by placing two bricks side by side in the trough, a sufficient interval being left between them to admit the delivery tube.

2, 3. Supports for Flasks, Tubes, &c. A very useful tripod-stand for supporting sand-baths, &c. may be made by taking three pieces of stout iron wire or thin rod, about 45 cm. long, bending them into this form ⊓, and binding them together by folds of thin iron wire, so as to form the stand represented in Fig. 53.

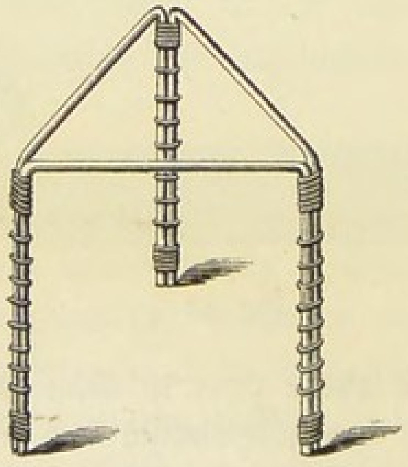

Fig. 53.

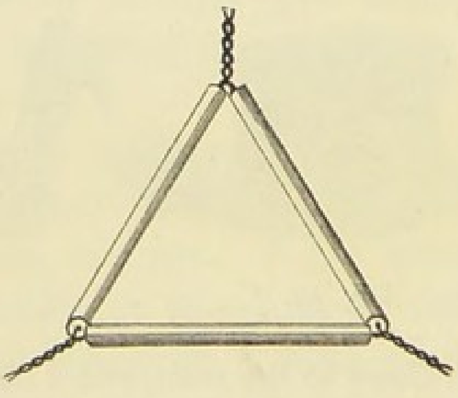

Fig. 54.

For small crucibles, a triangle support is extremely useful, Fig. 54.

It is made by taking three bits of tobacco-pipe stem about 6 cm. in length, passing through each a piece of iron wire of such a length that about 4 cm. projects at each end of the pipe stem, and then twisting together these projecting pieces of wire. It is intended to be laid on the ring of a retort-stand or on the tripod-stand above mentioned[1].

A simple form of **holder** is represented in Fig. 55, which may be constructed by the student himself. The base (12 × 24 cm.) and the upright (5 × 30 cm.) are made of deal about 2.5 cm. in thickness. The upright is mortised and glued into the base, and has a slit about 5 mm. wide cut in it to a distance of 20 cm. from the top. The arm *a* is of harder wood, such as birch or beech, and is 3 cm. square and 7 cm. long. The screw *b* is one of the common brass thumb-screws sold by ironmongers; a hole is bored in the arm *a* to receive it, and its nut is inserted in a small mortise cut in the

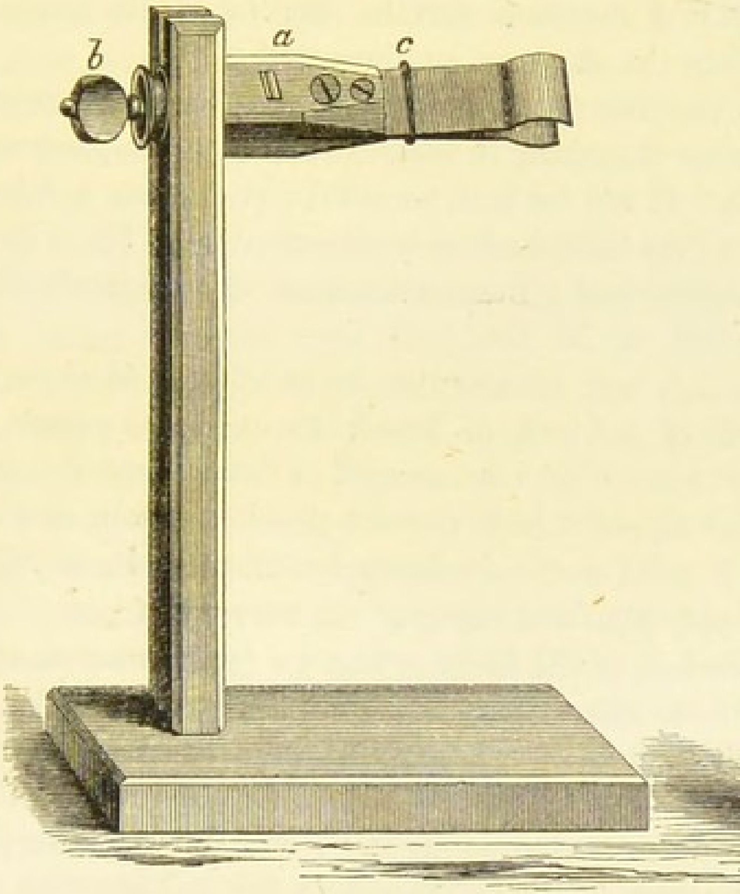

Fig. 55.

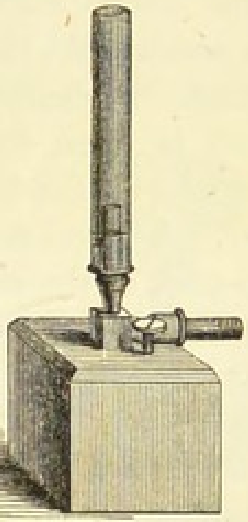

Fig. 56.

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[1] Such a triangle is also very useful for supporting a funnel, containing a filter, over a beaker. For this purpose a piece of thin board, about 6 × 12 cm., with a hole in the middle, will answer equally well.

arm about 2 cm. from the end. The screw passes through a collar (for which a small brass or iron pulley answers well) on the other side of the upright, and by tightening it the arm may be secured at any height. At the other end of the arm a saw-cut is made in which are placed two strips of stout tin or brass plate about 12 × 3 cm., firmly secured by the two screws shown in the figure. The outer ends of these strips are curved so as to grasp a tube or a retort, and their grip is tightened by slipping outwards the ring *c*. This latter is a common curtain ring 2.5 cm. in diameter, carefully flattened into the shape of an elongated ellipse.

The cost of the materials for such a stand is about eightpence or ninepence; the labour of making it would be, to many, a pleasure.

4. **Gas-burners.** It will be best to obtain at starting a fish-tail burner attached to a firm base, such as is represented in Fig. 5 (p. 4), since a blowpipe burner and a Bunsen's burner can be easily fitted to it.

A serviceable, though not ornamental, form of base is shown in Fig. 56. It consists of a block of wood, about 8 cm. square, on which is fixed, by means of two screws, a 'three-eighths angle burner' with fish-tail nipple, which may be procured from any gas-fitter. A stop-cock need not necessarily be fitted to it, since the stop-cock of the supply pipe will regulate the current of gas.

A Blowpipe Burner, of the form of Fig. 5 *a* (p. 4), may be made of a piece of glass tubing about 1 cm. in diameter and 5 cm. in length, having one extremity cut obliquely and flattened in the blowpipe-flame until it somewhat resembles Fig. 26 (p. 39) in shape. The flattening should not be carried so far as to close the opening, but a narrow slit about 1 mm. in width should be left. This tube may then be fitted over the fish-tail nipple by an india-rubber connector.

A Bunsen's Burner may be made, as shown in the figure, by placing over the fish-tail nipple a tube of glass or brass open at both ends, about 1.5 cm. in diameter and 12 or 14 cm. in length, secured in its right position by two wedges of cork or soft wood. Plenty of room should be left for air to enter at the lower end, and the tube should be raised or lowered a little, until a non-luminous flame is obtained. A tube of brass is, of course, the best, but a piece of 'composition' (lead and tin) gas tubing will answer well. Instead of leaving air-space at the bottom of the tube, it may be fitted close round the burner, and two large notches may be filed, one on each side, at such a height that when the tube is fixed in its place, the top of the fish-tail jet may be 1 mm. below the highest part of the notch.

A test-tube, the closed extremity of which has been cut off, makes a very useful burner; the only drawback being that the flame is slightly coloured by the sodium of the glass. This may be obviated by fitting over the top of the tube a short metallic ferrule, made by wrapping a strip of thin brass or tin plate, about 3 cm. broad, round a cylindrical rod a little smaller than the burner itself. The strip should be of such a length that its ends overlap each other, and the joint need not then be soldered. The elasticity of the metal will keep it in its place, when it is fitted over the end of the glass tube burner.

A still simpler form of burner may be made as follows. The gas-jet is formed by flattening one extremity of a short elbow tube, as in making the blowpipe burner, until a narrow slit remains. Over this is fitted a test-tube, as above described, by wedges of cork, and the elbow tube is passed through a notch cut in a large cork. This latter is fitted over the beak of an inverted funnel which serves as the base.

A Ring-burner, which will serve the purpose of an Argand burner, is represented in Fig. 57. It is nothing more than a flat, hollow, brass door- or cupboard-handle, about 4 cm. in diameter, round which eight or ten small nicks have been cut with a file, through which the gas issues. The hole into which the axis of the latch fitted should be enlarged with a rat-tail file and with a broach (for which latter the tapering end of a screw-driver will serve), until the cylindrical part of the fish-tail nipple will just enter it.

Fig 57.

8. A Sand-bath may be made by turning up the edges of a piece of tin plate about 16 cm. square, so as to form a shallow box 2 cm. in depth. A porcelain dish may be used as a sand-bath, but there is some risk of cracking it.

10. A Mouth Blowpipe, made of glass, is represented in Fig. 58. A good sound cork is selected, a hole is bored two-thirds

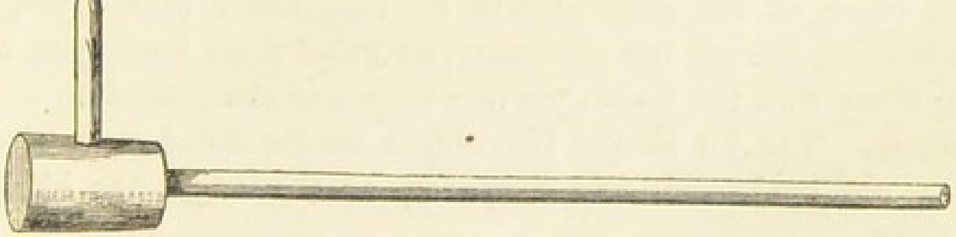

Fig. 58.

of the way through it, and another hole is then bored in the side to meet the first. Into this latter hole is fitted a short jet of hard glass tubing, and into the other hole is fitted a piece of glass tube about 20 cm. in length. A bulb may be blown on this latter tube near its lower end, to serve as a reservoir for condensing the moisture of the breath.

14. For a **Cork-borer** the tube of a steel-pen holder will answer pretty well, but it is hardly strong enough to last very long. Holes may be made in corks by means of the rat-tail file alone, the latter being worked into the cork like a gimlet. As, however, the file does not cut through, but pushes aside the particles of cork, there is some risk of splitting the cork.

18. **A Deflagrating Spoon** may be made of the bowl of a tobacco-pipe, partially filled up with plaster of paris (for the method of using this, see p. 238), in order to reduce its depth. A piece of copper wire should be twisted round the bowl, and the end bent up and passed through a large cork, or bung. Instead of a tobacco-pipe bowl, a small block of chalk or of plaster of paris, hollowed into a cup, may be used.

19. **A Deflagrating Jar**, which answers every purpose, may be made by cutting off the bottom of a large wide-mouthed glass bottle. This is best done by leading a crack round it by means of a piece of heated glass (p. 38). The crack should be begun at the edge, the red-hot glass being pressed against it for a short time, and then a drop of water applied. There is generally some little difficulty in inducing the crack to follow the heated glass evenly, owing to the irregular thickness of the glass near the bottom of the bottle, and the crack should never be left to itself for any time, lest it should extend itself in a wrong direction. A bottle may often be found which is thinnest where it should be strongest, viz. at the angle formed by the sides and the bottom. In this case the bottom may generally be detached evenly by a sharp blow of an iron rod introduced through the neck of the bottle.

The edges of the glass should be roughened with a file, and it is advisable, though not necessary, to grind them level upon a flat paving stone with the assistance of a little fine sharp sand, or emery, and water. The jar should be rubbed on the stone with circular strokes, not backwards and forwards, or the edges are likely to chip away.

20. Very useful **Gas Jars** may also be made out of bottles; the necks being cut off in the manner above described. For most

purposes, however, tall wide-mouthed bottles will answer without alteration. The small bottles, about 14 × 5 cm., in which quinine is sold, are particularly suitable for experiments; but gases should not be exploded in them.

24. Instead of the stoneware **Trays for Gas Jars**, common saucers may be used.

26. Florence Flasks will answer almost every purpose for which flasks are required. Their necks are rather narrow for a cork in which two tubes have to be fitted; the latter must be of small diameter, and the holes in the cork made with extra care. For preparing gases which are evolved without the necessity of applying heat, such as hydrogen and carbon dioxide, wide-mouthed bottles will answer quite as well as flasks.

36. Thistle Funnels. In place of these, ordinary funnels, attached to long pieces of glass tubing by india-rubber connectors, may be used. The connectors, however, will be slowly acted on if strong acids are brought in contact with them.

41. A simple form of **Spirit Lamp** is represented in Fig. 59. A cork is fitted to a wide-mouthed bottle, and a bit of glass tubing about 6 mm. in diameter and 8 cm. in length, passed through a hole bored in the cork, serves as the wick-holder. A small nick should be cut in the side of the cork to admit air, otherwise the spirit will not rise readily. A short wide test-tube is fitted over the cork as a cap, to prevent evaporation of the spirit when the lamp is not in use.

Fig. 59.

44. Flasks, and Retorts, the necks of which are broken, may be converted into evaporating dishes by leading a crack down from the broken part to within 5 or 6 cm. from the bottom, and then continuing it horizontally round the flask. A capsule about 5 cm. deep will be thus obtained, which will serve as well as, or even better than, a porcelain dish for evaporations, but cannot be used for ignitions. A clean iron spoon, not tinned, will often serve for the latter purpose.

48. A Drying Tube may be made out of a piece of glass tubing about 1.5 cm. in diameter and 15 or 20 cm. in length: a test-tube, if of stout glass, will answer the purpose, the closed end being cut off. The ends should be fused in the lamp, and turned outwards (see p. 39), for the sake of strength. They should be closed with corks into which short pieces of glass tube should be fitted. If a larger drying tube is required a paraffin-lamp-glass will answer well. If it is desired to use fragments of pumice soaked in hydrogen sulphate in order to dry a gas, the above form of drying tube cannot be used, as the acid which drains away would soon destroy the cork. A U-shaped tube must be obtained, or a tube such as is shown in Fig. 60 may be made without much difficulty. A test-tube of thick glass is selected, about 2 x 20 cm. A portion of the glass about 3 cm. from the closed end is moderately heated all round, and then a pointed blow-pipe-flame is directed upon one spot. The softened portion of the glass is forced inwards, by touching it with a piece of iron wire, so as to form a projection in the interior, reaching nearly to the centre. Two other similar projections are made by heating and pushing inwards other portions of the circumference, at the same distance from the closed end of the tube. Finally, the tube is annealed with especial care, the projections and the adjacent portions of the tube being heated again until the glass just begins to soften, and then cooled very slowly. A cork is now adapted to the tube, and fitted with two elbow tubes of small diameter, the one having each of its branches about 6 or 8 cm. in length, the other having one branch about 6 cm. in length, and the other branch sufficiently long to reach, when the cork is fitted into its place, nearly to the bottom of the test-tube, passing between two of the projections.

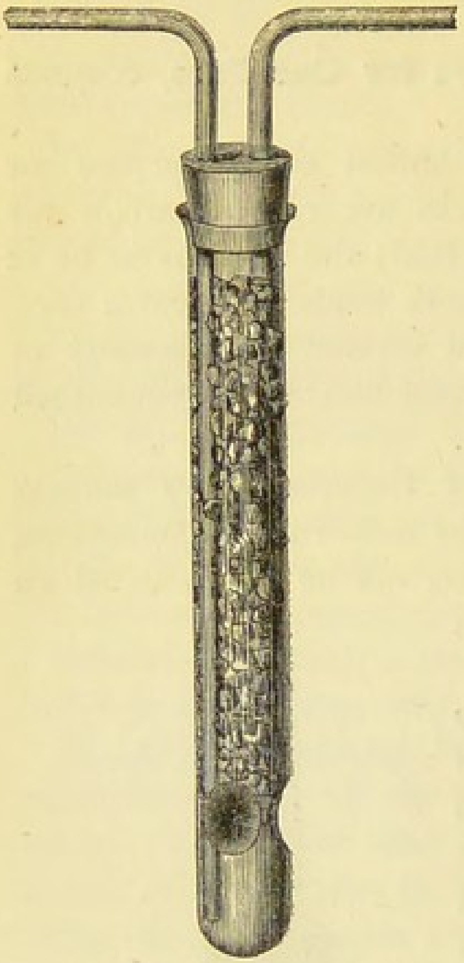
Fig. 60.

In order to fill the tube, the cork should be loosened and raised just so far as to admit of its being turned half round and then rested on the rim of the tube. There will now be sufficient room left by the side of the cork to introduce, first four or five pieces of pumice rather larger than peas, which will rest upon the ledge formed by the projections, and then a quantity of smaller bits of pumice, until the tube is nearly full. Finally, a little concentrated hydrogen sulphate should be poured in through a funnel, and the cork turned round again and gently pressed into its place. The acid will, as it descends, moisten the pumice, and any excess will collect at the bottom of the tube, and may be withdrawn by blowing through the short elbow tube, which will cause the acid to rise through the long tube.

One advantage of this form of drying tube is, that the pumice can be easily drenched with fresh acid, and the excess of acid withdrawn, without removing the pumice from the tube. Moreover, any number of these tubes can be arranged in an ordinary test-tube-stand, if required, or clustered together and held by india-rubber bands. A short wide-mouthed bottle forms a good support for a single tube, the latter being passed through a large hole in the cork.

69. Gas Blowpipes. The form of blowpipe represented in Fig. 14, p. 14, cannot be procured for much less than £1. A slightly different form, in which the feeder consists of a small pair of bellows, may be obtained at a rather lower price. A very cheap and simple gas blowpipe is represented in Fig. 61.

The air reservoir is a bladder, round which a large and strong india-rubber ring is placed to give it elasticity[1]. The air is supplied at intervals from the lungs through the tube *a*, at the other end of which is a valve to prevent the return of the air. This valve is represented in full size at *b*, and is thus constructed. A cork about 1.5 cm. in diameter is selected and a hole bored through it, into which the tube *a* is fitted so that its extremity may be just level with the surface of the cork. Across the end of the cork is laid a strip of oiled silk (oilskin) about 1 cm. or rather less in breadth, and its ends are turned down on each side of the cork and secured by an india-rubber ring, or folds of thread. The mouth of the bladder is closed by a cork about 2 cm. in diameter through which

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[1] A bladder, when allowed to dry, loses most of its pliability. To keep it moist and fit for use, it should be rubbed over with some glycerine, diluted with an equal bulk of water, a little of the liquid being also poured into the interior.

Another method of preserving a bladder is to allow it to dry in a distended state, and then to rub over it a rag dipped in sweet oil. If the bladder is now twisted and worked in all directions between the hands, it will become permanently pliable, and will not have the unpleasant smell of a moist bladder.

two holes are pierced: into one the tube *a* is fitted; into the other a short tube *c*, bent to an obtuse angle and terminating in a jet. Over this jet is fitted by a cork a common three-eighths **T**-piece (which may be obtained from any gas-fitter); and to the side-branch of this latter is adapted a short piece of glass (or, better, brass) tubing, to be connected with the gas supply. The valve having been fitted to the tube *a*, the cork is placed in the neck of the bladder, and tightly secured by several folds of string; the whole may then be supported in a Bunsen's holder, and the gas turned on and lighted at the open end of the **T**-piece. On blowing into the tube *a* the bladder will become distended with air, which will be forced out through the jet by the elasticity of the india-rubber ring. A very good and fairly uniform brush-flame may be

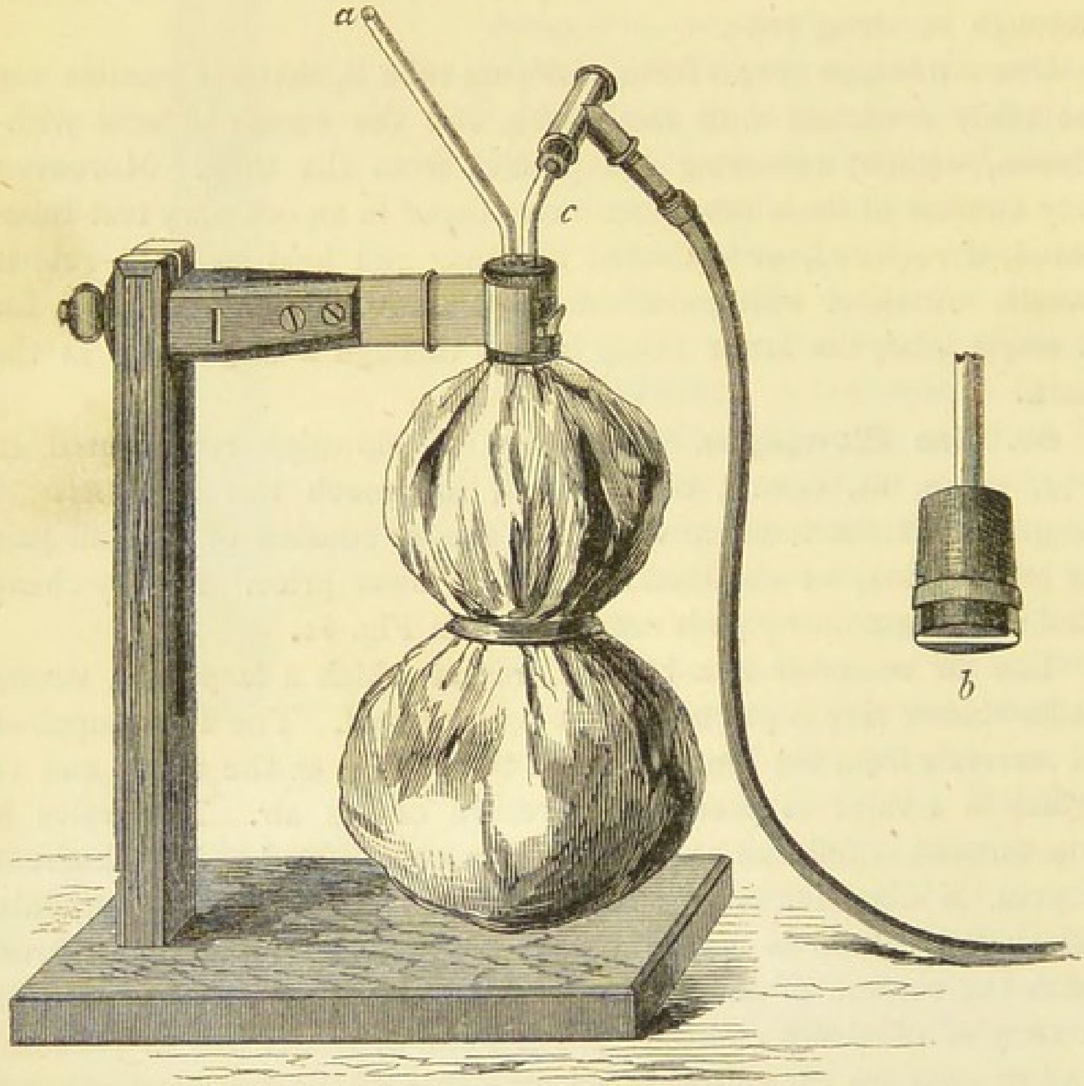

Fig. 61.

thus obtained, quite hot enough for most glass-blowing operations. Moreover, with a small air-jet, a low pressure of air, and a limited supply of gas, a well-defined pointed flame may be formed, which answers well for analytical blowpipe operations. The air-jet should not, in any case, be made larger than 1 mm. in diameter, otherwise the labour of supplying air from the lungs becomes considerable. An obvious expedient for avoiding this labour is, to connect the tube *a* by a long piece of india-rubber tubing with the nozzle of a common pair of bellows; the latter being fixed in a frame or box and placed on the floor. The upper board of the bellows may be kept raised by a spring, or by an india-rubber band, and pressed down by the foot.

The gas should not be left burning at the end of the **T**-piece longer than is required, since the latter will get very hot, and the cork may be loosened. Indeed it is decidedly better to have a brass jet screwed in; but this will add to the expense. The materials for such a blowpipe as is above described will not cost more than 1*s*. 6*d*.

71. A Retort and Receiver for distillations on a small scale are represented in Fig. 62. The retort is a very small flask, or a test-tube having its lower end expanded into a bulb, and connected by an india-rubber joint with a tube which has been bent to an acute angle and drawn out (but not very fine) just beyond the bend. The drawn-out portion passes through the cork nearly to the bottom of the receiver, which is also a test-tube. The cork should have several nicks in it, to afford escape for uncondensable vapours, and the receiver may be placed in an evaporating dish filled with cold water, or snow. Nitrogen tetroxide may be readily condensed in such an apparatus.

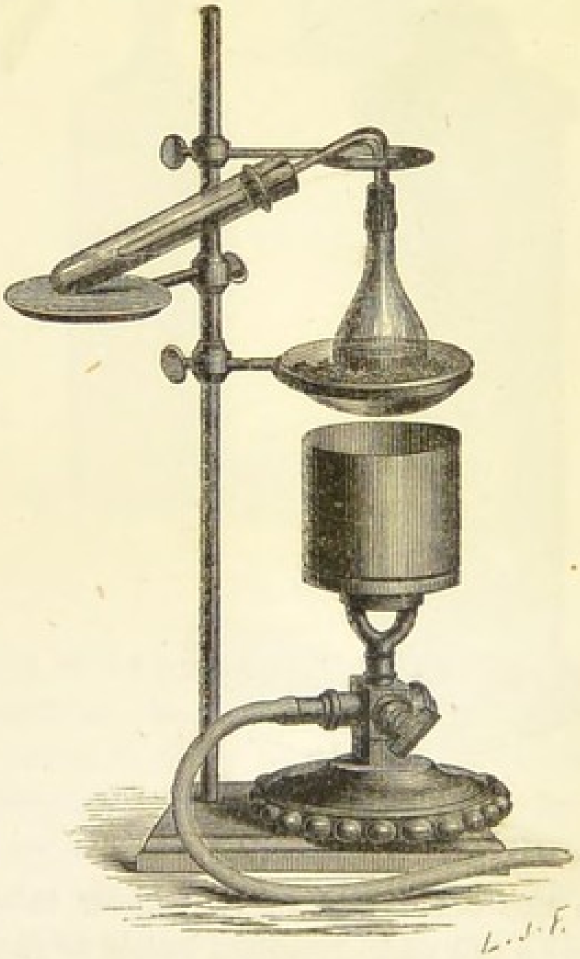

Fig. 62.

75. A Screw Pinch-cock may be made by cutting out two strips of tin plate, about 6 × 1.5 cm., turning over a narrow portion on each side, to give additional stiffness, boring a hole at each end (which may be done with a bradawl driven through by a hammer) and connecting the two strips by a couple of the small nuts and screws used for holding papers or letters together, which may be procured from a stationer's. Fig. 63 shows a pinch-cock thus made. Strips of hard wood, such as beech or box, may be used instead of the strips of tin.

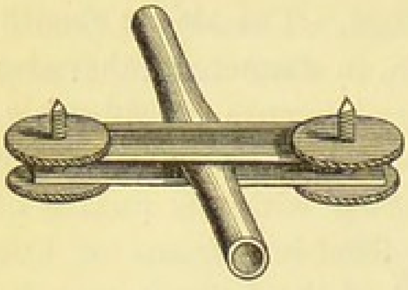

Fig. 63.

A convenient apparatus for obtaining a constant stream of a gas, such as hydrogen sulphide, which may be generated without the application of heat, is represented in Fig. 64. A paraffin-lamp-glass (one of those which bulge out near the middle) is obtained, and a cork is fitted to its lower extremity. In this cork five or six holes are bored, and before being finally fitted into its place it is thoroughly saturated with wax or paraffin, to protect it from the action of acids[1]. Some lumps of iron sulphide are placed in the glass, and a cork with a short elbow tube is fitted into the upper end of the glass, a piece of india-rubber tubing, carrying a screw pinch-cock, being adapted to the outer end of the elbow tube. The glass is now lowered into a tall wide-mouthed bottle or jar, and the latter is filled nearly

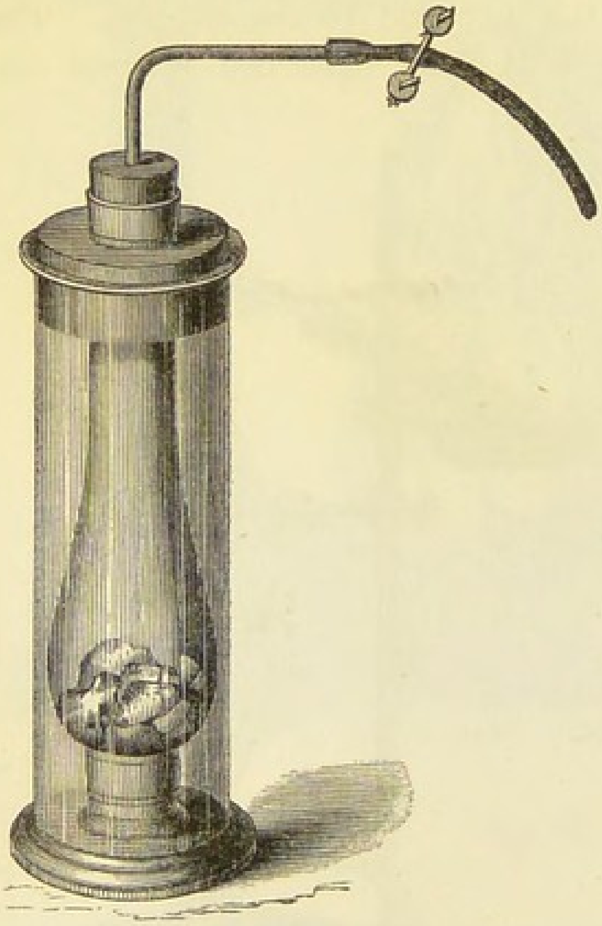

Fig. 64.

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[1] This may be done by placing it for a minute in a dish containing some paraffin or wax heated a little above its melting-point over a lamp.

to the neck with common hydrogen sulphate, previously diluted with four times its bulk of water, and allowed to cool. When the pinch-cock is opened, the acid will rise through the holes in the cork, and will act upon the iron sulphide causing an evolution of hydrogen sulphide, which may be led into a flask or tube, as required. When the pinch-cock is closed, the gas collects in the glass, and finally drives the acid back into the outer bottle; the evolution of gas ceasing when the acid is withdrawn from contact with the iron sulphide. The lamp-glass should be retained in its place in the bottle by being passed through a bung fitted into the neck of the latter, and having one or two nicks cut in it to admit air. The same form of apparatus will, of course, serve for other gases, such as hydrogen or carbon dioxide.

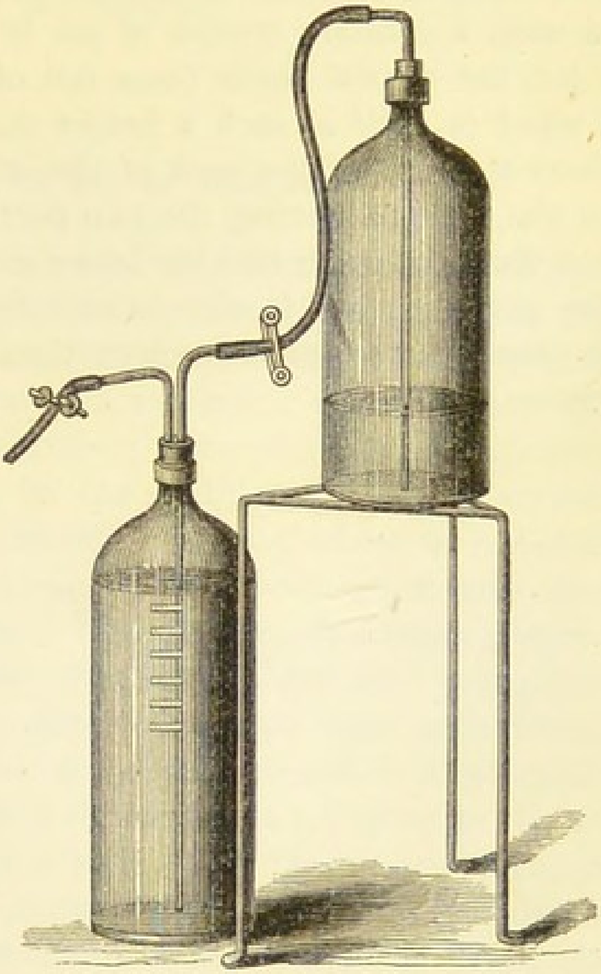

Fig. 65.

Fig. 65 represents a form of **gas-holder**, which may be easily constructed and will be found very useful. The bottles used are those which are known by the name of 'Winchester quarts,' and which may be procured from any chemist. A cork is adapted to one of the bottles and fitted with two elbow tubes, the one having both its branches about 8 or 10 cm. long, the other having one branch long enough to reach to the bottom of the bottle. An elbow tube similar to this latter is fitted to the other bottle by a cork which does not fit tightly into the neck. The two tubes last mentioned are connected by a piece of india-rubber tubing about 40 or 50 cm. in length, over which is slipped a screw pinch-cock. A piece of india-rubber tubing, also carrying a pinch-cock, is connected with the short elbow tube.

In order to use the apparatus, the first bottle is completely filled with water, the cork with the two elbow tubes being temporarily withdrawn. The short elbow tube is then connected with the delivery tube of the gas generating flask, and gas is passed into the bottle, both pinch-cocks being opened. The gas displaces the water in the bottle, which passes over into the other bottle; the latter being so placed that there may never be more than a slight difference in level between the water in the two bottles, but that the gas may always pass in under some pressure. When the first bottle is full of gas, both pinch-cocks should be closed, and the generating flask should be at once disconnected from the apparatus. If, now, a constant stream of gas is required, e. g. for burning at a jet, the second bottle (now full of water) should be placed on a stand or shelf at such a height that its bottom may be a little above the level of the neck of the other bottle. If the pinch-cock on the tube connecting the two bottles is now opened, water will flow from the upper into the lower one, and will exert a pressure on the gas contained in the latter. It will then only be necessary to open the other pinch-cock cautiously, in order to obtain a regular stream of gas.

APPENDIX B.

RECOVERY OF SILVER, GOLD, AND PLATINUM FROM RESIDUES.

1. Treatment of Silver Residues.

It has been already recommended (p. 202) that all waste solutions containing silver should be poured into a stock-bottle kept for the purpose, in order that when a sufficient quantity has accumulated the metal may be recovered from the mixture. The first step will be the conversion of any soluble silver salts which may be present into insoluble silver chloride, by the addition of a considerable excess of common hydrogen chloride. This, if added in sufficient quantity, will even throw down any silver chloride which may have dissolved in sodium hyposulphite. The mixture, after being warmed in a flask and shaken, should be filtered, and the precipitate thoroughly washed, first with dilute acid and then with water, and finally dried by placing the filter containing it in a porcelain dish on the sand-bath. When it is thoroughly dry, it must be shaken out of the filter, and mixed in a mortar with twice its weight of dried sodium carbonate and one-tenth its weight of potassium nitrate, a little borax being also added to act as a flux. While the mixture is being made, a common Cornish clay crucible, about 12 or 13 cm. in height, should be gradually heated by being placed mouth downwards on the top of an ordinary fire, if there is no wind-furnace at hand. When it is thoroughly warmed, it may be turned up and surrounded with hot coals. When it becomes red-hot, the mixture should be thrown into it, little by little, with an iron spoon, the fire being urged by a pair of bellows, so as to keep the crucible at a full red heat. The mixture will at first effervesce considerably, and should be stirred with an iron rod to prevent its frothing over, but it will finally fuse into a clear fluid, under which the silver will collect in a button. When this takes place, the crucible must be kept at a bright yellow heat for about five minutes longer, the contents being occasionally stirred to hasten the aggregation of the small particles of reduced silver disseminated through the mass. Finally, the crucible should be lifted from the fire by a pair of tongs, and the fluid mass poured out on a piece of slate or iron plate. When cold the silver may be separated from the flux by breaking off the latter with a hammer, and boiling the ingot with water to separate the last traces of slag.

It is a good plan to re-melt the ingot of silver in a small clean crucible, sprinkling a little borax over it. When thoroughly melted the metal should be poured out as before on a piece of slate, and an approximate estimate of its purity may be formed by observing the degree of 'spirting' which takes place as the silver solidifies, owing to the escape of absorbed oxygen. Unless the metal be very pure and especially free from copper and lead, this spirting will hardly take place at all, and the surface of the ingot will remain unruffled.

The lump of silver thus obtained may be either exchanged at a shop for the crystallised nitrate of silver, or it may be boiled for some time in a flask with hydrogen nitrate diluted with twice its volume of water, more of the acid being added when the action becomes slow[1]. The solution should be evaporated to complete dryness, in order to drive off the excess of acid, the residue should be redissolved in water, and the solution, after filtration, should be evaporated down and left to crystallise, the basin being covered with a glass plate or a sheet of paper to exclude dust. The crystals should be drained in a funnel and dried on filter-paper. The mother liquor, on further evaporation, will yield another crop of crystals, and the liquid drained from these may either be poured into the 'silver residues' bottle, or reserved for purposes in which absolute purity is not required.

This is decidedly the best method of reducing silver residues, since not only the chloride, but also the sulphide, iodide, &c., are reduced by fusion with sodium carbonate. If, however, the chloride only has to be dealt with, it may be reduced by hydrogen in the manner described, p. 201; the only disadvantages being that it is rather difficult to wash out the last traces of zinc salt from the spongy mass of silver, and also that organic impurities are liable to be carried down with the chloride, which form nitro-compounds when the metal is dissolved in hydrogen nitrate, and render the salt unfit for such delicate processes as photography.

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[1] If a perfectly pure salt is wanted, it will be necessary to precipitate the silver again as chloride from the hydrogen nitrate solution, and to reduce it with pure sodium carbonate in the manner above described.

2. Treatment of Platinum Residues.

These residues will usually contain the metal either as perchloride or as ammonium or potassium chloroplatinate. Any perchloride which may be present must first be thrown down by the addition of ammonium chloride as long as any precipitate is produced. The whole should then be filtered, and the residue on the filter dried and heated to redness in a porcelain dish or a clean clay crucible, to get rid of any organic impurities, and to decompose the chloroplatinates. The resulting porous mass, to which any waste scraps of platinum foil or wire may be added, should be thoroughly washed with warm water to remove all soluble salts, and then boiled for some time with a mixture of three parts of strong hydrogen chloride and one part of hydrogen nitrate. This will be best done in the open air, or at any rate the chlorous vapours must be led into a chimney or through a window, and not allowed to escape into the laboratory. When the mixture has boiled for half an hour, the solution should be poured off, fresh acids added, and the boiling repeated. The solutions should then be mixed, and the whole evaporated at a gentle heat nearly, but not quite, to dryness, a little hydrogen chloride being added at the last, to decompose any remaining hydrogen nitrate. The solution of crude platinum perchloride, obtained by warming the residue with water, should be filtered and mixed with a saturated solution of ammonium chloride, which should be added until no further precipitate is thrown down. This precipitate, of ammonium chloroplatinate, should then be collected on a filter, and carefully washed with water to which about half its volume of common methylated spirit should be added, in order to prevent the loss of any considerable quantity of the salt, since ammonium chloroplatinate is not wholly insoluble in water. Lastly, it should be dried on the filter, and strongly heated in a porcelain dish, until the whole of the salt is decomposed (p. 198). The spongy platinum thus obtained should be dissolved in aqua regia, and evaporated down, at first over the lamp to a small bulk and then on a water-bath[1] to complete dryness. The highly deliquescent residue should be transferred quickly to a well-stoppered bottle, or, better, dissolved at once in water for use.

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[1] In default of a regular water-bath for the above evaporations, the porcelain dish may be placed on the mouth of a beaker or saucepan rather smaller in diameter; the latter being previously half filled with water, which is kept gently boiling on a sand-bath.

A solution of convenient strength is made by weighing the spongy platinum before dissolving it in the acids, multiplying the weight in grammes by 25, and making up the solution to this number of cubic centimetres. This will give a solution 50 c.c. of which contains 3·39 grms. of platinum perchloride ($PtCl_4$ in 50 c.c.).

3. Treatment of Gold Residues.

These may be worked up in the same way as platinum residues, except that the solution in aqua regia must, after evaporation, be mixed with excess of a strong solution of hydrogen oxalate, and boiled for some time. The gold will thus be precipitated in the metallic state, and the yellow spongy mass obtained will readily dissolve in aqua regia. The solution should be carefully evaporated on the water-bath, and the residue dissolved in water. For each gramme of gold taken, 50 c.c. of solution should be made. The liquid will then contain 3.03 grms. of gold perchloride in 100 c.c. ($AuCl_3$ in 100 c.c.).

TABLE OF THE SOLUBILITY OF SALTS.

In this Table the name of a metallic radicle is placed at the top of each vertical row; the name of a non-metallic radicle is placed at the beginning of each horizontal row.

The letter placed at the junction of a vertical and horizontal row indicates the solubility of the (normal) compound of the two radicles.

W	signifies that the salt is	soluble in **water**.
(W)	" "	slightly soluble in **water** (1 in 200–1000 parts).
A	" "	soluble in **hydrogen chloride, hydrogen nitrate,** or **aqua regia.**
(A)	" "	difficultly soluble in **hydrogen chloride, hydrogen nitrate,** or **aqua regia.**
I	" "	insoluble in **water, hydrogen chloride, hydrogen nitrate,** and **aqua regia.**

	ARSENIC.	ANTIMONY.	TIN (protosalt).	TIN (persalt).	PLATINUM (persalt).	GOLD (persalt).	SILVER.	MERCURY (protosalt).	MERCURY (persalt).	LEAD.	BISMUTH.	COPPER.	CADMIUM.	IRON (persalt).	ALUMINIUM.	CHROMIUM.	COBALT.	NICKEL.	MANGANESE.	ZINC.	BARIUM.	STRONTIUM.	CALCIUM.	MAGNESIUM.	POTASSIUM.	SODIUM.	AMMONIUM.	HYDROGEN.
OXIDE	(W)	A I	A	A I	A	A	A	A	A	A	A	A	A	(A)	(A)	A I	A	A	A	A	(W)	(W)	(W)	A	W	W	W	W
NITRATE . . .	..	..	..	W	W	W	W	W*	W*	W*	W*	W	W	W	W	W	W*	W*	W	W	W	W	W	W	W	W	W	W
CARBONATE . .	..	..	A	A	A	..	A	A	A	A	A	A	A	A	..	A	A	A	A	A	A	A	A	A	W	W	W	W
ACETATE . . .	..	..	W	W	..	..	(W)	(W)	W	W	W	W*	W	W*	W	W	W	W	W	W	W	W	W	W	W	W	W	W
TARTRATE . .	W	A	(W)	W	..	..	A	A	A	A	(W)	A	(W)	W	W	W	W	A	(W)	(W)	A	(W)	(W)	W	(W)	W	W	W
OXALATE . . .	..	A	A	W	W	..	A	A	A	A	A	A	A	A	A	W	A	A	A	A	A	A	A	A	W	W	W	W
CYANIDE . . .	..	A	..	..	(A)	W	(A)	W	W	A	A	A	(W)	W	..	A	A	A	A	A	W	W	W	W	W	W	W	W
CHLORIDE . . .	W	W*	W	W	W	W	I	A	W	(W)	W*	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W
BROMIDE . . .	W	W	W	W	W	W	I	A	(W)	(W)	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W
IODIDE	(W)	A	(W)	(W)	A	A	I	A	(A)	(W)	A	..	W	W	..	W	W	W	W	W	W	W	W	W	W	W	W	W
FLUORIDE . . .	W	W	W	(W)	W	W	W	A	W	A	W	(W)	(W)	W	A I	W	(W)	(W)	A	(W)	A	A	A I	A	W	W	W	W
SULPHIDE . . .	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	W	W	W	W	W	W	W	W
SULPHITE . . .	..	A	(W)	W	W	..	A	A	A	A	A	W	(W)	(W)	A	W	A	A	A	(W)	A	A	W	W	W	W	W	W
HYPOSULPHITE .	..	..	W	..	..	..	A	A	A	A	..	W	W	W	..	..	W	W	W	W	(W)	W	W	W	W	W	W	W
SULPHATE . .	..	W	W*	W	W	W	(W)	W	W*	A I	W	W	W	W*	W	W	W	W	W	W	I	I	W	W	W	W	W	W
HYPOPHOSPHITE	..	..	..	..	..	..	W	..	..	(W)	..	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W	W
PHOSPHATE . .	W	W	A	A	..	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	A	W	W	W	W
BORATE . . .	..	..	(W)	..	..	..	(W)	..	(W)	(W)	(W)	(W)	(W)	(W)	(W)	(W)	(W)	W	(W)	(W)	(W)	(W)	(W)	(W)	W	W	W	W
SILICATE . . .	..	..	..	..	..	..	..	..	..	..	..	A	..	A	A I	..	..	..	A	A	A	A	A I	A I	W	W	..	(W)I
ARSENITE . . .	..	W	A	A	..	..	A	A	A	A	..	A	..	A	..	..	A	A	A	..	(W)	(W)	A	..	A	W	W	W
CHROMATE . .	..	A	A	A	A	..	A	A	A	A	A	(W)	A	A	..	A	(W)	W	W	W	A	(W)	(W)	W	W	W	W	W

* Basic salt, A.

Metals whose oxides or hydrates are soluble in solution of **potassium hydrate:**
ARSENIC, ANTIMONY, TIN, GOLD, LEAD, ALUMINIUM, CHROMIUM, ZINC.

Metals whose oxides or hydrates are soluble in solution of **ammonia or ammonium salts:**
ARSENIC, SILVER, COPPER, CADMIUM, COBALT, NICKEL, MANGANESE, ZINC, MAGNESIUM.

APPENDIX C.

TABLE OF THE SOLUBILITY OF SALTS.

This Table is intended to indicate the solubility in water and acids of the single salts formed by the union of the different radicles treated of in the foregoing pages, and has been compiled mainly upon the authority of Störer's 'Dictionary of Solubilities.'

The degrees of solubility are, however, so numerous that it is difficult or impossible to draw a sharp line between a soluble and an insoluble substance. The statements in the Table must therefore be only regarded as approximations; as relatively rather than absolutely true.

It is hardly necessary to remark that in cases where a substance is said to dissolve in an acid, the phenomenon really consists in the formation, by the action of the acid, of a salt soluble in water.

APPENDIX D.

TABLES OF WEIGHTS AND MEASURES.

The system of weights and measures used in this book is that which is known as the metric system.

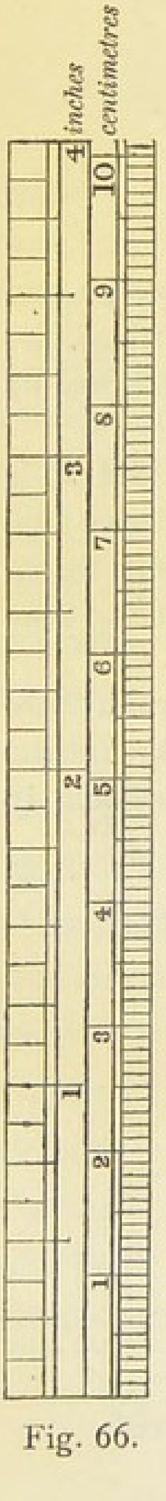

Fig. 66.

The unit of the system is the **metre**, the length of which is $\frac{1}{40,000000}$th part of the earth's circumference, as determined in 1796 by Delambre and others.

The greater and smaller measures of length are all derived from the metre by multiplying it or dividing it by 10 or a multiple of 10, and their names are formed by adding to the word 'metre' a prefix derived, in the case of the multiples, from a Greek numeral, in the case of the subdivisions, from a Latin numeral.

The following Table shows the metric measures of length, and their value in English measure.

Kilometre	=	1000 metres	=	39370.8 inches	=	0.6214 of a mile.
Hectometre	=	100 ,,	=	3937·08 ,,	=	109.36 yards.
Decametre	=	10 ,,	=	393·71 ,,	=	10.93 ,,
Metre	=	1 ,,	=	39·37 ,,	=	1.09 ,,
Decimetre	=	$\frac{1}{10}$,,	=	3·94 ,,		
Centimetre	=	$\frac{1}{100}$,,	=	0.39 ,,		
Millimetre	=	$\frac{1}{1000}$,,	=	0.039 ,,		

It may be convenient to remember that a decimetre is very nearly four inches, and Fig. 66 shows a decimetre and its subdivisions, compared with a scale of English inches.

The unit of volume is a cube, each of the sides of which measures one decimetre. It is called a **litre**, and its multiples and subdivisions are, like those of the metre, based on a decimal system. The subdivisions are, however, usually expressed in cubic centimetres (c.c.).

Kilolitre, *or* stere		= 1000 litres	= 220.01 gallons.
Hectolitre		= 100 ,,	= 22.00 ,,
Decalitre		= 10 ,,	= 2.20 ,,
Litre		= 1 ,,	= 1.76 pints.
Decilitre	= 100 c.c.	= $\frac{1}{10}$,,	= 3.5 fluid oz.
Centilitre	= 10 c.c.	= $\frac{1}{100}$,,	= 0.35 ,,
Millilitre (*or* cubic centimetre)	= 1 c.c.	= $\frac{1}{1000}$,,	= 0.035 ,,

The unit of weight is the weight of 1 cubic centimetre of water at the temperature of 4° Centigrade. It is called a gramme, and is, like the metre and the litre, multiplied and subdivided decimally. Thus,

Kilogramme	= 1000 grammes	= 2.205 lbs.	(avoird.)
Hectogramme	= 100 ,,	= 3.527 oz.	,,
Decagramme	= 10 ,,	= 0.35 oz.	,,
Gramme	= 1 ,,	= 15.43 grains.	
Decigrame	= $\frac{1}{10}$,,	= 1.54 ,,	
Centigramme	= $\frac{1}{100}$,,	= 0.154 ,,	
Milligramme	= $\frac{1}{1000}$,,	= 0.015 ,,	

TABLES OF ENGLISH MEASURES.

1. Length.

1 inch		=	2.54 centimetres.
12 inches	= 1 foot	=	30.48 ,,
3 feet	= 1 yard	=	91.44 ,,
5½ yards	= 1 pole	=	5.03 metres.
4 poles	= 1 chain	=	20.12 ,,
40 poles	= 1 furlong	=	201.16 ,,
8 furlongs	= 1 mile	=	1609.3 ,,

2. Volume.

1 cubic inch	=		16.38 cubic centimetres.
1728 cubic inches	= 1 cubic foot	=	28.31 litres.

---

1 drachm	= 0.216 cubic inch	=	3.55 cubic centimetres.
8 drachms	= 1 ounce	=	28.4 ,, ,,
20 ounces	= 1 pint	=	567.9 ,, ,,

2 pints = 1 quart = 1.136 litres.
4 quarts = 1 gallon = 4.543 „

---

1 gallon = 70,000 grains of distilled water at the temperature of 60°F (15.5°C).

3. Weight.

(a) *Apothecaries.*

1 grain = 0.0648 gramme.
20 grains = 1 scruple = 1.296 „
3 scruples = 1 drachm = 3.888 „
8 drachms = 1 ounce = 31.104 „
12 ounces = 1 pound = 373.248 „

(b) *Avoirdupois.*

1 grain = 0.0648 gramme.
27.34 grains = 1 drachm = 1.772 „
16 drachms = 1 ounce = 28.35 „
16 ounces = 1 pound = 453.6 „
112 pounds = 1 cwt. = 50.8 kilogrammes.

APPENDIX E.

THERMOMETRIC SCALES.

The temperatures mentioned in this book are all expressed on the Centigrade scale.

According to this scale, the space through which the column of mercury in a thermometer moves, when passing from the temperature at which water freezes to the temperature at which water boils, is divided into 100 equal parts or degrees, and the scale is extended below the freezing-point and above the boiling-point of water, in divisions of the same value.

The point at which the mercury column stands when the thermometer is immersed in melting ice is marked 0°, and the degrees below this point are distinguished by a *minus* sign, thus -1, -2, &c.

The point at which the mercury column stands when the thermometer is immersed in the steam from boiling water (the barometric column being 760 mm.) is marked 100°.

In the Fahrenheit scale, which is not yet superseded in this country, the space between the freezing-point and the boiling-point of water is divided into 180 degrees, and, moreover, the freezing-point of water is marked 32° instead of 0°, the boiling-point of water being consequently marked 212° instead of 180°.

The points to be borne in mind, then, in converting temperatures expressed on the one scale to the corresponding temperatures on the other scale, are—

1. The same space is divided on the Centigrade scale into **100** parts, on the Fahrenheit scale into **180** parts.

Consequently, 1 degree on the Centigrade scale is equal in length to ($\frac{180}{100}=$) $\frac{9}{5}$ of a degree on the Fahrenheit scale; and 1 degree on the Fahrenheit scale is equal in length to ($\frac{100}{180}=$) $\frac{5}{9}$ of a degree on the Centigrade scale[1].

---

[1] For since $100^\circ\mathrm{C} = 180^\circ\mathrm{F}$

$$100^\circ\mathrm{C} : 1^\circ\mathrm{C} :: 180^\circ\mathrm{F} : \tfrac{9}{5}{}^\circ\mathrm{F}$$

and $$180^\circ\mathrm{F} : 1^\circ\mathrm{F} :: 100^\circ\mathrm{C} : \tfrac{5}{9}{}^\circ\mathrm{C}.$$

2. The point marked 0° on the Centigrade scale is marked 32° on the Fahrenheit scale.

Consequently, in order to bring the two scales to the same starting-point, we must subtract 32 from the number of Fahrenheit degrees, before converting them into Centigrade degrees; and, in the reverse process, after converting the Centigrade degrees into Fahrenheit degrees, we must add 32.

The rules, therefore, may be thus expressed:—

1. To convert **Fahrenheit** degrees into **Centigrade** degrees,

Subtract 32 from the number of degrees, and multiply the remainder by $\frac{5}{9}$ (or $0.\dot{5}$)[1].

2. To convert **Centigrade** degrees into **Fahrenheit** degrees,

Multiply the number of degrees by $\frac{9}{5}$ (or 1.8) and add 32 to the product[2].

---

[1] $n°\text{F} = (n - 32)\frac{5}{9}°\text{C}$.

[2] $n°\text{C} = (\frac{9}{5}n + 32)°\text{F}$.

This rule may also stand as follows:—

Double the number of degrees, subtract $\frac{1}{10}$ of this product, and add 32 to the remainder.

$$\text{Thus,}\quad 25°\text{C} \times 2 = 50$$
$$50 - 5 = 45$$
$$45 + 32 = 77°\text{F}.$$

APPENDIX F.

ON CHEMICAL SYMBOLS.

The aim of these Exercises has been to present some of the facts of chemistry without entering upon questions of chemical theory. But symbols having been used to express the principal chemical changes which form the subject of the exercises, and the nature and strength of the various reagent solutions, a statement of the meaning of such symbols is subjoined, together with a Table of Atomic Weights.

In the beginning of Part II is an explanation of the term 'single substance.' A chemical change consists in the conversion of any quantity of one or more single substances into an equal quantity, by weight of one or more different single substances. The remembrance of some of the most important facts relating to these changes is facilitated by the adoption of the following hypothesis.

It may be the case that a portion, a gramme for instance, of any single substance is an aggregate of a vast but finite number of little particles, each of which has exactly the same properties as any other and as the whole mass. These little particles are, in almost all cases, themselves divisible; but when they are divided, it is not into smaller portions of the same substance, but into distinct parts which are called atoms, as being incapable of further division. These atoms unite into fresh groups which are the molecules of substances different from the original substance; and it is in this way, according to the hypothesis, that a chemical change takes place.

A molecule may consist of any number of atoms, from one up to a very large number. The molecules of the elements are supposed to consist either of single atoms or of two or more similar atoms united together. Differences in the properties of different substances may be due to the differences in the nature, or number, or arrangement, of the atoms of which their molecules consist.

The weight of a molecule is the sum of the weights of its atoms; and, since different atoms have very different weights, and different molecules consist of very different numbers of atoms, the weight of one molecule may be several hundred times as great as that of another.

Chemists have agreed to represent the various atoms by letters, that assigned to each being generally the initial letter of the name of that element whose molecule is made up of such atoms. Thus H represents the hydrogen-atom and O the oxygen-atom. But as the names of several elements have the same initial letter, the requisite variety has been obtained by taking the Latin instead of the English name, or by using two letters. Thus K represents the potassium-atom (Kalium), Co the cobalt-atom, Sb the antimony-atom (Stibium). The union of two or more atoms to form a molecule or group of atoms is represented by placing their symbols together, thus Co O represents a molecule of cobalt oxide, which consists of a cobalt-atom united to an oxygen-atom. When similar atoms are united, a numeral is affixed to the symbol of the atom to represent its repetition so many times; thus, the molecule of water is expressed by H_2O instead of H H O.

It will be plain that, according to the hypothesis stated above, the atoms are not actual portions of any known substance, except in the case of those substances whose molecules consist of single atoms, where, consequently, the atom is also a molecule. The smallest particle, or molecule, of common salt is represented by the formula Na Cl: its atoms Na and Cl can be transferred to other molecules, but cannot be obtained as distinct substances; we know them only as constituents of a number of different molecules. And in the same way the molecule of hydrogen is represented by H_2, the letter H representing, not a minute portion of hydrogen, but a constituent of hydrogen, a hydrogen-atom, a portion of matter weighing half as much as the smallest particle of hydrogen, which is transferable in chemical changes from one molecule to another.

The determination of the relative weights of different atoms depends upon (1) the accurate quantitative analysis of a number of single substances, (2) a decision as to the constitution of the molecules of these substances. For example, analysis shows that nine parts by weight of water can be decomposed into eight parts by weight of oxygen and one part by weight of hydrogen. Choosing the weight of the hydrogen-atom, as being the smallest relative weight we have to do with, as the unit of our system, if we decide that a molecule of water consists of one hydrogen-atom combined with one oxygen-atom, the weight of the oxygen-atom must be 8. If we decide that a molecule of water consists of one hydrogen-atom combined with two oxygen-atoms, we must take 4 to be the atomic weight of oxygen; or if we have reasons, as in fact we have, to regard the molecule of water as consisting of two hydrogen-atoms and one oxygen-atom, we must call the weight of the oxygen-atom 16.

The reasons which guide us in deciding what is the constitution of the molecule of a substance may be stated summarily under two heads: (1) that constitution is most probably true which affords the simplest account of the changes which the molecule is known to undergo, and which best exhibits the analogy between the substance in question and other substances which it resembles; (2) that constitution is most probably true which makes the molecular weight of the substance bear to the molecular weights of other substances the same ratio which its vapour-density bears to theirs. As this latter guide to the molecular constitution of substances is only available in the case of those substances which can be volatilized without decomposition at a moderate heat, the weights of those atoms have been most definitely fixed which enter into molecules of this kind.

It has also been observed that the relative weights of the atoms are in most cases nearly in inverse proportion to the specific heats of the elements which they form. This observation furnishes us with another criterion of the true atomic weight.

The following Table of Atomic Weights has been arrived at in the manner indicated.

TABLE OF ATOMIC WEIGHTS.

Al	represents an	Aluminium-atom	weighing	27·5
Sb	,,	Antimony-atom	,,	122
As	,,	Arsenic-atom	,,	75
Ba	,,	Barium-atom	,,	137
Bi	,,	Bismuth-atom	,,	210
B	,,	Boron-atom	,,	11
Br	,,	Bromine-atom	,,	80
Cd	,,	Cadmium-atom	,,	112
Ca	,,	Calcium-atom	,,	40
C	,,	Carbon-atom	,,	12
Cl	,,	Chlorine-atom	,,	35·5
Cr	,,	Chromium-atom	,,	52·5
Co	,,	Cobalt-atom	,,	59
Cu	,,	Copper-atom	,,	63·5
F	,,	Fluorine-atom	,,	19
Au	,,	Gold-atom	,,	196.7
H	,,	Hydrogen-atom	,,	1
I	,,	Iodine-atom	,,	127

Fe	represents an	Iron-atom	weighing	56
Pb	,,	Lead-atom	,,	207
Li	,,	Lithium-atom	,,	7
Mg	,,	Magnesium-atom	,,	24
Mn	,,	Manganese-atom	,,	55
Hg	,,	Mercury-atom	,,	200
Mo	,,	Molybdenum-atom	,,	92
Ni	,,	Nickel-atom	,,	59
N	,,	Nitrogen-atom	,,	14
O	,,	Oxygen-atom	,,	16
Pd	,,	Palladium-atom	,,	106.5
P	,,	Phosphorus-atom	,,	31
Pt	,,	Platinum-atom	,,	197.4
K	,,	Potassium-atom	,,	39
Se	,,	Selenium-atom	,,	79
Si	,,	Silicon-atom	,,	28
Ag	,,	Silver-atom	,,	108
Na	,,	Sodium-atom	,,	23
Sr	,,	Strontium-atom	,,	87.5
S	,,	Sulphur-atom	,,	32
Tl	,,	Thallium-atom	,,	204
Sn	,,	Tin-atom	,,	118
Ti	,,	Titanium-atom	,,	50
W	,,	Tungsten-atom	,,	184
U	,,	Uranium-atom	,,	120
V	,,	Vanadium-atom	,,	51.2
Zn	,,	Zinc-atom	,,	65

This Table, which includes all the atoms which are not of very rare occurrence, enables us to calculate the weight of any molecule whose formula is known to us, relatively to the weight of a hydrogen-atom which is assumed as the unit. Thus, the formula of calcium carbonate being $CaCO_3$, the weight of its molecule is $(40 + 12 + (3 \times 16) =)\ 100$; that is to say, a molecule of calcium carbonate weighs a hundred times as much as a hydrogen-atom.

A Table of Molecular Weights, which might be extended to all substances whose constitution has been determined, would have the following form.

H_2	represents a molecule, or	2	parts by weight, or	1 volume of	Hydrogen.
N_2	,,	28	,,	,,	Nitrogen.
Hg	,,	200	,,	,,	Mercury.
P_4	,,	124	,,	,,	Phosphorus.
NH_3	,,	17	,,	,,	Ammonia.
C_2H_4	,,	28	,,	,,	Ethylene.

&c. &c.

In the equations by which chemical changes are represented the sign = has not simply its algebraic sense of 'equals,' but should rather be read 'is converted into.' That the substances formed in a chemical change weigh as much as the substances that went to form them is a fact which renders the symbol appropriate, but is not all that it is understood to mean. The sign +, connecting the symbols of two molecules, denotes that these molecules disappear, or are formed, simultaneously; it may be expressed in reading by the word 'and.'

Thus the equation

$$K_2SO_4 + BaCl_2 = BaSO_4 + 2KCl$$

should be read thus, 'a molecule of potassium sulphate and a molecule of barium chloride are converted into a molecule of barium sulphate and two molecules of potassium chloride.' To obtain the relations by weight between the different substances we write down,

K_2 – 78	Ba – 137	Ba – 137	K – 39
S – 32	Cl_2 – 71	S – 32	Cl – 35·5
O_4 – 64		O_4 – 64	
K_2SO_4 – 174	$BaCl_2$ – 208	$BaSO_4$ – 233	74·5

whence we see that 174 parts by weight of potassium sulphate and 208 parts by weight of barium chloride are converted into 233 parts by weight of barium sulphate and 149 parts by weight of potassium chloride.

Since every gaseous molecule occupies the same volume, equations representing changes in which gases take part may be read off at once as expressing changes of volume. The common volume occupied by the smallest particles of the different kinds of matter in the gaseous state should be called one volume.

Thus, $2CO + O_2 = 2CO_2$ may be read 'two volumes of carbon protoxide and one volume of oxygen are converted into two volumes of carbon dioxide.' If in comparing the molecular weights and volumes of different substances we take a gramme for unit instead of the weight of an atom of hydrogen, the common volume occupied by 28 grms. of carbon oxide, 32 grms. of oxygen, 44 grms. of carbon dioxide, &c. is 22.4 litres.

A. V. H.

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