(HCl, NaCl) may also be determined by volumetric analysis with a standard solution of nitrate of silver (p. 624).

Combined chlorine is gravimetrically estimated in the form of chloride of silver, the operation being identical with that just described for silver salts (p. 657). 58.4 parts of pure, colorless, crystallized chloride of sodium (rock-salt) yield 143.1 of chloride of silver.

IODIDES.

Free iodine is estimated volumetrically by solutions of hyposulphite of sodium (*vide* p. 579).

Combined iodine is determined gravimetrically in the form of iodide of silver, the operations being conducted as with chloride of silver. Iodide of potassium may be used for an experimental determination: KI=165.1 should yield AgI=234.3. Of iodide of cadmium (*Cadmii Iodidum*, B. P., 1867) it is stated that "10 grains dissolved in water, and nitrate of silver added in excess, give a precipitate which, when washed with water and afterward with half an ounce of solution of ammonia, and dried, weighs 12.5 grains."

In presence of chlorides and bromides the iodine in iodides may be precipitated and weighed as iodide of palladium.

Moisture in iodine is estimated by loss on exposing a weighed quantity of iodine in a capsule over a dish of sulphuric acid under a small bell-jar, or by adding to a weighed sample five or six times as much mercury or twice as much zinc, and a little water, drying and weighing. The product is the amount of metal employed plus that of the *dry* iodine in the sample.

BROMIDES.

Free bromine may be estimated by shaking with excess of solution of iodide of potassium, and then determining the equivalent quantity of liberated iodine by a standard solution of hyposulphite of sodium (p. 636).

The bromine in bromides may be precipitated and weighed as bromide of silver, the manipulations being the same as those for chloride of silver: 0.2 to 0.3 of pure bromide of potassium may be used for an experimental analysis.

Ammonii Bromidum, U. S. P.: "1 gm. of the powdered and dry salt, when completely precipitated by nitrate of silver, yields, if perfectly pure, 1.917 gm. of dry bromide of silver." *Calcii Bromidum*, U. S. P.: "1 gm of the dry salt, when completely precipitated by nitrate of silver, yields, if perfectly pure, 1.878 gm. of dry bromide of silver."

CYANIDES.

The hydrogen cyanide (hydrocyanic acid) is usually estimated volumetrically (*vide* p. 624).

From all soluble cyanides cyanogen may be precipitated by nitrate of silver after acidulating with nitric acid, the cyanide of silver collected on a tared filter, dried at 100° C., and weighed.

Of the official Diluted Hydrocyanic Acid it is stated that 100 grains (or 110 minims), precipitated by solution of nitrate of silver, yield 10 grains of dry cyanide of silver.

Cyanide of Silver.

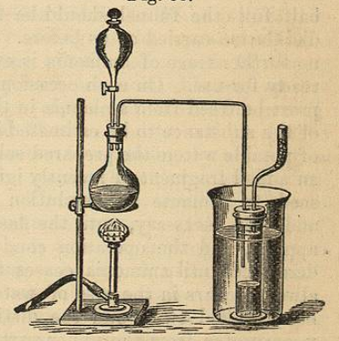
		In 1 molec. wt.	In 100 parts.
Silver . . .	Ag . .	107.7 . .	80.55
Cyanogen .	CN . .	26.00 . .	19.45
		133.7	100.00

NITRATES.

Nitrates cannot be estimated by direct gravimetric analysis, none of the basylous radicals yielding a definite nitrate insoluble in water. With some difficulty they may be determined by indirect volumetric methods.

Process.—The following (Thorpe's) method depends upon the fact (Gladstone and Tribe) that when zinc upon which copper is deposited in a spongy form is boiled with water hydrogen is evolved. Thorpe found that in a solution containing nitrates the nascent hydrogen converts the whole of the nitrogen of the nitrates into ammonia, which may be collected and estimated. (The oxygen of the nitrate is simultaneously converted into water, the nitrate-metal into hydrate, and the zinc into hydrate of zinc. The power of the copper-zinc couple is considered to depend largely on the hydrogen absorbed by the finely-divided metal.)

Fig. 83.



Estimation of Nitrates.

An apparatus such as shown in Fig. 83 should be constructed. A flask (about 100 c.c.) is fitted with an India rubber cork, perforated for a delivery-tube, which should be of strong glass tubing of about quarter-inch bore, and for a stoppered funnel, which should have about half the capacity of the flask. The whole is supported by a clamp or on wire-gauze. The outer jar shown in the figure should have a capacity of 2 or 3 litres, and the inner receiving-jar should be capable of holding 200 c.c. The latter is fitted with an India-rubber cork, perforated for the delivery-tube, and for another tube containing fragments of glass.

A few strips of clean zinc are boiled in a beaker with a 3-per cent. solution of sulphate of copper, the operation being repeated with a fresh portion of solution six consecutive times. A thick coating of finely-divided copper is deposited. The pieces of metal are well washed and introduced into the flask, which is then half filled with pure water. To avoid transference, the flask itself may be used instead of the beaker. The funnel also is filled with pure water. Into the inner receiver is put a little pure water very slightly acidulated with hydrochloric acid, and the glass fragments are also moistened with the dilute acid (to prevent possible loss of ammonia). Water is now placed around the inner receiver in the outer jar, and, the connections being sound, heat is applied with the view of freeing the apparatus itself from any trace of ammonia. When the contents of the flask are evaporated nearly to dryness, pure water is admitted from the funnel until the flask is again about half full (the funnel should be filled again at once), and the distillation carried on as before. This must be repeated until no further trace of ammonia is evolved, when the apparatus is ready for use. On each occasion that the apparatus is used it must be freed from ammonia in this way. A suitable quantity of the substance to be estimated is now introduced (in the case of potable waters the prepared solid residue from 100 c.c., with an added fragment of recently ignited lime, the size of a hemp-seed, to promote the evolution of the ammonia), and water added, if necessary, until the flask is half full. Heat is now applied, and the operation conducted in the manner already described until ammonia ceases to come over—a point which always occurs in the case of water-residues when the flask has been refilled twice and the distillate is about 100 c.c. The warm water from the upper part of the cooling-jar may be removed by a siphon or otherwise, cold water being introduced from time to time.

The ammonia being all evolved, disconnect the flask and receiver simultaneously (unless washing-bottle tubes are fitted), and treat the contents of the latter by the Nessler method, described on page 615.——Urea yields but traces of ammonia by this process, and neither the sulphates nor chlorides of the alkali-metals affect the result.——The method is only applicable to highly dilute solutions of nitrates, for with stronger solutions oxides of nitrogen are formed and escape.

Another process (Pelouze's improved by Fresenius) consists in adding the nitrate to an acid solution of a ferrous salt of known strength, and, when reaction is complete, estimating the amount of ferrous salt unattacked by volumetric solution of red chromate or of permanganate. Three molecular weights of converted ferrous salt indicate one molecular weight of nitric acid. Regeneration of nitric or nitrous acids by aërial oxidation of the nitric oxide evolved is prevented either by a current of carbonic acid gas or by using a closed flask in which is a *Bunsen valve* (*i. e.* a short attached piece of India-rubber tubing closed at the free extremity and having a sharp longitudinal slit in it a third of an inch long—a slit by which gases can escape, but cannot re-enter).

Potassii Nitras, U. S. P.: "If 1 gm. of the dried salt be moistened with 1 gm. of concentrated sulphuric acid, and the mixture be kept at a red heat until it ceases to lose weight, the residue should weigh 0.86 gm."

SULPHIDES.

Process 1.—Soluble sulphides (H_2S, NaHS, *e. g.*) may be determined volumetrically by adding to the aqueous liquid a measured excess of an alkaline solution of arsenic of known strength, neutralizing by hydrochloric acid, diluting to any given volume, filtering off the sulphide of arsenicum precipitated, taking a portion of the filtrate equal to half or a third of the original volume, and, after neutralizing by acid carbonate of sodium, estimating the residual arsenic by the standard iodine solution (*vide* p. 572). The process may be tried on a measured volume of sulphuretted hydrogen (the weight of which is easily calculated: 1 litre of hydrogen = 0.0896 gramme) absorbed by a strong solution of soda or potash.

Process 2.—Sulphur and sulphides may also be quantitatively analyzed by oxidizing to sulphuric acid and precipitating in the form of sulphate of barium. A couple of decigrammes of a pure metallic sulphide may be decomposed by careful deflagration with a mixture of chlorate of potassium and carbonate of sodium, the product dissolved in water, acidulated

with hydrochloric acid, solution of chloride of barium added, and the precipitated sulphate of barium purified and collected as described in connection with the estimation of barium (p. 645). Many sulphides may be oxidized in a flask by chlorate of potassium and hydrochloric acid, and then precipitated by chloride of barium. Experimental determinations may also be made on a weighed fragment of sulphur, about 0.1 grm., cautiously fused with a solid caustic alkali, and the product oxidized while hot by the slow addition of powdered nitrate or chlorate of potassium, or, when cold, by treatment with chlorate of potassium and hydrochloric acid, and subsequent precipitation by chloride of barium.

Note.—Fusions performed by help of a gas-lamp must be carefully conducted, for any alkali that may creep over the side of a crucible will certainly absorb sulphurous acid from the products of combustion of the gas, and error result.

Process 3.—Soluble sulphides may also be treated with excess of an alkaline arsenite, arsenous sulphide be then precipitated by the addition of hydrochloric acid, and the precipitate collected and weighed with the usual precautions (*vide* p. 593).

Weights of Equivalent Quantities of Sulphur and its Compounds.

Sulphur	S	32
Sulphuretted hydrogen	H_2S	34
Sulphate of barium	$BaSO_4$	232.8
Arsenious sulphide	$(As_2S_3) \div 3$	82
Bisulphide of iron	$(FeS_2) \div 2$	60
Sulphide of lead	PbS	238.5

SULPHITES.

Sulphites are usually estimated volumetrically by a standard solution of iodine (*vide* p. 629). Sulphites insoluble in water are diffused in that menstruum, hydrochloric acid added, and the iodine solution then dropped in.

If necessary, sulphites may be estimated gravimetrically by oxidation and precipitation in the form of sulphate of barium.

SULPHATES.

These salts are always precipitated and weighed as sulphate of barium, the manipulations being identical with those performed in the determination of barium by means of sulphates

(*vide* p. 645). The purity of Sulphate of Sodium (*Sodii Sulphas*, U. S. P.), and the presence of not more than a given amount of sulphuric acid in vinegar (*Acetum*, B. P.), are directed, in the British Pharmacopœia, to be ascertained by this process. Ten grains of sulphate of sodium yield 7.23 of sulphate of barium. Five ounces of vinegar should yield not more than about one-third of a gramme of sulphate of barium.

The amount of free sulphuric acid or hydrochloric acid in vinegar, lemon-juice, lime-juice, etc. may also be ascertained volumetrically by adding a known quantity of standard solution of soda, evaporating to dryness, incinerating, dissolving in water, and, by standard acid, estimating the quantity of soda still remaining free. The soda lost indicates the amount of free mineral acid (Hehner). Thresh estimates the chlorine in a sample of vinegar, adds a known additional amount of chlorine, preferably in the form of chloride of barium, evaporates, ignites; treats with water, adds bicarbonate of sodium to remove excess of barium, filters, and again estimates the chlorine. A loss of 70.8 of chlorine (Cl_2) indicates 98 of free sulphuric acid (H_2SO_4). The method of estimating free sulphuric, nitric, or hydrochloric acid proposed by Spence and Esilman is founded on their power of decolorizing a standard solution of ferric acetate.

Proportional Weights of Equivalent Quantities of Sulphates.

The sulphuric radical	SO_4	96
Sulphuric acid	H_2SO_4	98
Sulphate of barium	$BaSO_4$	232.8

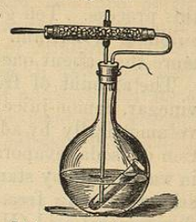
CARBONATES.

Carbonates are usually estimated by the loss in weight they undergo on the addition of a strong acid.

Process 1.—A small light flask is selected—of such a size that it can be conveniently weighed in a delicate balance. Two narrow glass tubes are fitted to the flask by a cork; the one straight, extending from about two or three centimetres above the cork to the bottom of the flask; the other cut off close to the cork on the inside and curved outward, so as to carry a thin drying-tube horizontally above the flask. (See Fig. 84.) The drying-tube is nearly filled with small pieces of chloride of calcium, a plug of cotton-wool preventing escape of any fragments at either end, and is attached by a pierced cord to the free extremity of the curved tube of the flask. A weighed quantity of any pure soluble carbonate is placed in the flask,

a little water added, a miniature test-tube containing sulphuric acid lowered into the flask by a thread and supported so that the acid may not flow out, the cork inserted, the outer end of the piece of the straight glass tube closed by a fragment of cork or wax, and the whole weighed. The apparatus is then inclined so that the oil of vitriol and carbonate may slowly react; carbonic acid gas is evolved and escapes through the horizontal tube, any moisture being retained by the chloride of calcium. When effervescence has ceased, the gas still remaining in the vessel is sucked out; this is accomplished by adapting a piece of India-rubber tubing to the end of the drying-tube, removing the small plug from the straight tube, and aspirating slowly with the mouth for a few minutes. If the heat produced by the action of oil of vitriol and solution is considered insufficient to expel all the carbonic acid from the liquid, the plug is again inserted in the tube and the contents of the flask gently boiled for some seconds. When the apparatus is nearly cold more air is again drawn through it, and the whole finally weighed. The loss is due to carbonic acid gas (CO_2), from the weight of which that of any carbonate is ascertained by calculation. Carbonates insoluble in water may be attacked by hydrochloric instead of sulphuric acid; granulated mixtures of carbonates and powdered tartaric or citric acid by enclosing the preparation in the inner tube and placing water in the flask, or *vice versâ*. The apparatus also may be modified in many ways to suit the requirements, convenience, or practice of the operator.

Fig. 84.



Estimation of Carbonates.

Process 2.—Carbonates from which carbonic acid gas is evolved by heat may be estimated by the loss they experience on ignition.

Process 3.—Free carbonic acid gas may be absorbed by a solid stick of potash or a strong alkaline solution, the loss in volume of the gas or mixture of gases indicating the amount originally present.

Weights of Equivalent Quantities of Carbonic Acid Gas and certain Carbonates.

Carbonic acid gas	CO_2	44
Carbonic acid	H_2CO_3	62
Anhydrous carbonate of sodium	Na_2CO_3	106
Crystalline carbonate of sodium	$Na_2CO_3,10H_2O$.	286
Anhydrous carbonate of potassium	K_2CO_3	138
Crystalline carbonate of potassium	$K_2CO_3+16\%$aq.	164.285
Carbonate of calcium	$CaCO_3$	100

OXALATES.

Process 1.—The oxalic radical is usually precipitated in the form of oxalate of calcium and weighed as carbonate, the manipulations being identical with those observed in the estimation of calcium (*vide* p. 646). The experiment may be performed on 0.3 or 0.4 grm. of pure crystallized oxalic acid, 126 parts of which should yield 100 of carbonate of calcium.

Process 2.—Oxalates may also be determined by conversion of their acidulous radical into carbonic acid gas, and observation of the weight of the latter. The oxalate, water, and excess of black oxide of manganese are placed in the carbonic acid apparatus (page 665), a tube containing oil of vitriol lowered into the flask, the whole weighed, and the operation completed as for carbonate. From the following equation it will be seen that every 88 parts of carbonic acid gas evolved indicate the presence of 126 parts of crystallized oxalic acid or an equivalent quantity of other oxalate:—

$$Na_2C_2O_4 + MnO_2 + 2H_2SO_4 = MnSO_4 + Na_2SO_4 + 2H_2O + 2CO_2.$$

The black oxide of manganese used in this experiment must be free from carbonates. The amount of materials employed is regulated by the size of the vessels.

PHOSPHATES.

Process 1.—From phosphates dissolved in water the phosphoric radical may be precipitated and weighed in the form of pyrophosphate of magnesium, the details of manipulation being similar to those observed in estimating magnesium (*vide* p. 647). Half a gramme or rather more of pure dry crystallized phosphate of sodium may be employed in experimental determinations. The official phosphate of ammonium (*Ammonii Phosphas*, U. S. P.) is quantitatively analyzed by this method. "2 gm. of the salt, dissolved in water and precipitated with test-mixture of magnesium, yields a crystalline precipitate, which, when washed with diluted water of ammonia, dried, and ignited, should weigh 1.68 gm." Half a gramme or less is a more convenient quantity if the operations be conducted with care. Solution of ammonia-sulphate of magnesium (U. S. P.) is prepared by dissolving 1 part of sulphate of magnesium, 2 of chloride of ammonium, and 4 of solution of ammo-

nia (10-per cent. NH_3) in 8 of distilled water; such a solution is of considerable use if several phosphoric determinations are about to be made.

Process 2.—Free phosphoric acid is most readily determined as phosphate of lead (Pb_32PO_4). Of the official solution of phosphoric acid it is stated that "on pouring 5 gm. of phosphoric acid upon 10 gm. of oxide of lead free from carbonate of lead and from moisture, evaporating and igniting, a residue will be obtained which should weigh 11.81 gm." In the case of the diluted acid, 5 gm. with 5 of lead oxide should yield 5.36. The oxide of lead must be quite pure: it should be prepared by digesting red lead in warm dilute nitric acid, washing, drying, and heating a resulting puce-colored plumbic oxide in a covered porcelain crucible. The increase in weight obtained on evaporating a given amount of solution of phosphoric acid with a known weight of perfectly pure oxide of lead (PbO) may be regarded as entirely due to phosphoric anhydride (P_2O_5),

$$3PbO + P_2O_5 = Pb_32PO_4,$$

the actual reaction being

$$3PbO + 2H_3PO_4 = Pb_32PO_4 + 3H_2O.$$

From these equations and the table of atomic weights (*vide* Appendix) the percentage of phosphoric acid (H_3PO_4) in any specimen of its solution may be easily calculated.

Process 3.—The strength of pure solution of phosphoric acid may be ascertained by specific gravity and reference to Tables.

Process 4.—Bone-earth, "superphosphate," the *Calcis Phosphas* of pharmacy, and other forms of phosphate of calcium known to be tolerably free from iron or aluminium, may be estimated by treating about half a gramme with hydrochloric acid somewhat diluted, filtering if necessary, warming, precipitating with excess of ammonia, collecting the precipitate (Ca_32PO_4), washing, drying, igniting, and weighing. "*Calcis Phosphas*," if pure, will in this process lose no weight.

Process 5.—Insoluble phosphates in ashes, manures, etc. are treated as follows: A weighed quantity of the material (1.0 to 10.0 grm.) is digested in hydrochloric acid diluted with three or four times its bulk of water; filtered (insoluble matter and filter being thoroughly exhausted by water); ammonia added to the filtrate and washings until, after stirring, a faint cloudy precipitate is perceptible; solution of oxalic acid dropped in until, after agitation for a few minutes, the opalescence is destroyed; oxalate of ammonia next added, the whole warmed, oxalate of calcium removed by filtration, and the filtrate concentrated if very dilute; the liquid treated with citric acid in such quantity that ammonia when added in excess gives a clear *lemon-yellow* solution (Warington), magnesian mixture poured in (as in Process 1), and the precipitate of ammonio-magnesian phosphate collected, washed, dried, and weighed, as already described in connection with the estimation of magnesium.

Relative Weights of Equivalent Quantities of Phosphoric Compounds.

Phosphoric acid	H_3PO_4	98
Pyrophosphate of magnesium .	$(Mg_2P_2O_7 = 222) \div 2 =$	111
Phosphate of lead	$(Pb_32PO_4 = 811) \div 2 =$	405.25
Phosphoric anhydride . . .	$(P_2O_5 = 142) \div 2 =$	71
Phosphate of calcium . . .	$(Ca_32PO_4 = 310) \div 2 =$	155
Superphosphate of calcium .	$(CaH_42PO_4 = 234 \div 2 =$	117

QUESTIONS AND EXERCISES.

1057. What quantity of pure rock-salt is equivalent to 4.2 parts of chloride of silver? *Ans.* 1.714.

1058. State the percentage of real iodide of potassium contained in a sample of which 8 parts yield 10.9 of iodide of silver. *Ans.* 96.3.

1059. What is the strength of a solution of hydrocyanic acid 10 parts of which, by weight, yield .9 of cyanide of silver? *Ans.* 1.82 per cent.

1060. How are nitrates quantitatively estimated?

1061. By what processes may the strength of sulphides be determined?

1062. How much real sulphate of sodium is contained in a specimen 10 parts of which yield 14.2 of sulphate of barium. *Ans.* 86.61 per cent.

1063. Give details of the operations performed in the quantitative analysis of carbonates.

1064. What amount of carbonic acid gas should be obtained from 10 parts of acid carbonate (or bicarbonate) of potassium? *Ans.* 4.4 parts.

1065. To what operation and what proportion of materials does the following equation refer?—

$$Na_2C_2O_4 + MnO_2 + 2H_2SO_4 = MnSO_4 + Na_2SO_4 + 2H_2O + 2CO_2.$$

1066. Explain the lead process for the estimation of phosphoric acid in the official solution.

1067. State the amount of superphosphate of calcium equivalent to 7.6 parts of pyrophosphate of magnesium. *Ans.* 8.01.

SILICATES.

Silica (SiO_1) may be separated from alkaline silicates, or from silicates decomposable by hydrochloric acid, by digesting

the substance in hydrochloric acid at a temperature of 70° to 80° C. until completely disintegrated, evaporating to dryness, heating in an air-bath, again moistening with acid, diluting with hot water, filtering, washing, drying, igniting, and weighing.

ESTIMATION OF WATER.

Water and other matters readily volatilized are most usually estimated by the loss in weight which a substance undergoes on being heated to a proper temperature. Thus, in the British Pharmacopœia crystalline gallic acid ($H_3C_7H_3O_5,H_2O$) is stated to lose 9.5 per cent. of its weight at a temperature of 100° C., oxalate of cerium ($CeC_2O_4,3H_2O$) 52 per cent. on incineration, carbonate of potassium about 16 per cent. on exposure to a red heat, sulphate of quinine ($2C_{20}H_{24}N_2O_2,H_2SO_4,7H_2O$) 14.4 per cent. at 100° C., arseniate of sodium ($Na_2HAsO_4,7H_2O$) 40.38 per cent. at 149° C., carbonate of sodium ($Na_2CO_3,10H_2O$) 60.3 per cent., phosphate of sodium ($Na_2HPO_4,12H_2O$) 63 per cent., and sulphate of sodium ($Na_2SO_4,10H_2O$) 55.9 per cent. at a low red heat; oxide of bismuth heated to incipient redness should not diminish in weight.

Process.—One or two grammes of substance is sufficient in experiments on desiccation, the material being placed in a watch-glass, covered or uncovered porcelain crucible, or other vessel, according to the temperature to which it is to be exposed. Rapid desiccation at an exact temperature may be effected by introducing the substance into a tube having somewhat the shape of the letter U, sinking the lower part of the tube into a liquid kept at a definite temperature by aid of a thermometer, and drawing or forcing a current of dry air slowly through the apparatus. Substances liable to oxidation may be desiccated in a current of dried carbonic acid gas. The weights of the U-tube before and after the introduction of the salt, and after desiccation, give the amount of water sought. In all cases the material must be heated until it ceases to lose weight. Occasionally it is desirable to estimate water directly by conveying its vapor in a current of air through a weighed tube containing chloride of calcium and reweighing the tube at the close of the operation; the increase shows the amount of water.

Note.—Highly dried substances rapidly absorb moisture from the air; they must therefore be weighed quickly, enclosed, if possible, in tubes (p. 640), a pair of clamped watch-glasses, or a crucible having a tightly fitting lid.

CARBON, HYDROGEN, OXYGEN, NITROGEN.

The quantitative analysis of animal and vegetable substances is either *proximate* or *ultimate*. Proximate quantitative analysis includes the estimation of water, oil, albumen, starch, cellulose, gum, resins, alkaloids, acids, glucosides, ash. It requires the application of much theoretical knowledge and manipulative skill, and cannot well be studied except under the guidance of a tutor. One of the best of the published works on the subject is by Rochleder, a translation of whose monograph will be found in the *Pharmaceutical Journal*, vol. i. 2d ser. pp. 562, 610; vol. ii. 2d ser. pp. 24, 129, 160, 215, 274, 420, 478. Another is a small book by Professor A. B. Prescott, *Outlines of Proximate Organic Analysis*, Van Nostrand, New York.

Ultimate quantitative organic analysis can only be successfully accomplished with the appliances of a well-appointed laboratory—a good balance, a gas-furnace giving a smokeless flame (7 or 8 centimetres wide and 70 or 80 centimetres long), special forms of glass apparatus, etc. *The theory of the operation* is simple: A weighed quantity of a substance is burnt to carbonic acid gas ($CO_2 = 44$) and water ($H_2O = 18$), and these products collected and weighed; 12 parts in every 44 of carbonic acid gas ($= \frac{3}{11}$) are carbon, 2 in every 18 of water ($= \frac{1}{9}$) are hydrogen; nitrogen if present escapes as gas. If nitrogen be a constituent, more of the substance is strongly heated with a mixture of the hydrates of sodium and calcium; these bodies then split up into oxides, oxygen, and hydrogen; the oxygen burns the carbon of the substance to carbonic acid gas, its hydrogen and nitrogen appearing as water and ammonia respectively; the carbonic acid and water are disregarded, the ammonia collected and weighed in the form of a double chloride of platinum and ammonium ($PtCl_4 2NH_4Cl = 442.8$), of which 28 parts in every 442.8 ($= \frac{1}{16}$) are nitrogen. The difference between the sum of the weights of hydrogen and carbon, and the weight of substance taken, is the proportion of oxygen in the body, supposing nitrogen to be absent. If nitrogen is present, the difference between the sum of the percentage of carbon, hydrogen, and nitrogen, and 100, is the percentage of oxygen. Shortly, carbon is estimated in the form of carbonic acid gas, hydrogen as water, nitrogen as ammonia, and oxygen by loss.

The following is the outline of the necessary manipulation:—The source of the oxygen for the combustion of carbon and hydrogen is black oxide of copper in coarse powder. 200 or 300 grammes of this material are heated in a crucible to low redness for a short time to expel every trace of moisture; then transferred to store-tubes (Fig. 85) resembling test-tubes, half

Fig. 85.

a metre long, and having a slightly narrowed mouth, the tube being held in a cloth to protect the hand while the hot oxide