A handbook of chemical manipulation

Williams, Charles Greville

London, 1857

Section VII. Operations Preparatory to Weighing

SECTION VII.

OPERATIONS PREPARATORY TO WEIGHING.

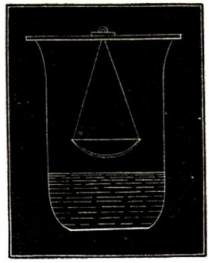
83. The estimations in chemical research are performed either by weight or measure; and as measures by bulk are founded upon, and referred for verification to standard weights, weighing becomes the chief means of quantitative determination; and as analysis depends for its successful performance upon the accuracy with which we estimate the weights of the substances eliminated by the various chemical methods used, it is essential to the student's progress that he should become perfectly familiar with all the manipulations connected with the use of the balance.

84. The most usual way of preparing substances for the balance-pan, is to convert them into the form of precipitates of known value, *i. e.* when dry, contain, or are equivalent to, a known amount of the substance to be estimated. But although this last-named method is the most common, it is by no means the only way in which we separate substances from the compounds which contain them; crystallization is not unfrequently as successful a means of separating bodies. In other cases compounds are exposed to temperatures capable of expelling one or more constituents, and leaving a residue of known composition in a state fit for estimation. It is also a frequent occurrence to expose mixtures to an elevated temperature, in a current of some gas which forms a volatile compound with one of its ingredients, which is accordingly expelled, leaving a residue capable of being weighed, as in the separation of antimony from lead. In these cases, and also in all the others which are met with in analytical research, it is of course absolutely essential that the substance to be weighed should exist in a state in which its condition is perfectly known, and more especially its relation to water. Chemists who are inattentive to this are constantly liable to the most serious errors. Even where precipitates will endure heating to redness, they

not unfrequently absorb moisture during cooling; in such instances they should be placed, if in a platinum, silver, or gold crucible, upon a large clean block of iron, which will remove the heat so rapidly by conduction, that if the lid is tolerably tight no appreciable moisture will be absorbed during the refrigeration. It is imperatively necessary to let the capsule or other vessel in which the ignition was made become quite cold before being placed in the balance-pan, as otherwise errors are occasioned by currents of air, which make the vessel and contents appear lighter than they really are. Another reason why it is essential to allow vessels to become of the temperature of the atmosphere before weighing is, that they may attain a state of hygrometric repose, otherwise they would keep for several minutes acquiring weight, as a film of moisture gradually deposited on the metal or glass.

85. Wet precipitates, or crystals, require very different treatment, according to their various natures: sometimes it is necessary to weigh substances which are either destroyed or have their constitution altered, by the slightest elevation of temperature; it is then usual to expose the matter in watch-glasses or capsules to a surface capable of rapidly absorbing moisture, such as oil of vitriol, quicklime, or chloride of calcium. If placed, under these circumstances, beneath the receiver of an air-pump, the operation is much hastened, but frequently an exposure to a desiccating surface in the manner figured in the margin (fig. 60) for twelve to twenty-four hours is quite sufficient. The chemist is scarcely ever at a loss for a substance capable of rapidly absorbing moisture; if neither of the substances mentioned above are at hand, which is seldom the case, chloride of zinc, acetate, carbonate or caustic potash may be used. It must of course be ascertained with certainty that the precipitate or other substance to be weighed is not only in appearance, but in reality, dry; this may be known by weighing at intervals, until after some hours' exposure it does not decrease in

Fig. 60.



weight. It is extremely convenient in this operation to have two watch-glasses of the same size ground together and provided with a brass clip, as in fig. 61. During the drying the substance is contained in one of the glasses as in fig. 60, and when it is wished to weigh, the other is placed on as a cover and kept in its place with the clip; it is then balanced in this manner. The weight of the glasses is of course ascertained previously, and may be scratched with a diamond on each of them. Some precautions in regard to the drying of organic preparations will be treated of in the section on the Manipulations in Organic Analysis. The copper hot-air chamber (fig. 52) is very convenient for drying at known temperatures, as the heat is indicated by the thermometer and may be easily regulated.

Fig. 61.

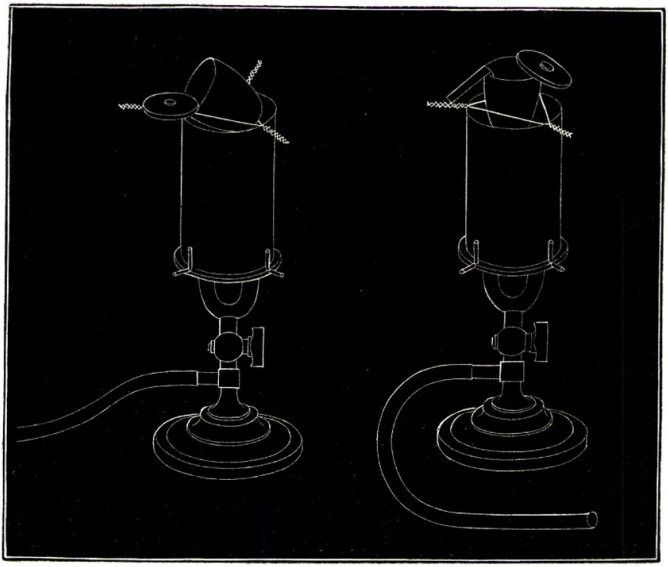
86. *Ignition of Precipitates.*—In most of the operations in mineral analysis, the precipitates, after being filtered off and washed, are dried, heated to redness, and, when cold, weighed; the operation requires, however, many modifications, according to circumstances. Where the precipitate is not affected by the ashes of the filter, as alumina, silica, carbonate of lime, &c., as soon as it is washed it is to be dried, which may be performed in many ways according to the means at hand; for instance, the aperture in the funnel may be stopped with a cork, and it may be placed in one of the apertures of the steam-bath (figs. 53 or 55), or the funnel may be placed in the hot-air chamber of the furnace, or on the ring of the retort-stand, and the latter may be placed on the plate of the furnace, fig. 1; or, if the paper-filter is sufficiently strong to bear removing from the funnel, it may be placed upon a porous tile, and, when the moisture is sufficiently absorbed, it is to be opened out, and will soon become sufficiently free from moisture to have the exsiccation completed in any of the other methods either already described, or to be pointed out further on. The hot tile is also extremely useful in drying preparations; of

course care must be taken to prevent absorption of any saline matters from the tile itself. Sometimes the filter dried at 212° may be weighed, and the substance collected on it; after washing and subsequent drying, it is to be weighed again, when the increase will indicate the quantity of the precipitate. Before adopting this method, it must be ascertained that the filter contains no soluble matters capable of being removed during the washing, or, if such be the case, the paper should be previously prepared. When the substance is to be heated to redness, it must first be considered whether it is capable of injuring the platinum crucible, and, if so, one of porcelain must be substituted; also, whether the ashes of the filter can do any injury through the carbon they contain. If not, the crucible is to be placed upon a sheet of highly glazed paper, the edges of which are quite smooth, so as to be incapable of retaining any of the powder. The filter is then to have its contents removed, either by shaking or with the aid of a spatula; if any of the substance remains on the latter, it should be wiped with a piece of the filter-paper, which is to be added to the rest; the crucible is now to be placed in a slanting position over the lamp, and, when red-hot, the filter, cut to small pieces with scissors, is to be placed in it, waiting until the flame has disappeared each time before adding more; when the whole of the filter has been added, the lid of the capsule is to be laid in the manner indicated in the engraving, fig. 63. By this means a gentle current of air will be induced in the crucible, too gentle to remove any of the ashes, but sufficient to entirely consume the filter to a white ash in a short time, unless the substance is somewhat soluble in the water used to wash the precipitate, as sometimes happens; when this is so, a little nitric acid may, in many cases, be put into the crucible, when cold, upon the substance, and the heat being again raised to redness, the filter will soon disappear.

Where this is not admissible, from any action taking place between the acid and the assay, the filter may be washed at the last with a rather dilute solution of nitrate of ammonia, which will, to a considerable degree, effect the same thing. It is often more convenient to take the dry filter and its contents, and, after fold-

ing it together, to insert it at once in the crucible, and place the latter vertically over the flame with the lid on loosely, until no

Fig. 62. Fig. 63.



more combustible gases are evolved, when the crucible may be arranged in the position seen in figs. 62 or 63, preferably the latter.

87. Where, from the easy alterability of the substances at a high temperature in presence of carbon, it is impossible to consume the filter in contact with it, the assay must be carefully removed from the latter, and be burnt alone, the paper being consumed upon the lid; and when the black colour has entirely disappeared, it may be added to the contents of the crucible, except where the ignited substance is wanted for ulterior examination. It is supposed that the weight of the crucible has been taken previous to the ignition, which is by far the best plan; in this case the weight so obtained has merely to be added to the weight of the

filter-ash, and the sum being deducted from that of the crucible and the ignited matter, we obtain the weight of the latter.

88. It is advisable to use the circular cut filters alluded to at p. 4. By taking a few of these, and burning them in a platinum crucible until all the carbon is oxidized, and dividing the weight of ash by the number of filters used, we obtain a number to be always deducted from the weight of the substance ignited with the filter. It is essential to perform these operations in a place quite protected from air-currents, which would endanger the loss of a portion of the assay; if this should happen, even to the slightest extent, it is imperative to reject the estimation, as it is impossible to know how much has been removed by the accident.

89. It is advisable to make precipitates tolerably dry before igniting them, or a chance is incurred of the substance being partially ejected during the operation, especially if it has a tendency to fly about when heated in a damp state. Where there is any danger of loss from decrepitation the lid may be kept on until it is over.

90. The manner of procedure is liable to so many variations according to circumstances, that it would be impossible to mention all; the operator will, therefore, do well to reflect before he ignites a substance with the habitude of which he is not perfectly familiar. Platinum salts of the volatile organic bases require precautions which may be mentioned, from the frequency with which they occur in some trains of investigation. In the first place, the oily bases form salts with platinum which are liable during ignition to rise like a horn out of the crucible, sometimes to an inch or more in height; care must therefore be taken to heat gently, or a loss of the metal will be incurred. In the next place, the crystals have a peculiar tendency to retain an oily or resinous impurity, which renders it inadmissible to burn them without previously pulverizing, and washing them with alcohol or ether, or a mixture of both, according to their solubility. Previous to washing, the powder generally hangs together as if moist, but afterwards it is as mobile as dry sand. It is necessary also to turn the mass of spongy metal after the ignition, so as to expose the under surface

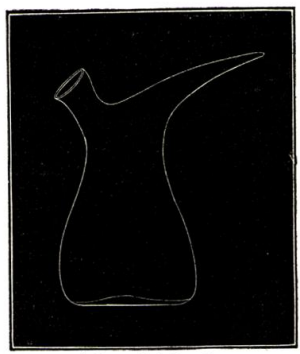
to the air, when it will be found to glow again from the combustion of the previously unconsumed carbon.

The tendency of silver to form explosive salts with many organic acids, especially those containing nitrogen, must not be lost sight of. The necessity of thoroughly washing precipitates previous to weighing them, and the method of ascertaining the complete removal of the precipitant, will be found in their proper places.

91. *Apparatus to contain substances while being weighed.*—Non-hygroscopic substances may be weighed in small capsules. Where the assay is liable to absorb moisture, a platinum crucible, with a well-fitting cover, is frequently the best instrument. If the substance is excessively deliquescent, it is advisable to add it to a counterpoised vessel of water, and ascertain the increase of weight; the same method, or a modification of it, is sometimes required in weighing substances which emit vapours at ordinary temperatures. Organic substances are generally weighed in small, wide-mouthed, stoppered bottles, or test-tubes with good corks. If in the former, the weight may be scratched on with a diamond; if in the latter, it should be written on the cork.

92. When liquids, such as acid or alkaline solutions, are to be added in known quantities to another until neutrality or some other point is reached, Schuster's alkalimeter (fig. 64) is much used, and is extremely convenient from the facility with which the drops may be regulated by the pressure of the finger upon the tubulure, and thus admitting more or less air. If filters with precipitates upon them have to be weighed after drying at 212°, it is necessary to effect the operation in a closed vessel, in consequence of the rapidity with which dry paper absorbs moisture: in this case the ground watch-glasses previously mentioned are convenient; or a very wide test-tube,

Fig. 64.



fitted with a good cork, may be used. The weight should be ascertained when empty, or, what is perhaps better, counterpoises for all the platinum crucibles, porcelain capsules, and other vessels in which weighings are to be effected, may be constructed from pieces of lead or brass; these being placed in the other pan of the balance, considerably facilitate the estimations.