A handbook of chemical manipulation

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Section XVII. Evaporation

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EVAPORATION.

310. The process of evaporation is one constantly employed in the laboratory: its object is to separate fluids from solids by volatilization of the former, and is opposed to distillation, from the fact that in the latter operation it is generally the fluid which is the valuable product, whereas in evaporation it is invariably the solid matter which it is sought to obtain.

It is generally a very easy process; the apparatus is commonly of the simplest and most inexpensive kind, but there are nevertheless many precautions to be taken with which it is essential the student should be familiar. Evaporation is very frequently the first step in crystallization, but as the latter operation is of every-day employment in all laboratories and in all kinds of researches, and is one involving some detail, it will have a section exclusively devoted to it.

311. *Evaporation over naked fire.*—In scientific research, it is not usual to trust fluids under examination in porcelain vessels over the naked fire; it is true that with care most porcelain will endure the temperature without fracture, but then it becomes necessary to devote more attention to the progress of the operation than can generally be spared. Moreover, it is not safe to risk contamination with the smoke and dust which furnaces, unless managed with extreme care, are liable to evolve. For these reasons, evaporation over the naked fire is more generally resorted to in the comparatively rough operations of technical and manufacturing chemistry.

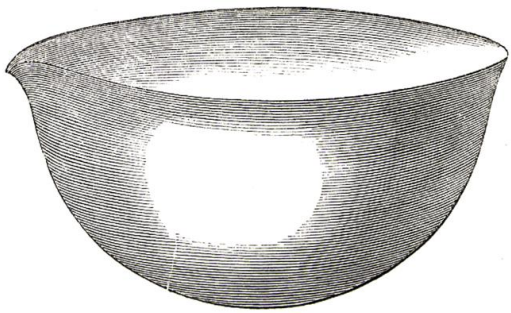
It may be mentioned, however, that the rings of the furnaces (figs. 1 and 17) are well adapted to support basins of different sizes over the fire.

312. The remarks in § 311 apply, although with somewhat less force, to exposure of evaporating dishes to the flame of a lamp; and it may be taken as a general rule, that if a flame

passes over the line formed by the height of the fluid in the basin, the latter will be fractured. If, therefore, it is necessary to expose a porcelain or glass vessel over a lamp-flame without the intervention of sand or other medium, care should be taken to prevent actual contact of the flame with the bottom of the vessel. If, for the flask supported over the gas-furnace in fig. 28, a basin be substituted, as in fig. 29, a very effective mode of evaporation is obtained; but, nevertheless, there are many analytical operations in which a wide-mouthed flask like that in the engraving alluded to, may be used with advantage in evaporating over the flame of a gas-lamp.

Where rapidity of evaporation is the chief object, as in evaporating saline liquids to dryness to obtain the solid matter, it is of course desirable that the vessel selected should be as open as possible, to enable the steam to escape without hindrance; in these cases, the basins shown in figs. 165, 166, and 167 are

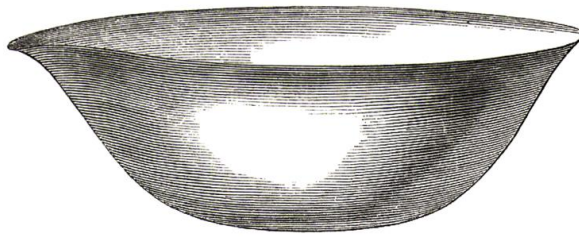
Fig. 165.



very well adapted for the purpose. They should be of the Bayeux or Meissen ware, both of which are extremely thin and light, and are therefore well adapted to withstand sudden changes of temperature. Their thinness, however, renders great care necessary in their use, especially in stirring the pasty mass which is generally obtained towards the end of the process, as if the stirring-rod is roughly handled, it is easy to push it through the bottom of the basin.

313. Almost all liquids containing solid matters in suspension emit while boiling, small drops, which are thrown up to a considerable height; the greater portion of these fall back into the basin, but others are carried over and become lost; this renders it very improper to let liquids in basins boil where the solid matter has to be estimated with precision; it is true, that by

Fig. 166.



placing another basin somewhat larger, over the one containing the boiling fluid, much of this loss is avoided, but then a large quantity of the steam formed condenses upon the basin used as a cover and falls back, thus greatly retarding the operation, and making it far more convenient, as a general rule, to expose the basin to the heat of the water- or sand-bath. In fact, as has been previously indicated, evaporation over the naked fire is

Fig. 167.

more adapted to the copper pans of the pharmaceutist than to the porcelain vessels of the scientific chemist. Almost all saline fluids, as they become thick and pasty by evaporation, require to be carefully stirred, to assist in the escape of the vapour arising from them; if this precaution be not attended to, violent bursts of steam take place, which are liable to give rise to considerable loss of material by projection, and even to cause the fracture of the vessel. Not only does stirring prevent this, but it

also enables the operation to be completed in a much less time than would otherwise be necessary.

314. Evaporations requiring to be performed without loss, as in the concentration of mineral waters or the estimation of their solid contents, are best performed in flasks during the first stage of the process, that is, until they are so far reduced in bulk that they may be transferred to an evaporating basin, in order that the operation may be concluded on the sand-bath. The evaporating basins for this purpose are best when made of platinum or silver. It must be remembered, that some flasks are constructed of such bad glass that they yield soluble matters with considerable facility when waters are boiled in them for a long time. When this is the case, it is better to evaporate the liquid in porcelain basins over the gas-furnace; it is not generally safe to place them upon the sand-bath.

315. When evaporations are to be conducted in flasks until nearly all the fluid is expelled, or if any effervescence takes place or the boiling is violent, it is a good plan to have a small capsule placed in the neck of the flask, to avoid loss of solid matters without preventing the escape of the steam. It is essential, where evaporations in open basins are taking place, to prevent the ingress of dust; this may be accomplished in many ways, two of which may be mentioned. If a diaphragm of bibulous paper be placed at a little distance above a fluid which is evaporating without ebullition, it will prevent the dust from falling in without much retarding the evaporation, or a plate of glass may be laid on the top of the basin without quite covering it. A very convenient plan is to support a large piece of glass by two bricks at a few inches above the vessel in which the operation is proceeding, or even a plank may be used, but it must be placed so far away that no vapour will condense upon it and fall back into the dish, as it would probably cause the introduction of impurities, which would in some instances involve the necessity of throwing away the material under examination.

316. If the matter operated upon is injured or decomposed by too high a temperature, it may be placed upon the water-bath;

this will prevent undue elevation of the heat, but at the same time retard the process. It must not be forgotten that stirring so greatly facilitates the operation, as often to reduce the time required more than half.

317. In many analytical operations of this class the gas sand-bath (fig. 29) is extremely convenient, and, moreover, allows of the regulation of the heat with considerable nicety.

318. It is not at all an uncommon occurrence to have to evaporate liquids and dry precipitates which are decomposed by even very moderate degrees of heat; in these instances it is necessary to resort to the use of substances which have a great tendency to absorb moisture; among which may be mentioned sulphuric acid, quicklime, chloride of calcium, nitrate of magnesia, chloride of zinc, &c. Those most generally used are the three first, and they may be made available either with or without the assistance of the air-pump. The simplest way of using these desiccating media is by placing them in a porcelain or glass vessel, and supporting the fluid to be evaporated upon a triangle of wire or glass, or a piece of wire-trellis over the drying substance: the whole arrangement is placed upon a piece of ground glass, and is covered with a glass shade or bell-jar having its edge ground true and greased; by this means the damp air is prevented from entering, and as the absorbent gradually removes the moisture from the included air, the fluid parts with its water, and this goes on until the fluid is dried up or the precipitate completely desiccated.

If the apparatus, instead of being merely placed under a bell-jar, be put on the plate of an air-pump and exhaustion is effected, the process will be brought much more speedily to a termination. In either case the operation is necessarily rather slow.

319. It is well known that the point at which liquids boil is the higher the greater the weight of the atmosphere, as shown by the barometer; and from this it will readily be seen that *in vacuo* the boiling-point of liquids is very considerably lower than when under the ordinary circumstances of atmospheric pressure: this principle has been much made use of in many manufacturing operations on the large scale, particularly in sugar-works and in

the preparation of the more delicate and easily injured pharmaceutical extracts; it is seldom if ever required to operate in this manner in chemical research, and a description of the manipulation would be foreign to the plan of this work; but evaporation *in vacuo at ordinary temperatures* is, on the contrary, an operation of every-day occurrence in the laboratory, and will therefore be described.

The air-pump, to be practically useful in experiments of this kind, must not only be capable of effecting a good vacuum, but, what is equally necessary, be able to retain it for several days, as the process of exsiccation *in vacuo*, although a very perfect one for removing mechanically or feebly combined water, is slow, and some substances require a week's exposure to effect a perfect drying.

320. One of the most convenient methods of operating, is to place a glass vessel containing sulphuric acid upon the plate of the air-pump and cover it with a very coarse brass-wire trellis, on which the substances to be dried are supported in capsules or watch-glasses; (it is an excellent plan to have a number of the latter always at hand, their surfaces having been ground to fit each other); if, now, the substance to be dried is weighed between them, and the upper one be removed and put underneath, and both are placed in the receiver together, it is easy to weigh the apparatus again in the same manner as at first, after a certain exposure to the absorbent surface, and by this means all danger of increase from atmospheric moisture during the weighing is prevented (§ 85).

321. It should be remembered, that in all cases of drying by exposure to desiccating agents, the rapidity is greater the more extended the surface and the thinner the stratum; it would be difficult, therefore, to find anything better adapted to purposes of this kind than watch-glasses; small capsules may, it is true, be used, but they are generally far less convenient. In order to ascertain whether the substance is perfectly dried, it may be weighed and exposed for a day or two and then reweighed; if no difference is observed, no further exposure is required, but if loss

is found it must be noted, and the substance is again to be desiccated for another day or two, and this alternate drying and weighing is to be continued until two estimations at tolerably long intervals are found to be the same.

322. There are many organic bodies which, although uninjured when heated to 212° in a nearly dry state, are, nevertheless, rapidly decomposed if introduced into the water-bath damp; such substances should be first dried over sulphuric acid until all outward appearance of moisture is removed, and may then be placed in the hot-air chamber or water-bath until perfectly dry. Some substances of a very explosive kind must be entirely dried *in vacuo*, as it would be dangerous to raise them to 212°, although it may be known that in general they require a higher temperature for explosion, as they are frequently very uncertain in their behaviour at moderately elevated temperatures, and it is singular that in a damp state some substances explode more readily than when dry; this is frequently the case with gun-cotton.

Where a great many desiccations have to be effected, it would be inconvenient to use more than one air-pump, and various methods have been contrived to enable one instrument to exhaust several receivers without adding very greatly to the expense; one, but not the most economical, is to have several ground plates capable of being attached to and removed from the pump, and having taps beneath to enable them to be closed before being removed.

323. Another process is that of Mr. Cooper's, recommended by Faraday, which consists of two plates of thick glass ground perfectly true, and having a small hole through each about one-quarter of a diameter away from the edge; these plates, with the holes opposite each other, are placed upon the pump plate, a tolerably plentiful supply of grease being rubbed over them; the receiver is then put in its position and the air exhausted; when a sufficient vacuum is obtained the plates are slightly moved, so that the two holes no longer coincide; they may then be removed without air penetrating into the receiver. Of course, by having several double pairs of plates, any number of receivers may be exhausted with one air-pump.

324. The sulphuric acid used for these purposes becomes diluted by frequent use, and must be replenished as this takes place: it is a good plan to have a mark made upon the glass pan which contains it, in order that the amount of increase may be known. When six parts have become seven, its action is too feeble to be worth keeping in the pan; it should therefore be removed and have fresh acid put in its place. It is advisable not to use this acid for any experiments of importance, as it generally contains many impurities; it will, however, answer very well for making hydrogen, sulphuretted hydrogen, or other purposes which do not require it to be pure.

325. Under certain circumstances, many other preparations having great affinity for water may be used instead of sulphuric acid, they have been alluded to in describing desiccation without the air-pump.

326. It has been said that some organic substances when introduced into the water-bath in a damp state are decomposed, although they bear the same temperature very well if previously air-dried; it is remarkable, also, that the presence of water in some cases tends to confer fusibility. Warren De la Rue found that carminic acid fuses if heated to 248° Fahr., unless it has previously been dried *in vacuo*. It is, moreover, by no means to be taken for granted in all cases, that a substance is sufficiently dry for analysis because it ceases to lose weight *in vacuo* over sulphuric acid, as many substances retain the last portions of water with such obstinacy as to render exposure to a temperature of 212° Fahr., or even higher, absolutely necessary.