# A handbook of chemical manipulation

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Section XVI. Pressure-Tube Operations

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## SECTION XVI.

## PRESSURE-TUBE OPERATIONS.

303. In the progress of modern organic chemistry a new method of manipulation has been gradually developed, and now become very generally adopted in certain trains of research. It consists in exposing substances to the action of reagents at an elevated temperature under considerable pressure. The reason for this procedure is, in general, that the reactions sought are most frequently sluggish at ordinary temperatures, and as the reagents used are often volatile, it would be difficult to effect the intended change by mere digestion in the ordinary way. It is true that an apparatus might be made, by means of which substances could be exposed to the action of volatile reagents at temperatures not sufficiently great to injure corks, but then it would be impossible to construct it so as to withstand a very powerful pressure; now, by the process about to be described, this objection is removed, and any temperature or pressure may be attained short of that capable of bursting the tube. Before proceeding to describe the process, it may be mentioned that the iodides and bromides of ethyl, methyl and amyl are greatly used in experiments of this description, as in the splendid research of Hofmann on the bases of the alcohol radicals, Frankland's isolation of the hydrocarbons forming the radicals of the same series, &c.

304. It is not always necessary to expose the pressure-tubes to a considerably elevated temperature, that of 212° Fahr. being sufficient in the greater number of instances; sometimes, however, a saline solution or oil-bath is required to raise the heat to the required degree, but then the tubes should be of greater strength than in the former case. It is generally better to have them of rather small bore, and to employ a greater number, than to use a few of considerable diameter, as the former are capable of withstanding greater pressures. To construct a pressure-

tube, it is merely necessary to seal one end and to keep it in the blowpipe-flame, turning it round constantly until it has become of the same thickness as the other parts. It is now to be heated strongly about 12 or 14 inches from the closed end, and dexterously turned in the flame until the glass has become of considerable thickness; it is then to be drawn out until of the form seen in fig. 162.

Fig. 162.


The part *a* is to serve the purpose of a funnel, and, if the liquid to be introduced is poured into it and a little air expelled, by warming *c*, the liquid rushes into the tube as the latter cools and the air consequently decreases in volume; by repeating this two or three times a sufficient quantity will have been introduced, and the tube is then to be closed by directing a blowpipe-flame upon the part *b*. It is sometimes advisable to reduce the amount of air in the tube, so that the only outward pressure, on exposure of the tube in the bath, will be caused by the vapour of the liquid and not by the expansion of the included air. This is readily managed by heating the liquid until it boils, and sealing the tube before the vapour has condensed. The tube is now of the shape seen in fig. 163, and this is a better form than fig. 164, because it admits of having the point broken off, and then being used again and again until the fine tube has become too short. No fear need be entertained of the tube bursting at the small portion if moderate care be used in its construction, as from its small internal diameter it is capable of withstanding a greater pressure than the large one; nevertheless, where very high temperatures are to be applied, it is advisable to thicken the glass of the larger tube at the point where the small one joins on, before introducing the substance. The tubes are now to be labelled, which is easily done by tying on them small pieces of zinc with the name of the contents scratched on, or still better, written on the clean metal with a quill-pen dipped in a weak solution of chloride of platinum, the latter forming an indelible and jet-black stain upon the zinc. If the tubes are to

be exposed only to the temperature of boiling water or a saline solution, they may generally be immersed as they are, but if any fear of their bursting is entertained, they may have a piece of rag tied round them, so that if such an accident should happen, the fragments may not be projected about, or the others in the bath be broken by the concussion. It is extremely convenient to have a tin-plate pierced with holes inserted in the bath, so that the tubes may be supported vertically, or, if this is not used, a small piece of lead may be enclosed in the rag used to cover the tubes, so as to sink them in the bath. Where high temperatures are employed, and, consequently, the pressures become very great, it is advisable to introduce the tubes into a pistol-barrel with a plug capable of being screwed in; a piece of stout iron gas-pipe answers very well. A small piece of tow first being thrust to the bottom of the barrel, the tube is to be introduced, then a little more tow, and, finally, the plug is to be screwed in. Where an oil-bath is required, it is frequently convenient to substitute tallow, as it is not so liable to emit unpleasant vapours. If solutions are employed as the media for applying heat, great care must be taken that the bath does not boil dry, as in this case explosions may occur, which, if not dangerous, are liable by the concussion to cause injury to the neighbouring apparatus. An arrangement upon the principle of Gay-Lussac's washing-bottle (fig. 114), may be employed with much advantage to keep the water or saline solution of the bath at a constant level.

Fig. 163.

Fig. 164.

Some chemists do not quite immerse the tube in the fluid, but allow the upper portion to project, so that a species of cohobation is continually going on; this is, however, not so rapid a method as by entirely immersing the tube.

No general rule can be given as to the time of exposure, it

varying from a quarter of an hour or less to a fortnight or even three weeks. Where ammonia is employed, with the intention of removing its replaceable hydrogen, and substituting some hydrocarbon for it, the volatile alkali must invariably be employed in an alcoholic instead of an aqueous solution, as with the latter the reaction is so sluggish, that after a week's exposure, and sometimes even much longer, less effect is produced than in an hour or two with the alcoholic ammonia. This arises from the reagents being soluble in alcohol but not in aqueous ammonia.

305. It is not always easy to know when the reaction is completed, without opening the tube and examining the contents; but a shrewd guess may frequently be given from the appearance of the fluid when cold. If, for instance, the ingredients when introduced are entirely fluid, and crystals appear on cooling the liquid, after an exposure of moderate duration, it is advisable to return the tube for a short time to the bath, and observe if the quantity appears to be increased on a second inspection; if this happens, the reaction may again be allowed to go on, until no further effect is produced.

306. It is better to permit the tube to cool completely before removing the rag, and to avoid incautious handling, or even a slight blow, especially when hot, as the tube sometimes flies to pieces in the operator's hands, and great danger is incurred of receiving severe injury.

For the same reason, when the tube is to be opened, the rag is only to be removed from that part of the tube where the file-mark is to be made; and even then, where gases are evolved, great care is to be taken that the end does not fly off with violence. Where the gases produced are to be examined or analysed, the point is to be broken off under mercury, a gas-jar full of the same metal being immediately over it, so that the gas may pass freely into it.

307. Where the iodides of the alcohol radicals are made to react in excess on alcoholic ammonia, it is usual, when the process is concluded, to distil off the former upon a water-bath

in a retort, as it may, with the addition of a little more, be used again for the same purpose.

308. As the pressure-tubes are generally rather troublesome to clean, it is better to preserve them if the same operation is to be repeated, a label being attached to assist identification.

309. It is generally unnecessary to use new tubes, unless required very strong, as, with the aid of the gas-blowpipe, fig. 48, they may be constructed with facility from the necks of broken retorts, which should always be preserved for this and other purposes. Other uses of broken retort-necks, &c. will be pointed out in the proper places.

It must be borne in mind that the tubes should always be of pretty considerable thickness, and that test-tubes from their thinness are totally unfitted for experiments of this kind. Mr. Squire, in his investigation of caprylamine, made use with advantage of soda-water bottles, well corked and wired down; what are termed carrara-water bottles, from their shape, would be more generally convenient, and probably resist a greater pressure.