

A Convenient Method of Distillation of the Alkali Metals. By R. J. CLARK, Lecturer in Physics and Carnegie Teaching Fellow in the University of Edinburgh. (Communicated by Prof. Sir E. RUTHERFORD.)

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The alkali metals, sodium, potassium and rubidium can be distilled easily in a good vacuum and obtained reasonably free from occluded gas in the following way. As potassium is now used for the absorption of mercury vapour and many experiments are being done on the others, an account of a convenient way of preparing pure specimens may be of some service to experimenters.

A still is first made like that shown in Fig. 1, which will serve for the preparation of trebly distilled specimens. The size shown will do for the distillation of about six or seven grams. The constrictions where the various sealings are to be done should be of only moderately thick walls, and the bore should be about 1.5 to 2 mm. The connections are on one side only for convenience in washing. When the still is made it is thoroughly cleaned and dried, and set vertically in a clamp (on the tube *S* by preference) and connected through a mercury-vapour trap to either a Gaede rotatory mercury-pump or to a small diffusion pump.

A sufficient quantity of the alkali metal is washed in some dry ether containing 4 or 5 per cent. of alcohol, and all the oily scale is knocked off it. After two or three washings when it is bright and clean beneath the ether, it is taken out, wiped off with filter paper and dropped into the still. A *small* wad of glass wool is next put in and the still is sealed off at *A* and exhausted.

As soon as the exhaustion is well under way all the still except *B* is heated with a Bunsen burner (non-luminous flame) and then allowed to cool. *B* is next heated *very gently* when the metal will melt, and breaking through its skin of oxide, run into the bulb *C*. If this heating is properly done the oxide will not fuse and it will be easy to seal *B* off. The residue left behind in *B* will be contaminated with oil, and it is important that none of it should get into *C*. When the metal melts a lot of gas comes off and the pump should be watched to see that it does not choke.

A small electrically heated oven is next inverted over *C*, and the metal distilled from one bulb to the next, the operator sealing off the bulbs in turn as they empty. The open end of the oven should come a little below the top of the next lower bulb, in order to keep the connecting tube warm. Finally, the metal may be run into an ampoule which is to be sealed off, or it may be run directly into the apparatus in which it is to be used.

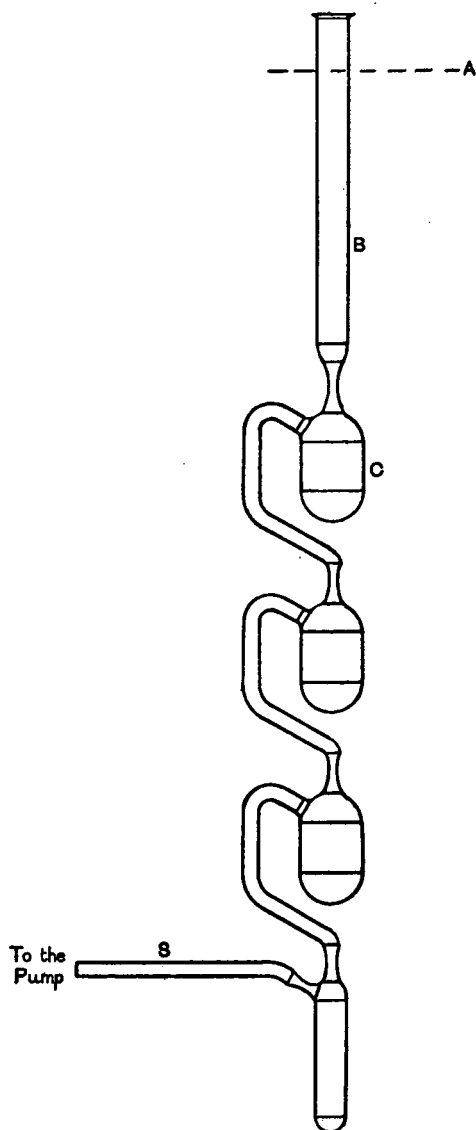


Fig. 1. (Scale $\frac{1}{3}$ natural size)

To distil sodium it is best to use either Pyrex (Jobling) or Monax (Moncrieff) or similar glasses; the temperature of the oven should be about 500 degrees centigrade. Potassium and rubidium can be distilled in soda-glass and a temperature of 400 degrees centigrade is ample for potassium and 350 for rubidium.

A suitable oven can be made from a piece of 20 gauge sheet iron pipe, 3 inches in diameter and 6 inches long. Over one layer of asbestos cloth a single winding of nichrome wire about number 22 s.w.g. is put on (the resistance will be about 25 ohms); this winding is covered with about three-quarter inch thickness of asbestos, and the whole is slipped inside a piece of 5 inch sheet iron pipe 7 inches long. One end is closed with three-quarter inch asbestos and a sheet iron disc on which the binding screws are mounted. This oven can be connected directly to the 110 volt mains. The temperature is regulated by a series rheostat and in practice the rate of distillation is a good guide to the proper temperature. Each distillation of 5 grams should take about 10 to 15 minutes.

This method has been used also to distil mercuric-chloride, mercurous-chloride, zinc bromide, arsenic trioxide and some similar substances in order to free them from occluded gases and non-volatile impurities. Ordinary soda-glass is suitable for all these substances, and a person skilled in this sort of manipulation will find it the most convenient, but any one else had better use a harder glass, as the chance of cracking a tube in sealing it, is then very much less.
