

sulfate and sodium sulfite to be the more desirable ones for analyses by absorption. Of these three, ferrous chloride, which does not appear to have been used previously for analytical purposes, is the most satisfactory.

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NOTES

Vacuum Oven.—For drying in a vacuum at elevated temperature and for the absorption of moisture, an efficient apparatus (the Abderhalden vacuum drying tube) is on the market. This, however, allows the drying of only small samples of material, as used for combustion, etc. The Abderhalden apparatus cannot be appreciably enlarged, as great difficulty would be encountered in opening the tapered ground joint of the larger size apparatus after the drying in vacuum had been finished. This difficulty has been met by the substitution of a flange ground to fit a tubulated desiccator top as shown in Fig. 1. The apparatus is of Pyrex, is inexpensive, and maintains its vacuum well.

The tube is allowed to project 15 cm. outside the oven, thereby preventing the heat of the oven from softening the lubricant used

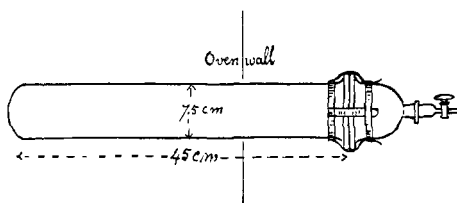


Fig. 1.—Vacuum drying tube.

upon the flange as a seal, and also providing a cooler section in which the absorbent (usually sodium hydroxide in copper trays) is placed.

To prevent the cover from falling when the vacuum is released, a copper band or hoop is placed around the tube next the flange. To this are soldered four copper strips at equal intervals. These are folded over the joint and their free ends are held in place by another hoop, which is slipped over the dome of the cover. The apparatus is evacuated with an oil pump and closed by a stopcock ground into the tubulature of the cover. Two such tubes may conveniently be placed side by side in the ordinary oven. The cover is removed easily by a turning motion, especially when the oven is still warm, not necessitating, as a rule, the use of a safety razor blade as applied to sticking desiccator covers.

It would seem possible to construct an efficient apparatus of larger size, either of Pyrex or copper. If copper is used, however, it may be necessary to cool the flange by means of a water jacket or spiral tube in order to maintain the seal. Concentric grooves in the rims might also prove helpful.

Thanks are due to the Corning Glass Works for the construction of the special glass apparatus and to A. G. Sterling of the Bureau of Chemistry for making the metal parts.

Thermoregulator.—The lower part of the regulator is the ordinary

reservoir filled with mercury or with toluene and mercury. The upper part is shown in Fig. 2. The apparatus is filled by introducing mercury at A until it reaches the level of Plug C. Plug C is inserted and the

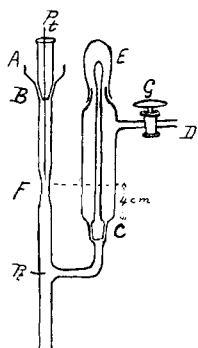


Fig 2.—Thermoregulator.

filling continued until the mercury is slightly above the seat of Plug B, which is then inserted. No air should be left entrapped between the plugs. Mercury should be present above the two plugs when closed to serve as a seal. This makes unnecessary the use of any lubricant on the plugs. A water pump is connected at D. The thermoregulator is heated to the required temperature, vacuum is applied, and the stem of the plug under the rubber cap E is lifted, without removing the rubber cap, until the platinum wire¹ just makes contact with the mercury surface in the constriction at F and the pump is disconnected. The regulator is then ready for use. The writer has most frequently used a stopcock instead of the plug C, which is more convenient. The lubricant at the stopcock does not seem to affect the efficiency of the apparatus.

The advantages of the thermoregulator are a contact in vacuum between wire and mercury and ease of filling and cleaning.

Thanks are due to W. B. Symonds for making the apparatus according to sketch.

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Sodium Hyponitrite.—Sodium hyponitrite has been prepared more easily, from more readily available materials, and with a slightly better yield than previously reported by Jones and the author.¹

Three and twenty-six hundredths g. of hydroxylammonium chloride was dissolved in the least possible amount of absolute ethanol. After cooling to -5° , slightly more than the calculated amount of sodium dissolved in the least amount of absolute ethanol was added. The sodium chloride was collected on a Büchner funnel and washed several times with absolute ethanol. To the filtrate was added 110 cc. of absolute ether and 3.24 g. of sodium dissolved in the least amount of absolute ethanol. The solution was cooled to -5° and twice the calculated amount of either

¹ Attention may also be drawn to the article by Beaver and Beaver, who have devised an efficient method for preventing sparking [*Ind. Eng. Chem.*, **15**, 359 (1923)].

¹ Jones and Scott, *THIS JOURNAL*, **46**, 2175 (1924).