



mixture from one container to another, thus incurring some mechanical loss and inconvenience.

A modified Craig tube has been reported (2) in which the crystals are retained in the recrystallizing compartment, during and after separation from the mother liquor, by a simple inverting technique. The tube described in this paper accomplishes the same purpose without the necessity for inverting prior to centrifugation. It may serve as a receptacle for successive recrystallizations without intermediate removal of the solute. This has particular value in the preparation of analytically pure material when only small amounts are available.

The details of the tube are given in Figure 1.

A piece of 14-mm. tubing, A, 5 to 6 cm. in length, was sealed to a 1.5- to 2.0-cm. piece of 2-mm. capillary tubing, B, the proximal end of which had been constricted, C, to an inside diameter of 0.1 to 0.2 mm. as estimated by means of a close-fitting stainless steel wire. The length of this constricted region was kept to a minimum to avoid plugging during use and particular care was taken to avoid forming an annular trough at the seal. A suitable length of $\frac{1}{8}$ -inch Teflon (trade-name of E. I. du Pont de Nemours & Co., for polytetrafluorethylene) rod was turned on a lathe to give a 2-mm. rod, F, the pointed end of which would fit snugly into the capillary and thus stopper the tube. The beveled inner wall, D, of the constricted capillary was ground with fine grinding powder and a suitable glass rod to provide a rough surface for holding the plug in place. For use, the tube was suspended in a 15-ml. conical centrifuge tube with a rubber washer, G, as a cushion. In this manner the mother liquor was collected in the bottom of the centrifuge tube. A cork, covered with aluminum foil, served to allow the preparation of lint-free crystals.

For recrystallization of a solid, the tube with plug in place is suspended inside the centrifuge tube. The solution is introduced directly from a filter and may then be concentrated, if necessary, by heating the naked tube (Teflon plug still in place) in a steam bath. By careful manipulation this process may be carried out without a stirring rod, owing to the peculiarity of the inner surface which serves as a built-in boiling tube. The recrystallization tube is then replaced in the centrifuge tube and cooled for recrystallization, and after removal of the plug is spun in the centrifuge to separate the two phases. This occurs rapidly and without evaporation of the mother liquor from the surface of the crystalline material. The operation may then be repeated for further purification by simply replacing the Teflon plug, adding fresh solvent, and repeating the cycle in the same tube.

Washing of the solid is unnecessary, as practically no mother liquor adheres to the crystals (4), and a more efficient purification is thus possible. In addition, recrystallizations may be carried out in solvents in which the solute is very soluble, since small volumes of solvent are handled readily (1, 4).

The tube is adaptable for use with most crystalline solids, if the formation of fine precipitates is avoided. The size described was found to be optimal; a larger model, designed for use with a 50-ml. conical tube, developed critical unbalance in the centrifuge as filtration progressed. This recrystallization device is simple in construction, can be made from readily available materials, and can be used with equipment already on hand in the laboratory.

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LITERATURE CITED

- (1) Craig, L. C., *IND. ENG. CHEM., ANAL. ED.*, **12**, 773 (1940).
- (2) Craig, L. C., and Post, O. W., *Ibid.*, **16**, 413 (1944).
- (3) Skau, E. L., *J. Phys. Chem.*, **33**, 951 (1929).
- (4) Weinsberger, A., "Technique of Organic Chemistry," p. 464, New York, Interscience Publishers, 1950.

Improved Recrystallization Tube. Charles E. Blades¹ and Wolfgang Schöniger², University of Wisconsin, Madison, Wis.

CONICAL centrifuge tubes have long been used for the recrystallization of small quantities of material, the mother liquors being removed by decantation or by means of a fine pipet. In order to improve the method for removing the surface layer of liquid from the crystallized material, Skau (3) and Craig (1) have designed filter tubes in which this supernatant liquid is separated by centrifugation through a filter septum. These tubes have found considerable use, but require a transfer of the crystallized

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