LETTER TO THE EDITOR

Centrifugal Filter

Sir: A metal modification of the centrifugal filteration tube (1) for preparing highly pure compounds by solvent crystallization has been designed for use with a No. 2 International Centrifuge. It has a higher capacity, and, since the purified crystals can be collected without exposure to the atmosphere, it has a wider range of applications.

The device (Fig. 1) consists of threaded male and female segments A and B, strainer C, and splash guard D. When assembled, these elements form an airtight closed container of 0.32 cm wall thickness and 16.3 cm overall length. Segments A and B, fabricated from 2 in. standard stainless steel pipe, were threaded with sharp V threads, 20 to the inch. Before threading, the tube forming the female part was swaged by the use of tube rollers to accommodate the male part. The end plates were of U.S. standard 12-gage sheet stainless steel, machined to fit with a 45° bevel. After welding, the entire tube was machined inside and out, and stainless steel posts E were inserted.

U.S. standard 24-gage stainless steel was used to make the strainer and the die-formed splash guard. The strainer, perforated with 0.8 mm holes drilled 4 mm between centers, can be attached to segment A by engaging spring hooks F with posts E. The splash guard has a funnel-shaped surface leading to a hole in the center.

The splash guard serves two purposes. It prevents rewetting and contamination of the crystals by splashes of mother liquor from the almost inevitable jostling of the container during deceleration or during removal from the centrifuge. It also prevents the mother liquor from getting into the threaded union between the male and female segments and causing sticking or "freezing" of the joint. The two segments are supplied with flat surfaces, G, that are adapted to fit wrenches to tighten or loosen the joint.

Procedure

The compound to be purified and the appropriate amount of the solvent are heated in segment A until solution is complete. Any solids that may have formed on the walls of the container at the solution surface because of solvent evaporation are washed down and dissolved by addition of a small amount of solvent down the side of the container. If necessary, the underside of the strainer is fitted with a disk of filter paper, slit so as to fit down around the spring clips. The strainer and splash guard are then placed on top of segment A. Any change in concentration due to evaporation is then prevented by completing the assembly and tightening the joint.

The whole system is cooled very slowly, preferably in an air bath, and brought to equilibrium at the temperature required to give the desired proportion of crystals and mother liquor (as previously determined in a glass tube). The assembly is then inverted, thermally lagged with insulating material if necessary, placed in a properly counterbalanced centrifuge cup, and centrifuged for at least 20 min. Most of the mother liquor is thus removed from the crystals at the specified equilibrium temperature before any substantial temperature change can take place. All of the crystals are retained within segment A by strainer C, and the mother liquor is collected in segment B. The container is then carefully lifted from the centrifuge and allowed to come to equilibrium at room temperature.

Disassembly can be accomplished without spilling the crystals and with virtually no exposure to the atmosphere by quickly replacing segment B with a clean segment B or with an appropriate stopper and then turning the assembly right side up. A higher degree of purity can be attained by repeating the crystallization process.

The improved container makes it possible to use solvent crystallization to purify compounds with melting points considerably below 0 C. The procedure is the same as for solid compounds, but the filtration temperature may necessarily be far below zero. Since the crystals melt below room temperature and the centrifuge is at room temperature, the time interval between starting the centrifuge and collecting the sample must be as short as possible. Thermal lagging should be used around the container in the centrifuge cup, and disassembly must take place immediately after removal from the centrifuge. The pure products can be collected without exposure to the atmosphere by quickly removing segment B, in a moisture-free atmosphere if desired, and substituting a clean segment B in which the pure liquid product can be collected by centrifuging at room temperature.

Another important feature of these containers is that the whole purification procedure can be carried out conveniently in an inert atmosphere, i.e., by performing all

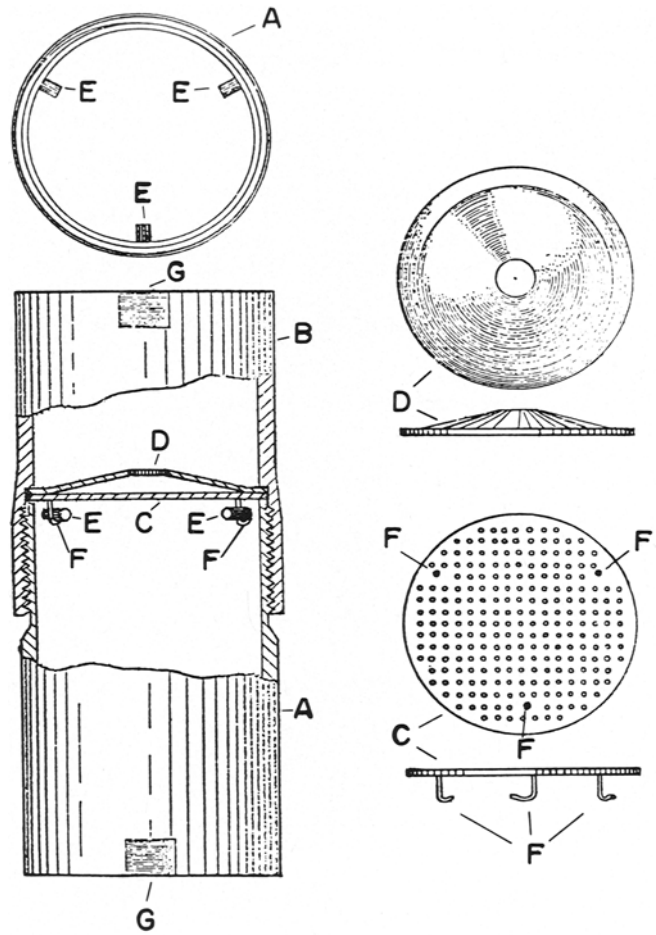

FIG. 1. Centrifugal filter: (A) crystallization chamber, (B) receiver, (C) strainer, (D) splash guard, (E) posts, (F) spring hooks, (G) flat surfaces.

transfers of materials, preparation of the solution, disassembly, and collection of the pure crystals, in a dry-nitrogen glove box. These containers will be very useful, therefore, in preparing pure crystals of air-sensitive compounds that have to be synthesized and handled in an inert atmosphere. Isolation of the crystals from the crude reaction mixture and their purification by successive solvent crystallizations could be accomplished without any exposure to the atmosphere.

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REFERENCE

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