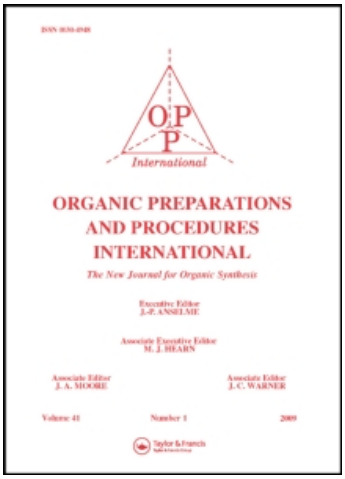

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A SOLVENT PAIR RECRYSTALLIZATION APPARATUS FOR GROWING SINGLE CRYSTALS

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A SOLVENT PAIR RECRYSTALLIZATION APPARATUS FOR GROWING SINGLE CRYSTALS

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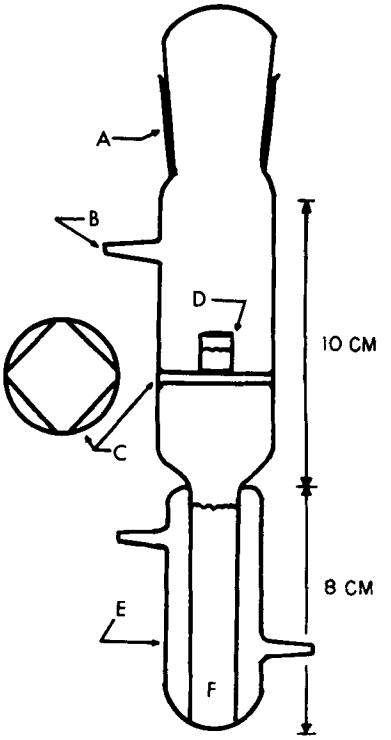
One of the more inherently difficult tasks in the laboratory is the growing of single crystals of organic compounds which are of high quality (e.g., suitable for an x-ray diffraction study). A basic difficulty in one of the most often used techniques, namely, solvent-pair crystallization, is controlling the rate in which the insoluble "solvent" is added to the saturated solution. If large, nicely formed single crystals are desired, this must be done at a slow, constant rate. The apparatus described herein has thus far been 100% effective in producing excellent single crystals for our x-ray structure studies in those cases in which our previous efforts had failed.

The process involves an apparatus (Figure 1) using a closed chamber refluxing of the insoluble "solvent" F into the saturated solution D. The vapor pressure of F must be higher (or easily increased by circulating a warming liquid, such as water, through the jacket E) than D. Solvent pairs (soluble, insoluble) which we have found effective with water as the warming liquid include; acetonitrile-ether, chloroform-ether, ethanol-ethyl acetate, and hexane-ethyl acetate. The apparatus, as illustrated is



designed for milligram quantities and requires 12-24 hours for crystallization to occur. There is no apparent reason why the apparatus cannot be scaled up to gram quantities.

A is a pyrex 45/50 teflon coated joint with the inner section sealed immediately above the joint. B is an outlet for a vacuum pump to remove excess solvent. Under the usual operating conditions, B is sealed off with a small piece of rubber tubing and a pinch clamp. The square sample support platform, C, allows the vapor to pass from the lower into the upper chamber. The saturated solution, D, initially fills about 1/3 of a small vial (approximately 1/2" in diameter and 1" tall). As diffusion progresses, D will gradually fill and crystals of the desired compound will form in the bottom of the sample container, D. Finally, E is a jacket through which the warming liquid can be circulated to increase the

volatility of the insoluble "solvent". Hot tap water at the normal boiling point of F (approximately 55° C) has proven effective as the warming liquid in the previously mentioned solvent-pairs.

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