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AIR FORCE CAMBRIDGE RESEARCH LABORATORIES, OFFICE OF AEROSPACE RESEARCH, UNITED STATES AIR FORCE, L.G. Hanscom Field, Mass.
**Improved Closed-System Evaporation Crystallizer**

**JOHN L. SAMPSON AND MARY A. DIPIETRO**

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An improved closed-system evaporation crystallizer employing a concentration gradient for growing single crystals has been developed and tested by growing crystals of several materials from solution. The device is based on a Soviet design, the baffle system of which permits some of the unsaturated liquid to bypass the nutrient on the way to the growing zone. The present design has its baffles differently arranged to enhance the transport of the material from the nutrient region to the crystallizing region.

The outer container b of the crystallizer (Fig. 1), which has a tight-fitting dome-shaped top a, is placed in a water bath up to approximately the level of the liquid inside to maintain a temperature above ambient. Solvent evaporates from the surface of the liquid within the crystallizer, condenses on the dome and upper wall of the outer container, and runs down into outer chamber c. This chamber is separated from chamber e by cylinder d, whose top extends above the liquid level, but whose bottom has openings to allow free communication between chambers c and e. Nutrient material h is placed at the bottom of either or both of these chambers. As solvent accumulates in chamber c, hydrostatic pressure causes flow past the nutrient material, some of which dissolves to cause a higher concentration in chamber e than in chamber c. Cylinder f, closed at the bottom and with its top below the liquid level, allows liquid to pass into inner chamber g while keeping solid nutrient out. A seed k consisting of a small single crystal is suspended in chamber g at approximately the top of cylinder f. The liquid in the vicinity of the seed is agitated by a magnetic stirring rod i, externally driven.

Although chambers c and e communicate, the more nearly saturated liquid in e is usually denser than the relatively unsaturated liquid in c, and the liquid level in c is often noticeably higher. In the case of sodium chloride a difference of \( \frac{1}{2} \) in. in a 6-in. height (chamber c) was noted. This is in close agreement with the height difference expected on the basis of pure water in c and saturated solution of specific gravity 1.2 in e, and indicates that the rate of flow of solvent is great enough to overcome diffusion effects, which would tend to equalize the concentration throughout the crystallizer.

Several models of this device have been developed, differing in dimensions, materials, and details of construction. One type uses a hot plate and insulation jacket rather than a water bath, and another type employs direct stirring in lieu of the externally driven magnet. Although laboratory-scale models of 4 to 6 in. o.d. have been used throughout these experiments, a larger apparatus of 12-in. diameter is now undergoing tests.

Thus far the following crystals have been grown from aqueous solution: aluminum ammonium sulfate, nickel ammonium sulfate, sodium silicofluoride, barium nitrate, barium silicofluoride, and silver sulfate. Small crystals of sodium chloride have also been grown from an undoped aqueous solution and from a methanol solution. Seeds of several of these materials have been grown from the stock chemicals by dropping a few grains of powder into the saturated solution in chamber g.

![Diagram of the crystallizer.](image-url)

The closed system, in which the solvent is not lost but recirculates, allows the crystallizer to be operated for long periods with a minimum of attention. The device, therefore, shows promise for growing crystals of materials of low solubility. No studies have been made as yet of the purity or dislocation density of the crystals grown in this device. Research is continuing on optimum temperature and stirring rate.

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