

The GROWING of ROCHELLE SALT CRYSTALS *for* RADIO EXPERIMENTS

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THIS project was selected on account of personal interest in the radio field and the desire to experience the satisfaction of building some of one's own equipment. The intention was to construct a microphone by utilizing the piezo-electric properties of rochelle salt crystals. Work was started in December, 1935, and the study continued through several months' spare time. Although the project was not entirely completed, it is reported here in the belief that the results may be of use to others.

The problem resolved itself into two parts: first, the production of satisfactory rochelle salt crystals of suitable size, shape, and other characteristics; and second, the selection, removal, grinding, and mounting of certain sections of these crystals. This report deals principally with the first part; some references are given for the second.

A partial search of the literature was made. Nicholson¹ mentions crystals of various compounds (both in-

organic and organic) that exhibit piezo-electric properties; he gives directions for producing rochelle salt seed crystals, then the larger ones, then desiccating with alcohol to increase strength and piezo-electric properties, finally mounting and investigating the effects of compression and torque. The large crystals are obtained by allowing the temperature of a seeded warm solution to fall naturally to that of the surroundings, and are rapidly formed. Some interesting experiments are outlined.

Another reference² states that above 40° a saturated solution of potassium sodium tartrate deposits crystals of rochelle salt and sodium tartrate, while above 55° a mixture of potassium and sodium tartrates appears.

Ishimoto³ employed solutions saturated at 30° and 32°, and produced 6- to 7-cm. crystals in two or more weeks by cooling at the rate of 0.2° to 0.4° per day

² THORPE, "Dictionary of Applied Chemistry," Longmans, Green and Co., New York City, Vol. VI, 689 (1927).

³ ISHIMOTO, *Proc. Phys. Math. Soc. Japan*, [3], 2, 81 (1920) (through *Chem. Abst.*, 14, 2746 (1920)).

¹ NICHOLSON, *Electrical Experimenter*, 7, 762-4 (Dec., 1919).

using a toluene thermostat. He sealed the solutions almost airtight to prevent appreciable evaporation. Other references having less direct bearing on the problem are omitted from this report.

EXPERIMENTAL WORK

Rochelle salt of U.S.P. quality, from two sources, was carefully recrystallized and used for these experiments.

The two common procedures of slowly lowering the temperature, and slowly evaporating the solvent were considered. Neither a constant-temperature bath nor a thoroughly insulated room was available. Preliminary tests indicated the extreme solubility of potassium sodium tartrate in water, and difficulty in securing stable conditions for crystallization. It was evident that some kind of control was necessary to avoid supersaturation and subsequent rapid growth of many small crystals. Solvents made by mixing water with one of several organic liquids such as glycerin, glacial acetic acid, and acetone gave unpromising results of various types.

Nicholson's method of adjusting the specific gravity of the solution to 1.33 at 50°, followed by seeding at 38° over mercury and allowing to cool to the surrounding temperature, gave coarse-appearing, rather symmetrical crystals. Some of these cracked easily and did not appear promising for this study, although perhaps excellent for some purposes. It seemed that to produce slowly-grown, clear crystals the temperature must fall very slowly.

Results with both the seed crystals and the large ones tended to vary greatly. It was found that by adding just enough mixed alkali (a solution containing 4.5 per cent. KOH and 3.5 per cent. NaOH) to make the rochelle salt solution turn red litmus paper blue, satisfactory and consistent crystallization was obtained. No more accurate method for adjustment of hydrogen-ion concentration was at hand. No attempt was made to ascertain whether acidic material was present in either of the U.S.P. materials or in the recrystallized products. These crystals so formed gave solutions apparently neutral to litmus paper. Five times the minimum amount of alkali produced no change in the stable crystallization conditions.

The procedure finally developed made use principally of slow cooling, with a little evaporation of solvent, to produce both seed and large crystals. The slow cooling was controlled by having the container (beaker) in a calorimeter in a closed wooden cabinet or in an insulated crystallizing box, in a closed and somewhat insulated photographic darkroom located in a concrete building. A maximum-minimum thermometer beside the calorimeters showed that temperature variations were small. Factors seeming to influence start and growth of crystals included specific gravity of solution, room temperature, rate of cooling, rate of evaporation (which in turn depends partly upon coverage, ratio of surface to volume, and relative humidity of air in room), and dust present in the air.

SEED CRYSTALS

The best seed crystals were grown by making a concentrated solution of the recrystallized salt, filtering hot, taking about 50 ml. in a 250-ml. beaker in a calorimeter, adding one or two drops of the KOH-NaOH solution, covering with a watch glass supported on two glass triangles, allowing to stand at room temperature (20°) until first signs of crystals appeared, reducing evaporation rate by removing first one of the triangles, then the second, finally inverting the watchglass, and removing the crystals at suitable stages by means of a horn spoon (glass rods seemed to start new crystals by vibration on beaker).

LARGE CRYSTALS FROM SEEDS

The large crystals grown from seeds were produced best by taking some pure mercury in a clean 250-ml. beaker, adding 200 ml. of a filtered concentrated solution of the recrystallized salt, then adding four drops of the KOH-NaOH solution, warming and adjusting with distilled water to a specific gravity of 1.280 at

50°, covering with a watchglass, and allowing to air-cool on an asbestos mat to 33°. (It was necessary to transfer the rochelle salt solution to a tall, narrow cylinder in order to use an accurate hydrometer and adjust the specific gravity at 50°, but permanent crystallization did not start if the beaker was promptly covered.) The covered beaker was then transferred to the calorimeter, the thermometer left in, and when the temperature had dropped to the 28° to 25° range a selected seed crystal was introduced, the thermometer carefully withdrawn, and the experiment set aside in the cabinet or crystallizing box. A large seed crystal was used with the 28° temperature and a small one (one-sixteenth inch) with the 25°. The experiment was then observed, and reworked if the seed dissolved or if new ones formed. After forty-eight to seventy-two hours a large, perfectly clear crystal, unsymmetrical from end view, was ready in each beaker.

LARGE SELF-SEEDED CRYSTALS

In some crystals produced by the seeding method, evidences of stress were apparent and a few cracked with little encouragement. To avoid this, "spontaneous" or "self-seeded" crystals were grown slowly as follows: 200 ml. of the filtered concentrated solution was introduced on top of the mercury in the 250-ml. beaker, four drops of the KOH-NaOH solution added, the mixture covered with a watch glass, warmed to 55° to insure absence of crystals, the specific gravity adjusted to 1.290 at 50°, and the covered beaker containing the mixture placed in the calorimeter on the table, leaving the thermometer projecting out from the beaker lip. When the temperature was down to 40° the watch glass was removed until first signs of crystals appeared, and then replaced. The calorimeter with the covered beaker was next placed in the cabinet or in the crystallizing box (room temperature was 20°, relative humidity sixty per cent.), and after standing sixteen to seventy-two hours large crystals were ready to be removed and blotted. In some cases successive crops were removed by this procedure; the specific gravity dropped about 0.03 when a crystal or crop had separated.

From observations it appeared that with these procedures a general adaptation over ordinary conditions would be: for each 1° increase in room temperature (20° standard) and each 50 ml. decrease in solution volume (200 ml. standard), the specific gravity at 50° should be increased by 0.004 and the seeding temperature increased by 1°. Larger crystals might have been obtained by starting with larger volumes in larger calorimeters, and further experiments would have been in this direction.

"Dessication" of these large crystals by means of ethyl alcohol, acetone and methyl alcohol rendered them whitish on the exterior but clear and tougher on the inside. Some descriptions of crystal types, methods of cutting sections with reference to the axes, treatment, and mounting in microphones are covered in various references.⁴⁻⁸

SUMMARY

Methods are outlined for producing both rapidly-grown and slowly-grown rochelle salt crystals. The crystals of the latter type were mostly from 3 to 4.5 cm. in length, and those kept more than two years show no change. By controlling the speed of growth it was found possible to secure intermediate types of these crystals.

These procedures offer a fascinating opportunity for studying the influence of growth rate on shape and clearness of crystals. References are also given so that one may investigate the piezo-electric properties of rochelle salt and other crystals.

⁴ SCOTT, C. B., *Radio News*, **14**, 214 (Oct., 1932).

⁵ SAXL, I. R., *ibid.*, **15**, 16 (July, 1933).

⁶ ANON., *Popular Science Monthly*, **123**, 57 (Oct., 1933).

⁷ MCGOWN, D. B., *Radio*, **16**, 16 (Sept., 1934).

⁸ ANON., *Popular Science Monthly*, **127**, 56 (Dec., 1935).