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THE GROWTH OF CROCOITE SINGLE CRYSTALS

Ferenc E. Rosztoczy[1] *Stanford Research Institute, Menlo Park, California.*

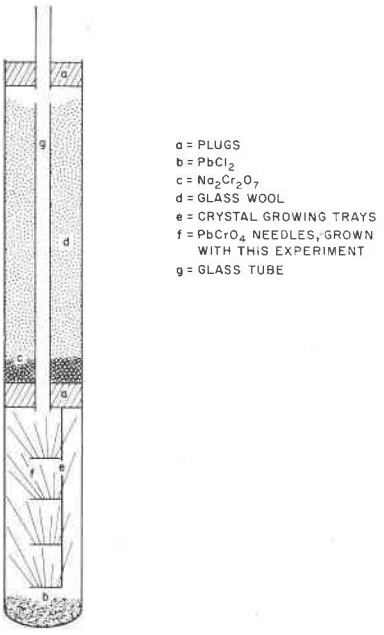
The generally accepted methods of growing single crystals are: a. from saturated solutions, b. from the fused state, c. from the vapor phase, d. the hydrothermal method. Lead chromate was found insoluble in every solvent investigated (in water its solubility product is only 1.8×10^{-14}) with the exception of strongly acidic solutions. It starts to decompose at 100–200° below its melting point where its vapor pressure is still very low. As a result, there is only one work published on the growth of lead chromate single crystals. O'Connor and Buchanan (1952) grew large (5×3 mm) $PbCrO_4$ crystals by slow evaporation, at 80° for 12 weeks, of a 0.05 M solution (2 liters) of $Pb(NO_3)_2$ in 1N HNO_3, to which was added, daily for 6 weeks, 50 ml of 0.05 M $K_2Cr_2O_7$ solution. Further growth, using these crystals as seeds, was never achieved.

In connection with our hydrothermal research program we investigated several possibilities of growing large single crystals of lead chromate.

Hydrothermal method. Solubility measurements were attempted with lead chromate in a platinum reactor up to 450° C. and 20,000 psi. In each case, all the $PbCrO_4$ which dissolved was decomposed.

Chemical reaction in solution. $PbCl_2$ was placed on the bottom of a glass tube, b, in Fig. 1; the upper part of the tube was separated from the lower part by a rubber plug, a, through which passed a glass tube. $Na_2Cr_2O_7$ crystals, c, were placed above the plug and, to slow down the diffusion of the $CrO_4^=$ ions, the upper part of the tube was filled with glass wool, d. The amount of Pb^{++} ions present was limited by the small solu-

[1] Present address: Bell Telephone Laboratories, Murray Hill, N. J.



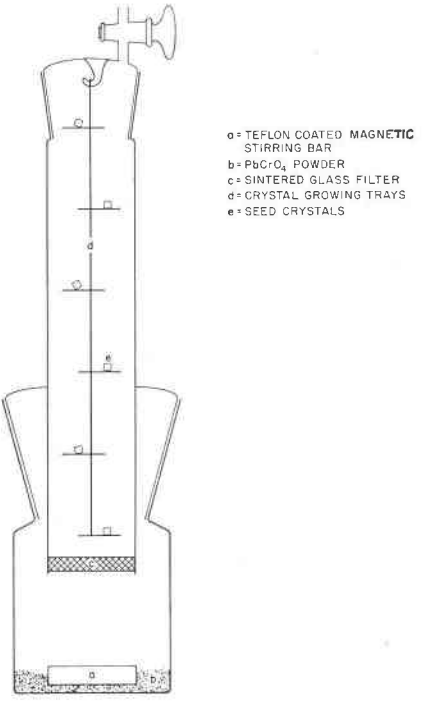


FIG. 1 FIG. 2

FIG. 1. Growing of lead chromate crystals by chemical reaction.

FIG. 2. Acid hydrothermal growing of lead chromate crystals.

bility of $PbCl_2$. The growth of $PbCrO_4$ crystals occurred on the trays, e, in the lower chamber. $PbCrO_4$ needles (30–40 mm long) were grown within a few days. Unfortunately, the needles were extremely thin, their diameters being less than 0.1 mm. Attempts to obtain larger-diameter needles by seeding the solution with $PbCrO_4$ crystals failed.

Fused salts. Every attempt to fuse $PbCrO_4$ (in air, sealed under vacuum, or sealed under O_2 pressure) without decomposition failed.

Acid hydrothermal method. A glass apparatus was built to grow $PbCrO_4$ single crystals from hot, strongly acidic solutions by using a temperature gradient (Fig. 2). The assembly was placed on a Magne-Stir, which stirred it continuously and kept it at a constant temperature. The $PbCrO_4$ powder, b, was placed in the lower, dissolution chamber and strongly agitated by a magnetic stirring bar, a. The crystal growing tower was separated from the lower chamber by a sintered glass filter, c. This filtered out the $PbCrO_4$ powder particles, and also served as a buffer to

separate the two parts of the chamber and set up independent convection currents in the tower, achieving a temperature 15–20° lower there. The seed crystals, e, were placed on pyrex or platinum trays, d. At the beginning of the experiment the assembly was filled with a hot 4N HNO_3 solution saturated with $PbCrO_4$. With 1–3 mm seeds, single crystals $15 \times 8 \times 8$ mm were grown in 1 or 2 weeks.

Crocoite (from Dundas, Tasmania), $PbCrO_4$ crystals grown by O'Connor and Buchanan (reported as orthorhombic), and crystals grown in this laboratory were used as seed crystals; all three proved to be satisfactory. The crystals grown by this method had very well-developed shining faces and were brownish-red in color, quite different from the color of common $PbCrO_4$ crystals.

The crystals grown by the acid hydrothermal method clearly exhibited the highest monoclinic symmetry, one 2-fold axis and a plane of symmetry perpendicular to it.

The available four, seemingly different, types of $PbCrO_4$ crystals (crocoite, $PbCrO_4$ grown by O'Connor and Buchanan, $PbCrO_4$ grown in the laboratory by chemical reaction, and those grown by the acid hydrothermal method) were examined by *x*-ray powder and single crystal methods, including the Weissenberg method. Our studies showed that all the samples consisted only of monoclinic lead chromate crystals. No evidence for the presence of orthorhombic lead chromate could be found. The powder photographs of the different crystals were identical with those reported in the literature for naturally occurring crocoite. Weissenberg diffraction photographs of single crystals indicated monoclinic symmetry, and the unit cell dimensions and space groups are in agreement with those given for crocoite by various other workers. These results indicate that the $PbCrO_4$ crystals grown earlier by O'Connor and Buchanan and reported as orthorhombic might have been actually monoclinic.

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