UNIT CELL MEASUREMENTS OF Pb_3O_4, Pb_2O_3 AND Tl_2SO_4

Sir:

X-Ray measurements and space groups recently determined in these laboratories for Pb_3O_4, Pb_2O_3 and Tl_2SO_4 are listed below. The Pb_3O_4 and Pb_2O_3 represent new structure types.

1. Pb_3O_4. Tetragonal: $a_0 = 8.86$ Å.; $c_0 = 6.66$; density, 9.1; $4.2 \simeq 4$ Pb_3O_4 per unit cell; extinctions, $h0l$ interferences present only when h is even; probable space group, $P\bar{4}b2$ (D_{2d}^7). These crystals were previously described in a paper from this Laboratory[1] as monoclinic. It is now desired to correct this conclusion which was the result of the high distortion of the crystals, produced under the conditions of their formation (high temperature and pressure). Goniometer X-ray patterns, similar to those shown for Pb_2O_3 in Fig. 1, establish the tetragonal nature of the material.

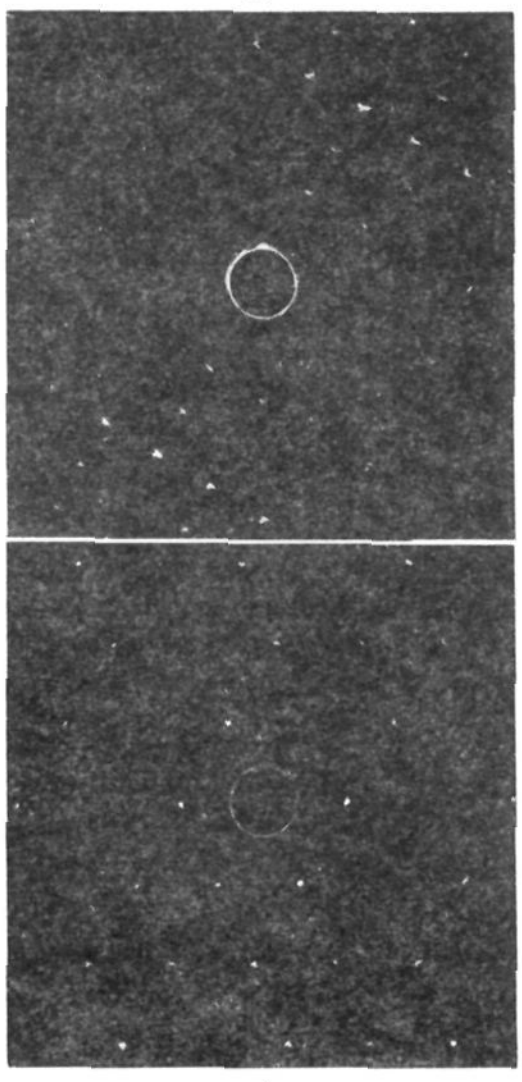

Fig. 1.—Goniometer patterns for Pb_2O_3, $\rho d = K\lambda$. a. $hk0$ interferences ($K = 3.46$, $\lambda = 1.54$ Å.). b. $h3$ interferences ($K = 3.00$, $\lambda = 1.54$ Å.).

(1) G. L. Clark, N. C. Schieltz and T. T. Quirke, THIS JOURNAL, **59**, 2305 (1937)

2. Pb_2O_3. *Monoclinic:* $a_0 = 7.03$ Å.; $b_0 = 5.62$; $c_0 = 3.93$; $\beta = 82°$; density, 9.925; $2.00 \simeq 2$ Pb_2O_3 per unit cell; apparent extinctions, $0k0$ present only when k is even; probable space groups, $P2_1/m$ (C_{2h}^2) or $P2_1$ (C_2^2). Patterns of various apparently untwinned crystals of this material, originally described as triclinic,[1] require a monoclinic crystal symmetry (see Fig. 1).

3. Thallous sulfate, Tl_2SO_4. Orthorhombic: $a_0 = 10.68$ Å.; $b_0 = 6.02$; $c_0 = 7.81$; density, 6.77; $4.08 \simeq 4$ Tl_2SO_4 per unit cell; extinctions, $0kl$ present only when $(k + l)$ is even; $hk0$ present only when h is even; space group, $Pnma$ (V_h^{16}). Patterns are similar to those of the isomorphous K_2SO_4.

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SEPARATION OF STARCH INTO ITS TWO CONSTITUENTS

Sir:

Although numerous attempts have been made to separate the two constituents of starch (α-amylose, amylopectin, erythroamylose and β-amylose, amylose, amyloamylose) in pure form (ultrafiltration, electrodialysis, etc.), there seems to exist no method that would allow the isolation of the two amyloses in sufficiently large quantities and high purity. In searching for such a method it occurred to us that perhaps the preferential adsorption of either constituent on the surface of some suitable material would lead to complete separation. Preliminary experiments carried out on activated carbon, fuller's earth and Brockmann alumina showed that selective adsorption did occur, in that the β-amylose became firmly bound to these materials, whereas the α-amylose remained in solution. Our best results, however, were obtained by the use of cellulose as adsorbent. It is well known that the ancient peoples employed starch for sizing papyrus and paper. Further, it is a matter of common experience that starched linen, even after it has been washed several times in water, retains its ability to give blue coloration with iodine. The preferential adsorption of β-amylose by cotton in considerable amount (1.7%) was recently reported by Samec [*Ber.*, **73A**, 88 (1940)]. We have found that the cotton-β-amylose adsorbate, which is formed instantaneously when a cold 1% corn starch paste