

m-Isopropylbenzoic acid was prepared by the carbonation of *m*-isopropylphenyl magnesium bromide⁸ either with gaseous carbon dioxide at 0°, or, more conveniently, with solid carbon dioxide. Crawford and Stewart prepared the acid by the same method. The pure acid melted at 51–52° and on oxidation with dilute nitric acid at 180° gave isophthalic acid.

The anilides and *p*-bromophenacyl esters were prepared by standard methods.

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A Preliminary X-Ray Investigation of Re_2O_7

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In connexion with work on chromium oxides, dichromates, and polychromates, the author has preliminarily investigated the crystal structure of Re_2O_7 . This compound was first prepared by W. Noddack in 1928 by direct oxidation of Re-metal by oxygen^{1,2}, who in this way obtained it as extremely deliquescent, canary-yellow, "hexagonal" plates and prisms. The compound begins to sublime at 250° C at atmospheric pressure. Vapour density measurements (at 520° C) indicated the existence of Re_2O_7 -molecules in the gas phase.

In this preliminary investigation the oxide was prepared according to the

method given in *Inorganic Syntheses*³, starting from Re-metal of 99.5 % purity (Mackay). After the reaction and subsequent sublimation of the Re_2O_7 , the sample was preserved by sealing off the reaction tube using a torch. All manipulations of the substance had to be carried out in a carefully dried atmosphere.

The single crystal photographs were taken with the crystals sealed up in thin tubes of lithium borate glass, using CrK and CuK radiation. Numerous crystals were tried, all of which, however, consisted of two or more individuals with the *c*-axis (the prism axis) in common. The powder photograph was taken in a 19 cm Bradley camera with CuK radiation using a sample obtained directly from the reaction vessel after drawing out one end of the sealed-off Pyrex tube into a capillary.

The Laue symmetry, derived from Laue and Weissenberg diagrams (*h*0*l*, *h*1*l*) was D_{2h}^5/mmm . The unit cell dimensions obtained from the powder photograph were found to be

$$a = 15.25 \pm 0.1 \text{ \AA}; b = 5.48 \pm 0.02 \text{ \AA};$$

$$c = 12.5 \pm 0.1 \text{ \AA}; V = 1044.6 \text{ \AA}^3.$$

The density of the compound as determined by Biltz and Lehrer⁴ ($d = 6.103$) evidently indicates a unit cell content of 8 (7.92) formula units of Re_2O_7 .

The only reflexions systematically missing were *hk*0 with *k* odd, which suggests the symmetry to be $D_{2h}^5/Pmnc$.

Further studies on the crystal structure are planned.

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