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Short Communications

o- and *m*- *Iso*propylbenzoic Acids

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During the course of work, which will be described in a separate communication, it became necessary to prepare pure samples of the three isomeric *iso*propylbenzoic acids. The *p*-isomer, cumic acid, is well known and adequately characterised, but the methods which have been employed for the preparation of the *o*- and *m*-isomers are not entirely satisfactory, and no convenient derivatives of these acids have been described. Thus, *o*-*iso*propylbenzoic acid has been obtained by the reduction of 3,3-dimethylphthalide with hydrogen iodide and red phosphorus [1], and as a by-product in preparations giving predominantly the *p*-isomer [2]. Likewise, *m*-*iso*propylbenzoic acid has been obtained by the alkylation of benzoic acid with di-*iso*propyl ether using hydrogen fluoride as a catalyst [3]. It has been stated that the acid melts at about 20°, or is an oil [4,5].

When the present work was nearing completion a paper by Crawford and Stewart [6] appeared, in which syntheses of the two acids in question were described. However, as the melting points of the acids we obtained are significantly higher than those recorded by Crawford and Stewart, and, furthermore, two derivatives of each acid were prepared, it seems desirable to record our own findings.

o-*Iso*propylbenzoic acid was prepared by hydrolysis of *o*-*iso*propylbenzonitrile obtained from *o*-cumidine by the method recommended for the preparation of *o*-tolunitrile [7]. The pure acid melted at 63—63.5°, and on oxidation with dilute nitric acid in a sealed tube at 180° gave phthalic acid.

Substance	m. p. * °C	Crystal Form and solvent	Analysis ** Found, % C	H	N	Required, % C	H	N
o-*Iso*propyl-benzoic acid	63—63.5	needles, petrol ether	73.1	7.38	—	73.1	7.37	—
Anilide	137—8	plates, EtOH/H_2O	80.5	7.04	5.89	80.3	7.16	5.85
p-Bromophenacyl ester	54—5	needles, EtOH/H_2O	60.0	4.69	—	59.8	4.74	—
m-*Iso*propyl-benzoic acid	51—2	prisms, petrol ether	73.1	7.32	—	73.1	7.37	—
Anilide	117—8	needles, EtOH/H_2O	79.8	7.28	5.94	80.3	7.16	5.85
p-Bromophenacyl ester	96	plates, EtOH	60.5	4.81	—	59.8	4.74	—

* All melting points uncorrected.
** Analyses by W. Kirsten, Uppsala, and A. D. Campbell, University of Otago.

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*m-Iso*propylbenzoic acid was prepared by the carbonation of *m-iso*propylphenyl magnesium bromide[8] 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 *iso*phthalic 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 Cr*K* and Cu*K* 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 Cu*K* 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 ($h0l$, $h1l$) was D_{2h}/mmm. The unit cell dimensions obtained from the powder photograph were found to be

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

$$c = 12.5 \pm 0.1 \text{ Å}; \; V = 1044._6 \text{ Å}^3.$$

The density of the compound as determined by Biltz and Lehrer[4] ($d = 6.103$) evidently indicates a unit cell content of 8 (7.92) formula units of Re_2O_7.

The only reflexions systematically missing were $hk0$ with k odd, which suggests the symmetry to be $D_{2h}^5/Pmmc$.

Further studies on the crystal structure are planned.

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