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Preliminary Note

Preparative and X-Ray Crystallographic Data on Potassium Dithorium Trisphosphate, $KTh_2(PO_4)_3$

B. Matković, M. Šljukić, and B. Prodić*

Institute »Ruđer Bošković«, Zagreb, Croatia, Yugoslavia

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The purpose of this and subsequent studies of the alkaline, thorium, uranium (IV) or zirconium phosphates is to determine their crystal structures and so deduce the coordination number and polyhedron of the heavy atom. The common formula of these compounds is $M^IM_2^{IV}(PO_4)_3$, where M^I can be lithium, sodium, potassium, rubidium or cesium, and M^{IV} thorium, uranium (IV) or zirconium. The crystallographic data for sodium dithorium and diuranium (IV) trisphosphates have recently[1] been determined. Here the prefix *tri* was used as is quoted in the literature,[2] *e. g.* sodium uranium (IV) triphosphate for $NaU_2(PO_4)_3$. It is better to use the prefix *tris* to avoid confusion with the triphosphate anion $(P_3O_{10})^{5-}$. We shall therefore use the name potassium dithorium trisphosphate for $KTh_2(PO_4)_3$. The crystallographic data for this compound are reported here.

$KTh_2(PO_4)_3$ has previously been described by L. Troost and L. Ouvrard.[3] We obtained the single crystals by heating a mixture of 3.0 g. K_2HPO_4, 1.3 g. ThO_2 and 2.5 g. KCl to a temperature of 1200° C for 24 hours in a platinum crucible. The insoluble residuo, left after boiling in water, was again heated to a temperature of 1200° C with 3.0 g. K_2HPO_4 and 3.0 g. B_2O_3 for a further 24 hours. The obtained crystals are insoluble in all acids. Their chemical composition determined by analysis (ThO_2 = = 67.48, P_2O_5 = 27.28%) corresponds to the formula $KTh_2(PO_4)_3$. An analogous method was used for the preparation of sodium dithorium trisphosphate.[1]

The crystals of $KTh_2(PO_4)_3$ are colourless, transparent monoclinic prisms, space group probably C 2/c—C_{2h}^6 with unit cell dimensions

$$a = 17.55 \text{ Å}$$
$$b = 6.86 \text{ Å}$$
$$c = 8.14 \text{ Å}$$
$$\beta = 101^0\,52'$$
$$D_m = 5.44 \text{ gcm}^{-3}$$
$$D_x = 5.46 \text{ gcm}^{-3}$$
$$Z = 4$$

The unit cell dimensions were obtained from oscillation and Weissenberg photographs using CuK α radiation. The density was determined pycnometrically. The presence of hkl reflection only for $h + k = 2n$ and h0l reflection only for $l = 2n$ indicated the space groups C 2/c or C_c. There are four formula weights per unit cell. Because the piezoelectric effect was not found, the space group is probably C 2/c. The test for its piezoelectric effect was made by the Bergman method as modified by Iitaka.[4] The dimensions of the unit cell are very close to those obtained for sodium dithorium trisphosphate and sodium diuranium (IV) trisphosphate.[1]

* Permanent address: Department of Analytical Chemistry, Faculty of Science, The University, Sarajevo, Bosnia and Herzegovina, Yugoslavia

A complete crystal structure determination of potassium dithorium trisphosphate is now in progress with three-dimensional data. The coordinates of the thorium atom $x = 0.154$, $y = 0.093$, $z = 0.034$ were determined from Patterson projections and improved by a three-dimensional Fourier synhesis.

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IZVOD

Preparativni i kristalografski podaci o kalijevom ditorijevom fosfatu, $KTh_2(PO_4)_3$

B. Matković, M. Šljukić i B. Prodić

Opisana je metoda za dobivanje monokristala kalijevog ditorijevog fosfata i određeni su njegovi kristalografski podaci metodom rendgenske difrakcije.

Kristali kalijevog ditorijevog fosfata su monoklinski, prostorna grupa je vjerojatno C 2/c (C_{2h}^6), sa dimenzijama elementarne ćelije:

$a = 17.55$ Å
$b = 6.86$ Å
$c = 8.14$ Å
$\beta = 101^0\,52'$
$D_m = 5.44$ gcm^{-3}
$D_x = 5.46$ gcm^{-3}
$Z = 4$

Koordinate torijevog atoma određene su iz Pattersonovih projekcija ($x = 0.154$, $y = 0.093$, $z = 0.034$). Rad na strukturi toga spoja je u toku.

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