Short Communications

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 *words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible. Publication will be quicker if the contributions are without illustrations.*

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Crystallographic data for orthorhombic uranium(IV) metaphosphate and plutonium(IV) metaphosphate.*

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Sketchy optical data and a measured density for an orthorhombic uranium(IV) metaphosphate [$U(PO_3)_4$] have been reported by Colani (1907). This phase is apparently isomorphous with thorium metaphosphate [$Th(PO_3)_4$] (Colani, 1907) for which lattice dimensions and number of formula units per cell have been determined by Zachariasen (1954). A triclinic modification of $U(PO_3)_4$ will be described in a subsequent communication. Further work is not contemplated on the substances described below.

Orthorhombic crystals of $U(PO_3)_4$ and isomorphous crystals of plutonium(IV) metaphosphate [$Pu(PO_3)_4$] were grown by B. J. Thamer, G. E. Meadows and B. H. Morrison of this laboratory from solutions of uranium dioxide and of plutonium dioxide, respectively, in metaphosphoric acid. Quantitative chemical analysis by C. H. Ward of this laboratory of a nearly pure sample of the uranium compound gave, by weight, 40·6% uranium and 21·6% phosphorus. In view of the small amount of material available and the extreme difficulty with which it is brought into solution for analysis, these values are considered in satisfactory agreement with those calculated for $U(PO_3)_4$, 42·97% uranium and 22·37% phosphorus. The powder pattern for $Pu(PO_3)_4$ was prepared by the late E. Staritzky of this laboratory.

Crystal morphology

System and class: orthorhombic dipyramidal, *mmm*. For crystals of the uranium salt no piezoelectric effect was detected with a Giebe–Scheibe-type apparatus.

Axial elements: *a*:*b*:*c*.

	$Th(PO_3)_4$	$U(PO_3)_4$	$Pu(PO_3)_4$
Calculated from cell dimensions	0·595:1:0·464	0·599:1:0·461	0·600:1:0·461
Measured	—	0·591:1:0·465	—

Habit: prismatic {011} with minor {010}, terminated by {100} and {120}; also, equally developed {011} and {120}.

Cleavage: {100}.

Interfacial angles (polar):

	$U(PO_3)_4$		
	Calculated from cell dimensions	Measured with two-circle goniometer	Measured on microscope stage
$(011) \wedge (0\bar{1}1)$	49·5°	49·8°	49·8°
$(100) \wedge (120)$	50·1	49·8	50·0

X-ray diffraction data

Diffraction symbol and space group: *mmmPnab*, indicating uniquely space group $Pnab(D_{2h}^{14})$.

	$Th(PO_3)_4$	$U(PO_3)_4$	$Pu(PO_3)_4$
Cell dimensions (Å):*			
a_0	9·04 ± 0·04	8·95 ± 0·01	8·93 ± 0·02
b_0	15·19 ± 0·07	14·94 ± 0·01	14·89 ± 0·03
c_0	7·05 ± 0·03	6·89 ± 0·01	6·87 ± 0·02
Cell volume (Å^3):	969	921	913
Formula units per cell:	4	4	4
Formula weight:	548·04	553·99	554·98
Density (g.cm.$^{-3}$):			
Calculated†	3·76	3·99	4·03
Measured pycnometrically	—	3·95	—
Literature	3·922 (Johnsson, 1889)	3·9 (Colani, 1907)	—

* Dimensions for $Th(PO_3)_4$ originally listed by Zachariasen (1954) as ‘a_1 = 7·04 ± 0·03, a_2 = 15·16 ± 0·07, a_3 = 9·02 ± 0·04’, all in kX.; converted to Å by factor 1 kX. = 1·00202 Å. Dimensions for $U(PO_3)_4$ from calibrated Weissenberg and precession photographs and for $Pu(PO_3)_4$ from powder photograph, using copper radiation: $\lambda\,(K\alpha_{\text{unresolved}})$ = 1·5418, $\lambda\,(K\alpha_1)$ = 1·54050, $\lambda\,(K\alpha_2)$ = 1·54434, $\lambda\,(K\beta_1)$ = 1·39217 Å.

† Weight of unit atomic weight = 1·6602 × 10^{-24} g.

Optical properties

Optic orientation: $X = c$, $Y = b$, $Z = a$.

Extinction: parallel on {011}, symmetrical on {100}.

	$U(PO_3)_4$	$Pu(PO_3)_4$
Refractive indices (5893 Å):		
n_X	1·633 ± 0·002	1·654 ± 0·002
n_Y	1·640 ± 0·002	1·661 ± 0·002
n_Z	1·660 ± 0·002	1·686 ± 0·001
Geometric mean	1·644	1·667
Lorentz–Lorenz refraction (cm.^{3}):		
Measured	50·2	51·2
Calculated*	50·0	—
Optic sign:	Positive	Positive
Optic axial angle (5893 Å):	$2V_Z$ = 52°	58°
Color:		
In bulk	Bright green	Pink
Parallel to *X* and *Y*	Faint greenish tinge	Pinkish tan
Parallel to *Z*	Bright green	Greenish tan

* Calculated from values for UP_2O_7 (Douglass & Staritzky, 1956) and UO_2.

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* Work done under the auspices of the U.S. Atomic Energy Commission.

Table 1. *Partial X-ray powder diffraction patterns*

	$U(PO_3)_4$		$Pu(PO_3)_4$		
hkl	d_c (Å)	d_o (Å)*	d_c (Å)	d_o (Å)*	I/I_1†
011	6·256	6·25	6·24	6·25	75
120	5·735	5·71	5·72	5·71	55
111	5·127	5·12	5·11	5·11	65
200	4·474	4·46	4·46	4·44	20
121	4·408	4·39	4·40	4·37	15
031	4·036	4·02	4·02	4·01	100
220	3·839	3·83	3·83	3·81	25
201	3·752	3·74	3·74	3·73	25
040	3·735		3·72		
131	3·680	3·64	3·67	3·64	65
211	3·640		3·63		
002	3·445	3·44	3·44	3·42	5
221	3·354	3·35	3·34	3·33	< 5
022	3·128	3·12	3·12	3·10	10
231	2·997	2·99	2·99	2·98	35
122	2·953	2·95	2·94	2·94	60
240	2·867	2·855	2·859	2·847	50
320	2·771	2·766	2·764	2·759	25
202	2·730	2·725	2·723	2·712	25
311	2·693	2·689	2·686	2·677	30
212	2·685		2·678		
151	2·621	2·609	2·613	2·603	30
321	2·571	2·562	2·564	2·560	5
222	2·564		2·557		
042	2·532	2·525	2·524	2·518	35
060	2·490	2·478	2·482	2·474	5
142	2·437	2·430	2·429	2·423	10
331	2·399	2·390	2·393	2·382	20
160	2·399		2·391		
232	2·394		2·387		
251	2·338	2·328	2·331	2·320	15
340	2·331		2·325		
013	2·270	2·268	2·263	2·260	15
400	2·238	2·232	2·233	2·223	10
312	2·230		2·224		
341	2·208	2·200	2·202	2·192	60
242	2·204		2·198		
113	2·200		2·194		
322	2·159	2·156	2·153	2·149	25

* Philips 114·6 mm. diameter powder camera, Straumanis film mounting; sample contained in 0·2 mm. diameter glass capillary with walls 0·01 mm. thick.

† Relative peak intensities above background from densitometer measurements of powder photograph of the uranium salt; values for the plutonium salt are not significantly different. Copper radiation with nickel filter; Eastman Kodak Type *A* film for which density was calibrated as a function of exposure time.

Table 2. *Absorption spectrum of* $U(PO_3)_4$

Band maxima in millimicrons, relative intensities (*VS*, *S*, *MS*, *M*, *MW*, *W*, *VW*) and relative band widths (*vw*, *w*, *m*, *n*) as viewed with Zeiss prism microspectrometer ocular

Parallel to *X*			Parallel to *Y*			Parallel to *Z*			In bulk	
680	*MW*	*n*	670	*S*	*n*	670–660	*VS*	*vw*	665	*VS*
669	*S*	*n*	662	*VS*	*m*	635–625	*M*	*vw*	656	*MS*
666	*S*	*n*	649	*S*	*m*	620–610	*M*	*vw*	648	*S*
660	*M*	*n*	638	*MW*	*w*	585	*MW*	*n*	637	*MW*
655	*S*	*m*	593	*MW*	*w*	544	*M*	*vw*	618	*W*
650	*M*	*n*	581	*M*	*n*	480	*MW*	*vw*	599	*VW*
639	*W*	*n*	537	*MS*	*vw*	442	*W*	*m*	587	*VW*
632	*MW*	*n*	493	*VW*	*vw*	439	*MS*	*vw*	542	*M*
598	*W*	*m*	475	*M*	*vw*				477	*MW*
589	*MW*	*m*								
544	*MS*	*w*								
537	*MS*	*w*								
484	*W*	*w*								
470	*MW*	*w*								

Table 3. *Absorption spectrum of* $Pu(PO_3)_4$

Band maxima in millimicrons and relative intensities (*VS*, *S*, *MS*, *M*, *MW*, *W*, *VW*) as viewed with Zeiss prism microspectrometer ocular

Parallel to *Z*			Parallel to *X* and *Y*	
665	*S*	wide	665	*S*
635	*VS*		635	*VS*
572	*MW*		572	*MW*
559	*W*			
552	*W*			
			522	*W*
518	*MW*		516	*MW*
509	*W*		509	*M*
497	*MS*			
			487	*M*
483	*M*			
			473	*VW*
454	*M*	wide	463	*W*
448	*MW*	wide	448	*MS*

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The disordered structure of $NaNO_2$ at 185 °C.*

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The discovery of ferroelectricity in sodium nitrite (Sawada *et al.*, 1958) has prompted renewed interest in the structural details of this compound. The structure has been examined at room temperature by several investigators (Ziegler, 1931; Truter, 1954; Carpenter, 1952, 1955; Kay & Frazer, 1961), and is accurately known. The transformation at about 160 °C., the ferroelectric Curie point, was found originally in an X-ray study by Strijk & MacGillavry (1943, 1946). They attempted a complete

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