

## Phurcalite

BY PAUL PIRET AND JEAN-PAUL DECLERCQ

Laboratoire de Chimie Physique et de Cristallographie, Université de Louvain, 1 place Louis Pasteur, B1348 Louvain-la-Neuve, Belgium

(Received 5 January 1978; accepted 25 January 1978)

**Abstract.**  $\text{Ca}_2[(\text{UO}_2)_3(\text{PO}_4)_2(\text{OH})_2](\text{OH})_2 \cdot 4\text{H}_2\text{O}$  (a mineral from Bergen an der Trieb, Saxony, Federal Republic of Germany), orthorhombic, *Pbca*,  $a = 17.426$  (3),  $b = 16.062$  (3),  $c = 13.592$  (3) Å,  $Z = 8$ . The structure consists of  $[(\text{UO}_2)_3(\text{PO}_4)_2(\text{OH})_2]_n^{2n-}$  layers connected by Ca, OH and  $\text{H}_2\text{O}$ . The U atoms

have seven- and eightfold coordination (bipyramidal); Ca is sevenfold coordinated (irregular).

Table 1. Experimental conditions

Radiation: Mo *K* $\alpha$ ,  $\lambda = 0.71069$  Å  
 $\omega$  scan:  $-0.8^\circ$  to  $+0.8^\circ$  (96s) (background  $2 \times 20$ s)  
 $2\theta_{\text{max}} = 50^\circ$   
 Total number of independent reflexions: 3327  
 Total observed  $|I| \geq 2.5 \sigma(I)$ : 1583  
 Crystal dimensions along **a**, **b**, **c**: 0.05, 0.016, 0.20 mm  
 $\mu(\text{Mo } K\alpha) = 405.2 \text{ cm}^{-1}$

Table 2. Atomic coordinates and thermal parameters  
[ $U = B/(8\pi^2)$ ]

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> (Å <sup>2</sup> )
U(1)	0.3430 (2)	0.2139 (2)	0.6128 (2)	0.0194 (6)
U(2)	0.4514 (2)	0.2175 (2)	0.8688 (2)	0.0191 (6)
U(3)	0.2376 (2)	0.2343 (2)	0.8656 (2)	0.0182 (6)
Ca(4)	0.1469 (11)	0.0461 (12)	0.0312 (13)	0.018 (4)
Ca(5)	0.0906 (11)	0.1103 (12)	0.2895 (14)	0.018 (4)
P(6)	0.1577 (13)	0.2473 (14)	0.6239 (17)	0.016 (3)
P(7)	0.0304 (14)	0.1945 (15)	0.8806 (18)	0.020 (5)
O(8)	0.338 (4)	0.391 (4)	0.097 (4)	0.021 (10)
O(9)	0.346 (5)	0.180 (5)	0.141 (6)	0.049 (19)
O(10)	0.435 (4)	0.117 (4)	0.923 (5)	0.021 (10)
O(11)	0.473 (4)	0.318 (4)	0.829 (5)	0.023 (11)
O(12)	0.236 (4)	0.373 (4)	0.376 (5)	0.029 (15)
O(13)	0.238 (4)	0.344 (4)	0.827 (5)	0.023 (13)
O(14)	0.346 (4)	0.302 (4)	0.272 (5)	0.024 (13)
O(15)	0.198 (4)	0.205 (4)	0.703 (5)	0.029 (15)
O(16)	0.212 (4)	0.272 (4)	0.028 (5)	0.021 (14)
O(17)	0.344 (4)	0.261 (4)	0.954 (4)	0.020 (12)
O(18)	0.475 (4)	0.242 (4)	0.540 (5)	0.022 (12)
O(19)	0.483 (4)	0.171 (4)	0.702 (5)	0.023 (14)
O(20)	0.097 (3)	0.249 (4)	0.360 (4)	0.019 (8)
O(21)	0.083 (4)	0.202 (4)	0.622 (5)	0.033 (15)
O(22)	0.143 (3)	0.160 (3)	0.149 (4)	0.014 (4)
O(23)	0.064 (3)	0.110 (4)	0.945 (4)	0.015 (13)
O(24)	0.474 (4)	0.070 (4)	0.145 (5)	0.028 (16)
O(25)	0.035 (4)	0.481 (4)	0.654 (5)	0.027 (14)
O(26)	0.368 (5)	0.051 (6)	0.388 (7)	0.058 (25)
O(27)	0.276 (4)	0.477 (4)	0.554 (5)	0.031 (16)
O(28)	0.331 (5)	0.014 (6)	0.776 (6)	0.050 (20)
O(29)	0.150 (6)	0.460 (6)	0.211 (7)	0.053 (23)

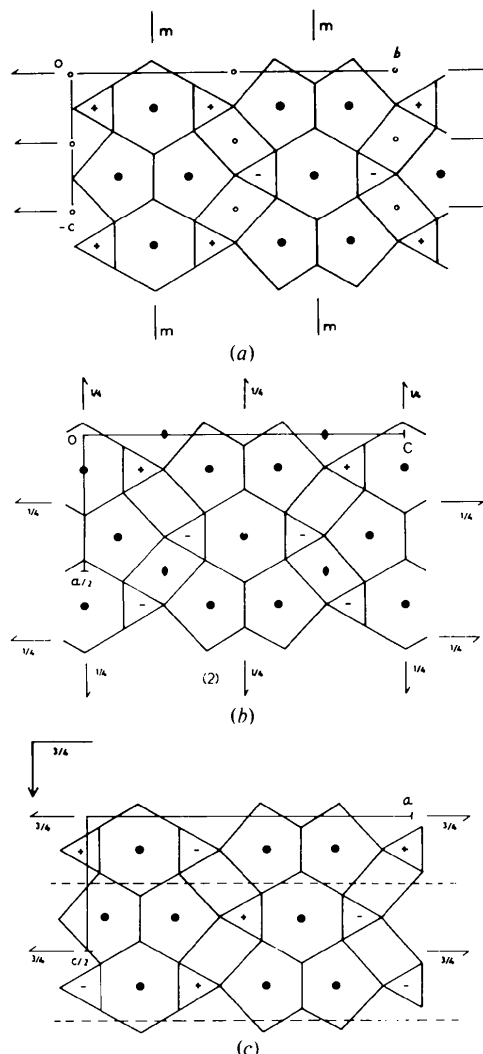


Fig. 1. Idealized  $[(\text{UO}_2)_3(\text{PO}_4)_2(\text{OH})_2]_n^{2n-}$  layer, with symmetry elements of the real layer. Full circles are  $\text{UO}_2$ , plus signs P—O, and minus signs O—P. (a) Dumontite, space group  $P2_1/m$ . (b) Phosphuranylite, space group  $C222_1$ . (c) Phurcalite, space group *Pbca*.

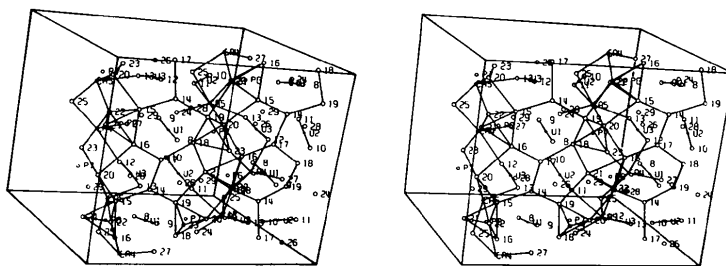


Fig. 2. Stereoscopic drawing (Johnson, 1965) of part of the unit cell of phurcalite viewed approximately along *b*, with *a* parallel to the interocular line; *y* varies from 0 to  $\frac{1}{4}$ .

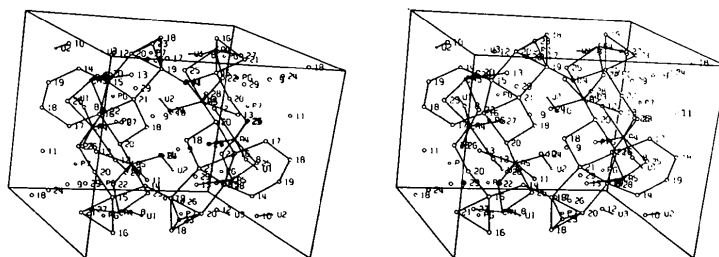


Fig. 3. Stereoscopic drawing of another part of the unit cell; *y* varies from 0.24 to 0.76.

Table 3. *Interatomic distances (Å) less than 3.00 Å*

Standard deviations are about 0.06 Å for U—O, 0.07 Å for Ca—O and P—O, and 0.09 Å for O—O.

U(1)—O(8)	1.70	U(1)—O(14)	2.18	U(2)—O(14)	2.28	U(3)—O(14)	2.35
—O(9)	1.75	—O(15)	2.82	—O(17)	2.30	—O(15)	2.36
Mean	1.725	—O(16)	2.57	—O(18)	2.45	—O(16)	2.33
		—O(17)	2.20	—O(19)	2.45	—O(17)	2.25
U(2)—O(10)	1.80	—O(18)	2.55	—O(21)	2.32	—O(20)	2.47
—O(11)	1.75	—O(19)	2.82	Mean	2.36	Mean	2.35
Mean	1.775	Mean	2.52				
U(3)—O(12)	1.73			Ca(4)—O(8)	2.66	Ca(5)—O(11)	2.84
—O(13)	1.83			—O(12)	2.92	—O(13)	2.72
Mean	1.78			—O(22)	2.43	—O(20)	2.42
				—O(23)	2.13	—O(22)	2.27
P(6)—O(15)	1.45	P(7)—O(18)	1.64	—O(25)	2.61	—O(24)	2.31
—O(16)	1.65	—O(19)	1.45	—O(26)	2.50	—O(25)	2.54
—O(21)	1.49	—O(20)	1.50	—O(27)	2.31	—O(28)	2.42
—O(22)	1.54	—O(23)	1.71	Mean	2.51	Mean	2.50
Mean	1.53	Mean	1.58				
O(8)—O(14)	2.78	O(11)—O(19)	2.93	O(14)—O(15)	2.75	O(18)—O(19)	2.49
—O(17)	2.86	—O(21)	2.77	—O(17)	2.66	—O(20)	2.52
—O(27)	2.98	—O(25)	2.83	—O(19)	2.61	—O(23)	2.62
—O(28)	2.88	—O(26)	2.91	—O(28)	2.98	O(19)—O(20)	2.51
O(9)—O(14)	2.64	O(12)—O(14)	2.64	O(15)—O(16)	2.43	—O(23)	2.64
—O(17)	2.86	—O(15)	2.74	—O(20)	2.86	O(20)—O(23)	2.61
—O(18)	2.92	—O(28)	2.81	—O(21)	2.28	O(21)—O(22)	2.47
—O(24)	2.85	O(13)—O(15)	2.89	—O(22)	2.48	O(22)—O(25)	2.94
O(10)—O(14)	2.88	—O(16)	2.99	—O(29)	2.78	O(23)—O(26)	2.95
—O(17)	2.83	—O(17)	2.86	O(16)—O(17)	2.51	O(24)—O(29)	2.94
—O(18)	2.86	—O(20)	2.91	—O(21)	2.62	O(25)—O(28)	2.91
—O(21)	2.99	—O(22)	2.94	—O(22)	2.72	O(26)—O(29)	2.83
O(11)—O(17)	2.97	—O(26)	2.95	O(17)—O(18)	2.57	O(27)—O(29)	2.68

**Introduction.** Phurcalite is a rare mineral recently described by Deliens & Piret (1978). The results are here completed with the structure determination. The space group was determined from Weissenberg photographs (systematic absences:  $hkl$ : none;  $0kl$ :  $k = 2n + 1$ ;  $h0l$ :  $l = 2n + 1$ ;  $hk0$ :  $h = 2n + 1$ ). Final cell dimensions and intensities were measured on a Picker four-circle diffractometer with the experimental conditions given in Table 1. Intensities were corrected for absorption (Busing & Levy, 1957). Scattering factors were those given by Cromer & Waber (1965) for neutral atoms; U was corrected for anomalous scattering (Cromer, 1965). The structure was solved by the use of the Patterson function. Refinement by Fourier methods and least squares (block-diagonal and full-matrix) gave a final residual  $R$  of 0.12 for all observed reflections.\* Calculations were performed with the XRAY system (1972). The final coordinates and temperature factors are given in Table 2.

**Discussion.** The structure consists of  $[(\text{UO}_2)_3(\text{PO}_4)_2(\text{OH})_2]^{2n-}$  layers similar to those in phosphuranylite,  $\text{Ca}[(\text{UO}_2)_3(\text{PO}_4)_2(\text{OH})_2] \cdot 6\text{H}_2\text{O}$  (Shashkin & Sidorenko, 1974), dumontite,  $\text{Pb}_2[(\text{UO}_2)_3(\text{PO}_4)_2(\text{OH})_2](\text{OH})_2 \cdot 3\text{H}_2\text{O}$  (Piret-Meunier, Léonard & Van Meerssche, 1962), and also very probably bergenite, renardite, arsenuranylite and hügelite (Figs. 1 and 2). The layers are connected by two Ca, two OH and four

$\text{H}_2\text{O}$  (Fig. 3). Principal bond lengths are listed in Table 3. The coordination figures are: for U(1) a hexagonal bipyramid, for U(2) and U(3) pentagonal bipyramids, for Ca(4) and Ca(5) irregular polyhedra with seven vertices, and for P(6) and P(7) tetrahedra. The mean U—O distance in the uranyl groups is shorter in the eightfold coordination ( $1.725 < 1.78 \text{ \AA}$ ); in contrast the mean U—O distance in the base of the hexagonal pyramid is longer than that in the pentagonal pyramid ( $2.52 > 2.355 \text{ \AA}$ ). Ca atoms are bonded to two O atoms of  $\text{PO}_4$  (of the same layer), two of  $\text{UO}_2$  (of different layers) and three of the interlayer space.

The authors thank Dr M. Deliens (Musée Royal de l'Afrique Centrale, Tervuren, Belgium) for supplying crystals. J-PD is indebted to the Fonds National Belge de la Recherche Scientifique for a research fellowship.

#### References

- BUSING, W. R. & LEVY, H. A. (1957). *Acta Cryst.* **10**, 180–182.  
 CROMER, D. T. (1965). *Acta Cryst.* **18**, 17–23.  
 CROMER, D. T. & WABER, J. T. (1965). *Acta Cryst.* **18**, 104–109.  
 DELIENS, M. & PIRET, P. (1978). To be published.  
 JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.  
 PIRET-MEUNIER, J., LÉONARD, A. & VAN MEERSSCHE, M. (1962). *Bull. Cl. Sci. Acad. R. Belg.* **48**, 751–760.  
 SHASHKIN, D. P. & SIDORENKO, G. A. (1974). *Dokl. Akad. Nauk SSSR*, **220**, 1161–1164.  
 XRAY system (1972). Tech. Rep. TR-192, edited by J. M. STEWART. Computer Science Center, Univ. of Maryland, College Park, Maryland.

\* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33355 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

*Acta Cryst.* (1978). **B34**, 1679–1681

## A Refinement of Potassium Chlorate

BY J. W. BATS\*

*Twente University of Technology, Chemical Physics Laboratory, PO Box 217, Enschede, The Netherlands*

(Received 17 January 1978; accepted 4 February 1978)

**Abstract.**  $\text{KClO}_3$ , monoclinic,  $P2_1/m$ ,  $Z = 2$ ,  $a = 4.6535$  (2),  $b = 5.5841$  (3),  $c = 7.0515$  (5)  $\text{Å}$ ,  $\beta = 108.723$  (6) $^\circ$ ,  $V = 173.54$  (2)  $\text{Å}^3$  (at 293 K); single-crystal diffractometer data up to  $\sin \theta/\lambda = 1.15 \text{ \AA}^{-1}$ ; Mo  $K\alpha$  radiation, graphite monochromator. The

structure was refined both from all reflections and from the high-order reflections only. The average O—Cl—O angle is  $106.4$  (1) $^\circ$ , the average Cl—O length  $1.497$  (2)  $\text{Å}$ .

**Introduction.**  $\text{KClO}_3$  was recrystallized from water. A crystal  $0.13 \times 0.16 \times 0.23 \text{ mm}$  was selected for data collection.

\* Present address: Institut für Kristallographie, Universität Frankfurt/Main, Senckenberg-Anlage 30, 6000 Frankfurt/Main, Federal Republic of Germany.