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 $[(UO_2)_3(PO_4)_2-(OH)_2]_n^{2n-}$ layers similar to those in phosphuranylite, $Ca[(UO_2)_3(PO_4)_2(OH)_2].6H_2O$ (Shashkin & Sidorenko, 1974), dumontite, $Pb_2[(UO_2)_3(PO_4)_2-(OH)_2](OH)_2.3H_2O$ (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

---

* 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.

H_2O (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 Å); 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 Å). Ca atoms are bonded to two O atoms of PO_4 (of the same layer), two of UO_2 (of different layers) and three of the interlayer space.

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 Refinement of Potassium Chlorate

BY J. W. BATS*

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

Abstract. $KClO_3$, monoclinic, $P2_1/m$, $Z = 2$, $a = 4·6535$ (2), $b = 5·5841$ (3), $c = 7·0515$ (5) Å, $\beta = 108·723$ (6)°, $V = 173·54$ (2) Å^3 (at 293 K); single-crystal diffractometer data up to $\sin\theta/\lambda = 1·15$ Å^{-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)°, the average Cl—O length 1·497 (2) Å.

---

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

Introduction. $KClO_3$ was recrystallized from water. A crystal 0·13 × 0·16 × 0·23 mm was selected for data collection.

Measurement of the lattice constants and intensities was performed on a Philips PW 1100 diffractometer. A $\theta/2\theta$ scan was applied with a scan range $\Delta\theta = (2\cdot0 + 0\cdot5 \text{ tg } \theta)^\circ$ and a scan speed of $0\cdot05^\circ \text{ s}^{-1}$. One sphere of data was measured up to $\sin \theta/\lambda = 1\cdot0$ Å^{-1}, yielding 5808 reflections. In addition, those reflections with $1\cdot0 < \sin \theta/\lambda < 1\cdot15$ Å^{-1} were measured for which a preset time count indicated that they might be measurable, yielding a further 226 reflections.

Related reflections observed at different times during the experiment showed a gradual decrease of about 9% for the weaker reflections, as a result of radiation damage. However, an increase of up to about 10% was observed for the strongest intensities, due to a reduction of extinction. Similar effects are often found during X-ray exposure of chlorates and perchlorates (Zachariasen, 1965; Berglund, Thomas & Tellgren, 1975).

The radiation decay was accounted for by the application of a linear scale factor, calculated from the reflections not affected by extinction. No attempt was made to correct for the drift in the extinction. It was believed, however, that after averaging of equivalent reflections a data set would be obtained with the extinction effect mainly corresponding to an average defect structure, so that existing extinction corrections would be valid. The data were corrected for absorption ($\mu = 20\cdot6$ cm^{-1}); the transmission factor ranged from $0\cdot695$ to $0\cdot797$. The related reflections were weighted averaged, resulting in 1679 unique reflections of which

Table 1. *Positional parameters*

Conventional refinement

	x	y	z
K	0·35533 (6)	0·25	0·70916 (4)
Cl	0·05446 (6)	0·25	0·17582 (5)
O(1)	−0·28069 (23)	0·25	0·11541 (19)
O(2)	0·15799 (15)	0·46184 (13)	0·30427 (12)

High-order refinement

	x	y	z
K	0·35544 (6)	0·25	0·70918 (6)
Cl	0·05382 (8)	0·25	0·17560 (6)
O(1)	−0·28199 (37)	0·25	0·11589 (38)
O(2)	0·15905 (30)	0·46205 (20)	0·30547 (27)

Table 2. *Bond distances* (Å) *and angles* (°)

	Conventional refinement	High-order refinement
Cl—O(1)	1·4789 (10)	1·4822 (18)
2 × Cl—O(2)	1·4736 (7)	1·4807 (12)
2 × K—O(2″)	2·8045 (7)	2·8019 (13)
K—O(1)	2·8214 (12)	2·8223 (23)
2 × K—O(2′)	2·8564 (7)	2·8582 (14)
2 × K—O(2)	2·9514 (9)	2·9447 (18)
2 × K—O(1′)	3·1181 (6)	3·1152 (13)
2 × O(1)—Cl—O(2)	106·47 (4)	106·55 (8)
O(2)—Cl—O(2)	106·78 (7)	106·20 (13)

109 with $I \leq 0$ were ignored. The weight of an individual reflection was taken as $w(I) = [\sigma^2(I)_{\text{counting}} + 0\cdot0006I^2]^{-1}$. The weight of an averaged reflection was taken as the sum of the weights of the individual reflections.

Initial positional parameters were taken from Aravindakshan (1958). Scattering factors were from Fukamachi (1971). For the K, Cl and O atoms, the anomalous-dispersion factors of Cromer & Liberman (1970) were applied. An isotropic extinction correction was used (Becker & Coppens, 1974). The minimum value of y defined as $y = F_o^2/F_{\text{corr}}^2$ was $0\cdot75$.

A refinement for all reflections (conventional refinement) resulted in $R(F) = 4\cdot7\%$, $R_w(F) = 3\cdot2\%$ and a goodness-of-fit $S^* = 2\cdot18$. A refinement for the 1134 reflections with $\sin \theta/\lambda > 0\cdot65$ Å^{-1} (high-order refinement) resulted in $R(F) = 7\cdot3\%$, $R_w(F) = 3\cdot5\%$ and $S = 1\cdot32$.†

The atomic coordinates are reported in Table 1, bond lengths and angles in Table 2.

Discussion. The present work confirms the structure reported for KClO$_3$ (Aravindakshan, 1958), but is more accurate. The average O—Cl—O angle of

* Defined as $S = [\sum w(|F_o| - |F_c|)^2/(\text{NO−NV})]^{1/2}$, with NO the number of observations and NV the number of variables.

† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33382 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

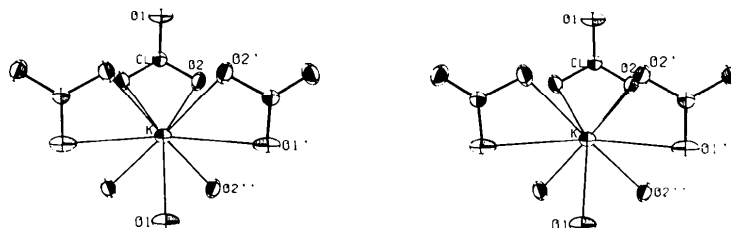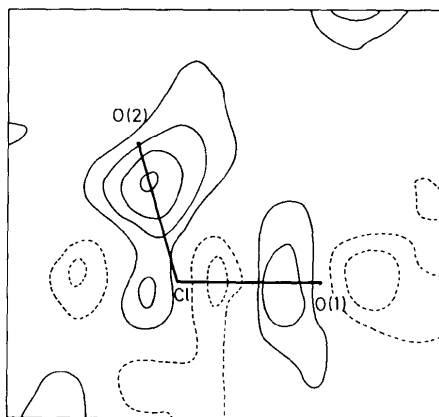

Fig. 1. Stereoscopic view of the coordination of the K atom. The thermal ellipsoids are the 50% probability surfaces.

Fig. 2. Difference synthesis in a section containing the O(1)—Cl—O(2) group, based on the reflections with sin $\theta/\lambda \leq 0.55$ Å^{-1} and the atomic parameters from the high-order refinement. Contour interval 0·1 e Å^{-3}; zero contour omitted.

106·4 (1)° compares well with the 106·8 (2)° found in NaClO$_3$ (Burke-Laing & Trueblood, 1977).

The librations of the ClO$_3$ group cannot be derived uniquely from the thermal parameters; however, a TLX analysis (Pawley, 1963) resulted in an average Cl—O length of 1·497 (2) Å, in agreement with the 1·502 (3) Å in NaClO$_3$. Each O atom is involved in three K—O bonds, leading to a ninefold coordination of the K atom (Fig. 1). The position of the K atom in the conventional

refinement is identical with that in the high-order one. The Cl atom is shifted along its anticipated lone-pair direction by 0·0034 (4) Å in the conventional refinement. The O atoms show displacements of 0·0069 (22) and 0·0099 (15) Å into the Cl—O bonds in the conventional refinement, compared with the high-order results. These shifts can be attributed to the aspherical distribution of the valence electrons, due to chemical bonding.

A final difference synthesis in a section through the chlorate group is shown in Fig. 2. Though some features are seen which may be related to bonding electrons, the figure is not very accurate. A study at low temperature would be required to give a clearer picture of the distribution of the bonding electrons.

References

Aravindakshan, C. (1958). Z. Kristallogr. 111, 35–45.
Becker, P. & Coppens, P. (1974). Acta Cryst. A30, 129–147.
Berglund, B., Thomas, J. O. & Tellgren, R. (1975). Acta Cryst. B31, 1842–1846.
Burke-Laing, M. E. & Trueblood, K. N. (1977). Acta Cryst. B33, 2698–2699.
Cromer, D. T. & Liberman, D. (1970). J. Chem. Phys. 53, 1891–1898.
Fukamachi, T. (1971). Technical Report of the Institute for Solid State Physics, Univ. of Tokyo, Series B12.
Pawley, G. S. (1963). Acta Cryst. 16, 1204–1208.
Zachariasen, W. H. (1965). Acta Cryst. 18, 703–705.

Di-μ-chloro-bis[4,6-η-(1-chlorocyclooctatrienyl)]dipalladium(II)

By N. C. Baenziger, Carol Valley Goebel, B. A. Foster and J. R. Doyle

Department of Chemistry, University of Iowa, Iowa City, Iowa 52242, USA

Abstract. (ClC$_8$H$_8$PdCl)$_2$, monoclinic, $P2_1/c$, a = 12·570 (1), b = 7·287 (4), c = 9·874 (5) Å, β = 96·28 (5)°, Z = 2, D_m = 2·00 (2), D_x = 2·08 g cm^{-3}, FW 562·9, $\sum \Delta|F|/\sum F_o$ = 0·030 from 1492 reflections. The complex is a chlorine-bridged dimer with Pd forming allylic-type π bonds to a boat-shaped cyclooctatetraene molecule which has undergone addition of a Cl atom trans to the allylic-type bond. Pd—C = 2·114 (4), 2·104 (3), 2·134 (3) Å; C—C(allyl) = 1·405 (5), 1·421 (5) Å; dihedral angle (allylic group/Pd coordination plane) = 119·8°.

Introduction. Intensity data (1492 observed present, 168 observed with intensity less than three standard deviations above the background) were collected using a four-circle diffractometer, a stepped θ–2θ scan, and graphite-monochromatized Mo $K\alpha$ radiation to 2θ = 52°. Intensity was measured at a minimum of seven steps over the angular diffraction range of a peak; the actual peak position was determined from these steps and additional steps were measured so that three steps were measured on each side of the peak. The step size, adjusted to allow for α_1–α_2 separation, ranged from