

the plane of the benzene ring [torsion angle C(5)–C(6)–O(19)–C(20) =  $-2.8(2)^\circ$ ]; this coplanarity results in a close approach between C(20) and C(5) [2.805(3) Å] which causes expansion of the angle C(5)–C(6)–O(19) [ $124.4(1)^\circ$ ] and contraction of the angle C(7)–C(6)–O(19) [ $115.2(1)^\circ$ ] (Sheldrick, Akkrigg & Geddes, 1980; Koetzle & Williams, 1976). The shortened distance C(7)–O(11) 1.367(2) Å and angle C(7)–O(11)–C(12)  $117.1(1)^\circ$  are, perhaps, indicative of some  $sp^2$  character of the atom O(11) (Durant, Bufkens, Lefebvre, Evrard & Michel, 1985). The phenyl ring is planar ( $\chi^2 = 37.4$ ) and is nearly perpendicular to the least-squares plane of the isocoumarin skeleton [dihedral angle  $91.7(5)^\circ$ ].

The molecular packing viewed along *a* is shown in Fig. 3. The molecules are held in the crystal by van der Waals interactions.

One of the authors (KS) thanks UGC (India) for financial assistance.

#### References

- DUAX, W. L., WEEKS, C. M. & ROHRER, D. C. (1976). *Topics in Stereochemistry*, Vol. 9, edited by N. L. ALLINGER & E. L. ELIEL, pp. 271–383. New York: John Wiley.
- DURANT, F., BUFKENS, F., LEFEVRE, F., EVRARD, G. & MICHEL, A. (1985). *Acta Cryst.* **C41**, 243–245.
- JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- KOETZLE, T. F. & WILLIAMS, G. J. B. (1976). *J. Am. Chem. Soc.* **98**, 2074–2078.
- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- MICHEL, A. G. & DURANT, F. (1976). *Acta Cryst.* **B32**, 321–323.
- NARDELLI, M. (1983). *Comput. Chem.* **7**, 95–98.
- SCHMALLE, H. W., JARCHOW, O. H., HAUSEN, B. M. & SCHULZ, K. H. (1982). *Acta Cryst.* **B38**, 2938–2941.
- SHELDRIK, B., AKKRIGG, D. & GEDDES, A. J. (1980). *Cryst. Struct. Commun.* **9**, 999–1004.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

## SHORT-FORMAT PAPERS

*Contributions intended for publication under this heading should follow the format given in the Checklist for Authors [Acta Cryst. (1985). C41, 1–4].*

*Acta Cryst.* (1989). **C45**, 810–811

### Structure of Rubidium Uranyl(VI) Trinitrate

BY ALLAN ZALKIN, LIESELOTTE K. TEMPLETON AND DAVID H. TEMPLETON

*Materials and Chemical Sciences Division, Lawrence Berkeley Laboratory, 1 Cyclotron Road, Berkeley, CA 94720, USA, and Department of Chemistry, University of California, Berkeley, CA 94720, USA*

(Received 8 September 1988; accepted 1 November 1988)

**Abstract.**  $\text{RbUO}_2(\text{NO}_3)_3$ ,  $M_r = 541.51$ , hexagonal,  $R\bar{3}c$ ,  $a = 9.384(4)$ ,  $c = 18.899(6)$  Å,  $V = 1441.3(14)$  Å<sup>3</sup>,  $Z = 6$ ,  $D_m = 3.72$ ,  $D_x = 3.743(4)$  g cm<sup>-3</sup>,  $\text{Mo K}\alpha$ ,  $\lambda(\alpha_1) = 0.70930$  Å,  $\mu = 209.7$  cm<sup>-1</sup>,  $F(000) = 1428$ ,  $T = 296$  K,  $R = 0.014$  for 261 independent reflections with  $I > 2\sigma(I)$ . The uranyl ion is coordinated in bidentate fashion by three nitrate ions in its equatorial plane. Bond lengths are U–O = 1.746(4) (uranyl), 2.474(3) (nitrate), N–O = 1.205(6) (terminal), 1.268(4) Å (bridge). The anomalous-scattering term  $f'$  for U measured at 0.71 Å is  $-10.7(2)$ .

**Experimental.** Crystals were prepared by slow evaporation of an aqueous solution of rubidium nitrate, uranyl

nitrate and nitric acid. The measured density is quoted from Hoard & Stroupe (1943). A prismatic crystal  $0.038 \times 0.063 \times 0.16$  mm (8 faces, elongated on *c*) was glued to a glass fiber and mounted on a Picker diffractometer equipped with a graphite monochromator. Cell dimensions were derived from 14 reflections in the range  $13 < \theta < 27^\circ$ . Integrated intensities were measured by  $\theta$ – $2\theta$  scan for 1295 reflections permitted by the space group in the ranges:  $\theta$  up to  $25^\circ$  for  $h = -11$  to 0,  $k = 0$  to 10,  $l = 0$  to 22 and up to  $27.5^\circ$  for  $h = -12$  to 0,  $k = 0$  to 11,  $l = -24$  to 0. After analytical correction for absorption ( $2.09 < A < 3.53$ ) and adjustments of up to 2% based on variation of two intensity standards, reflections which were equivalent were averaged giving 378 unique ones and 261 with

Table 1. Fractional atomic coordinates and thermal parameters with e.s.d.'s in parentheses

$$B_{eq} = \sum(B_{ij}a_i^*a_j^*a_r \cdot a_r)/3.$$

	x	y	z	$B_{eq}(\text{\AA}^2)$
Rb	0	0	0	3.44
U	0	0	$\frac{1}{2}$	2.60
O1	0	0	0.15761 (20)	3.55
N	0.3103 (6)	0	$\frac{1}{2}$	3.43
O2	0.4388 (4)	0	$\frac{1}{2}$	4.55
O3	0.3031 (3)	0.1310 (4)	0.25566 (15)	3.66

Table 2. Interatomic distances ( $\text{\AA}$ ) and bond angles ( $^\circ$ )

U—O1[2*]	1.746 (4)	Rb—O3[6]	3.304 (3)
U—O3[6]	2.474 (3)	O1—U—O3	92.48 (6)
N—O2[1]	1.205 (6)	O3—U—O3 <sup>1</sup>	51.25 (15)
N—O3[2]	1.268 (4)	O1—U—O3 <sup>1</sup>	87.52 (6)
Rb—O1[2]	2.979 (4)	O2—N—O3	122.5 (3)
Rb—O2[6]	3.186 (1)	O3—N—O3 <sup>1</sup>	115.0 (5)

Symmetry code: (i)  $x-y, -y, \frac{1}{2}-z$ .

\* Number of neighbors.

$F^2 > 2\sigma(F^2)$ ,  $R_{int} = 0.033$ . Refinement by least squares (on  $F$ ) with weights  $w = [\sigma(F)]^{-2}$ , derived from  $\sigma(F^2) = \{[\sigma_{cs}(F^2)]^2 + (0.01F^2)^2\}^{1/2}$ , was started with the coordinates of Barclay, Sabine & Taylor (1965) and anisotropic thermal parameters for all atoms. An empirical isotropic correction for extinction increased the largest  $F$  by 2%. The dispersion correction  $f'$  for  $U$  was included as a variable. The maximum final shift was less than  $0.0003 \sigma$ ; extremes of  $\Delta F$  synthesis were  $+1.4$  and  $-1.3 e \text{\AA}^{-3}$ ;  $R = 0.014$ ,  $wR = 0.012$ ,  $S = 1.15$ . Atomic scattering factors, including dispersion terms, for  $Rb^+$ ,  $U$ ,  $N$  and  $O$  were taken from *International Tables for X-ray Crystallography* (1974). Calculations were made with unpublished local programs. Atomic parameters are listed in Table 1 and interatomic distances and angles in Table 2.\* Fig. 1 shows a view of the structure.

**Related literature.** These crystals were prepared for the study of X-ray dichroism with synchrotron radiation (Templeton & Templeton, 1982), and the structure was redetermined to check the symmetry and molecular orientation, to learn the anisotropic thermal parameters, and as part of an investigation of uranium–oxygen bond distances (Templeton, Zalkin, Ruben & Templeton, 1985, and references therein). A study of Hoard & Stroupe (1943) using X-rays established the approximate structure. Coordinates determined by neutron diffraction by Barclay, Sabine & Taylor (1965) agree with those reported here within about the

\* Lists of anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51558 (3 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

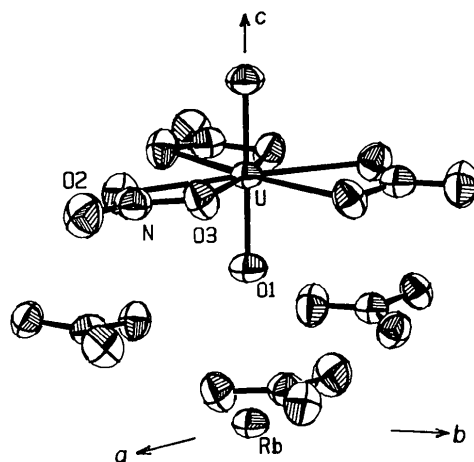


Fig. 1. View of the structure with 50% probability thermal ellipsoids (Johnson, 1976). Labels identify atoms in the asymmetric unit.

combined error estimates. The  $\sigma$ 's here are smaller by factors of 2 to 4. We observed no evidence of the monoclinic symmetry ( $C2/m$ ) reported by Kvapil, Kvapil & Tethal (1971). Our reflections with  $l = 2n$  can be indexed on their lattice, but odd- $l$  ones require the unit cell to be doubled. Crystals of  $CsUO_2(NO_3)_3$  and one form of  $NH_4UO_2(NO_3)_3$  are isomorphous with this rubidium salt (Groth, 1908). The theoretical value  $f' = -10.673$  calculated by Cromer & Liberman (1970) agrees with that found here,  $-10.7$  (2).

This research was supported by National Science Foundation Grant CHE-8515298 and by the Director, Office of Energy Research, Office of Basic Energy Sciences, Chemical Sciences Division, of the US Department of Energy under Contract No. DE-AC03-76SF00098.

## References

- BARCLAY, G. A., SABINE, T. M. & TAYLOR, J. C. (1965). *Acta Cryst.* **19**, 205–209.  
 CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.  
 GROTH, P. (1908). *Chemische Kristallographie*, Vol. II, pp. 150–152. Leipzig: Engelmann.  
 HOARD, J. L. & STROUPE, J. D. (1943). Atomic Energy Project Report A-1229. Reproduced by G. H. DIEKE & A. B. F. DUNCAN (1949). *Spectroscopic Properties of Uranium Compounds*, pp. 15–35. New York: McGraw-Hill.  
*International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)  
 JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.  
 KVAPIL, J., KVAPIL, J. & TETHAL, T. (1971). *J. Cryst. Growth*, **10**, 279–280.  
 TEMPLETON, D. H. & TEMPLETON, L. K. (1982). *Acta Cryst.* **A38**, 62–67.  
 TEMPLETON, D. H., ZALKIN, A., RUBEN, H. & TEMPLETON, L. K. (1985). *Acta Cryst.* **C41**, 1439–1441.