

which are  $\pm 0.013$  (3) Å from the plane. The intermolecular contacts are normal, except possibly for the shortest,  $S \cdots H(11)$  ( $-\frac{1}{2}x, \frac{1}{2} + y, \frac{1}{4}z$ ) = 2.76 (5) Å. Within experimental error, the cyclopentadienyl ring is equilateral and equiangular, in contrast to that of cyclopentadiene (Damiani, Ferretti & Gallinella (1976).

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### Bis(*N,N*-dimethylformamide)dinitratodioxouranium(VI), $C_6H_{14}N_4O_{10}U$

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**Abstract.**  $M_r = 540.23$ , monoclinic,  $P2_1/a$ ,  $a = 16.184$  (2),  $b = 8.465$  (1),  $c = 5.6176$  (5) Å,  $\beta = 101.87$  (1)°,  $V = 753.1$  (1) Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 2.38$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 10.263$  mm<sup>-1</sup>; final  $R = 0.028$  for 1188 observed reflexions. The coordination around the U atom is the usual centrosymmetric distorted hexagonal bipyramid.

**Introduction.** Uranyl complexes with several anionic bidentate ligands and dimethylformamide have been investigated to clarify the relationship between physical properties, structure and the nature of the metal–ligand bonds. Spectroscopic and thermogravimetric experiments are supported by the present crystal structure determination.

**Experimental.** Yellow prismatic single crystal,  $0.10 \times 0.10 \times 0.15$  mm, 2201 independent reflexions to  $\theta =$

$30^\circ$  at 295 K, four-circle diffractometer,  $\omega/2\theta$  scan technique, graphite-monochromated Mo  $K\alpha$  radiation, no crystal decomposition observed, absorption correction applied with *ORABS* (Schwarzenbach, 1972), absorption factors between 2.19 and 3.21; 1188 reflections observed [ $I > 2\sigma(I)$ ]; scattering factors for neutral atoms and anomalous-dispersion corrections for U from *International Tables for X-ray Crystallography* (1974); structure solved by Patterson method and successive Fourier syntheses; after several cycles of isotropic least-squares refinement with unit weights, a difference synthesis calculated with reflexions within  $\sin\theta/\lambda < 0.5 \text{ \AA}^{-1}$  showed all H atoms; to prevent bias on  $\langle \Delta F \rangle$  vs  $\langle F_o \rangle$  or  $\langle \sin\theta/\lambda \rangle$ , the last steps of the refinement were performed with weights  $w = 1/(a + b|F_o|)^2$ , where  $a = 2.02$  and  $b = 0.24$  for  $F_o < 10$ ,  $a = 6.88$  and  $b = -0.21$  for  $10 < F_o < 27$ ,  $a = 1.20$  and  $b = 0.01$  for  $F_o > 27$ ; after weighted refinement

with anisotropic temperature factors (fixed positional and thermal parameters for H atoms), final  $R = 0.028$  and  $R_w = 0.030$ ; \*  $F(000) = 500$ .

**Discussion.** Table 1 shows the final atomic parameters. Fig. 1 shows a perspective drawing (Johnson, 1965) of the molecule. The coordination around the U atoms is the usual centrosymmetric distorted hexagonal bipyramid, O(5) and O(5') being at 0.462 (6) Å from the plane defined by the other four O atoms around U. Table 2 contains a list of bond lengths and bond angles.

Most calculations were carried out with the XRAY70 system (Stewart, Kundell & Baldwin, 1970) on the Univac 1108 computer of the MEC (Madrid). Thanks are due to Professor S. Garcia-Blanco for his sponsorship.

\* Lists of structure factors, anisotropic thermal parameters and unrefined H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38092 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic parameters

$$U_{eq} = \frac{1}{3} \sum_i \sum_j a_i^* a_j^* a_r a_j \cos(a_r a_j)$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{eq}(\text{Å}^2 \times 10^4)$
U	0	0	0	319 (1)
O(1)	0.0424 (4)	0.0804 (7)	0.2895 (10)	451 (20)
O(2)	0.1428 (5)	-0.0343 (7)	-0.1097 (15)	542 (31)
O(3)	0.0895 (5)	0.1975 (8)	-0.1688 (13)	527 (24)
O(4)	0.2095 (5)	0.1463 (13)	-0.2659 (18)	849 (39)
O(5)	0.0691 (4)	-0.2421 (7)	0.1460 (11)	473 (21)
N(1)	0.1480 (6)	0.1072 (11)	-0.1836 (17)	501 (31)
N(2)	0.1266 (5)	-0.4040 (9)	0.4510 (14)	452 (24)
C(1)	0.1099 (6)	-0.2653 (11)	0.3560 (16)	437 (27)
C(2)	0.1793 (9)	-0.4243 (15)	0.6941 (19)	728 (43)
C(3)	0.0997 (11)	-0.5446 (13)	0.3162 (26)	870 (59)

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## Structure of (*p*-Bromophenyl)dichloro(phenyl)tellurium(IV), $(C_6H_5)(C_6H_4Br)TeCl_2$

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(Received 26 March 1982; accepted 18 August 1982)

**Abstract.**  $M_r = 431.6$ , triclinic,  $P\bar{1}$ ,  $a = 11.257$  (7),  $b = 10.817$  (4),  $c = 12.358$  (5) Å,  $\alpha = 82.61$  (4),  $\beta = 80.72$  (5),  $\gamma = 69.38$  (4)°,  $V = 1386$  (1) Å<sup>3</sup>,  $Z = 4$ ,  $D_c = 2.067$ ,  $D_m = 2.06$  Mg m<sup>-3</sup>, Mo  $K\alpha$ ,  $\lambda =$

0108-2701/83/010045-04\$01.50

Table 2. Bond lengths (Å) and bond angles (°)

U—O(1)	1.766 (5)	O(1)—U—O(2)	93.9 (3)
U—O(2)	2.528 (9)	O(1)—U—O(3)	86.9 (3)
U—O(3)	2.521 (7)	O(1)—U—O(5)	87.1 (3)
U—O(5)	2.397 (6)	O(2)—U—O(3)	50.2 (2)
N(1)—O(2)	1.28 (1)	O(2)—U—O(5)	66.4 (2)
N(1)—O(3)	1.23 (1)	O(3)—U—O(5')	64.4 (2)
N(1)—O(4)	1.23 (1)	O(5)—C(1)—N(2)	124.0 (8)
C(1)—O(5)	1.24 (1)	C(1)—N(2)—C(2)	121.6 (8)
C(1)—N(2)	1.30 (1)	C(1)—N(2)—C(3)	121.4 (8)
C(2)—N(2)	1.46 (1)	C(2)—N(2)—C(3)	116.9 (9)
C(3)—N(2)	1.43 (1)	O(2)—N(1)—O(3)	117.1 (9)
		O(2)—N(1)—O(4)	119.3 (9)
		O(3)—N(1)—O(4)	123.5 (9)

Symmetry operator: (i)  $-x, -y, -z$ .

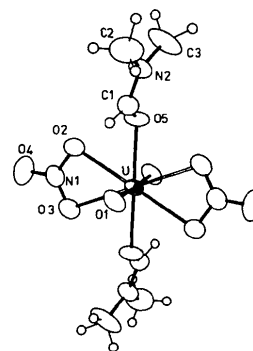


Fig. 1. Perspective drawing of the molecule. The U atom lies on a symmetry centre. Thermal ellipsoids are scaled at the 50% level.

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0.71069 Å,  $\mu = 5.63$  mm<sup>-1</sup>, 294 K. Final  $R = 0.048$ ,  $R_{wF} = 0.052$  for 3428 unique reflections. There are two independent molecules per unit cell and the crystal structure consists of discrete tetramers, in which

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