

1985). Molecular geometry calculations were performed using *CALC* (Gould & Taylor, 1985).

Related literature. Other small molecules such as PF₄Cl, CF₃Br and CF₃I undergo oxidative addition at Ir^I and Rh^I centres and we have determined the structures of some of the resulting Ir^{III} and Rh^{III} products (Blake, Cockman, Ebsworth, Henderson, Holloway, Pilkington & Rankin, 1987; Blake, Ebsworth, Holloway & Rieland, 1992).

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Structure of Pentakis(*N,N*-dimethylformamide)dioxouranium(VI) Tetrafluoroborate

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Abstract. [UO₂(C₃H₇NO)₅](BF₄)₂, $M_r = 809.11$, monoclinic, $C2/c$, $a = 21.077$ (5), $b = 13.296$ (3), $c = 11.525$ (2) Å, $\beta = 112.24$ (2)°, $V = 2989$ (1) Å³, $Z = 4$, $D_x = 1.798$ g cm⁻³, $\lambda(\text{Mo } K\alpha_1) = 0.71073$ Å, $\mu = 52.34$ cm⁻¹, $F(000) = 1560$, $T = 295$ K, $R = 0.029$ for 1935 independent observed reflections with $I > 3\sigma(I)$. The structure consists of packed UO₂(dmf)₅ cations [dmf = (CH₃)₂NCHO] and BF₄ anions. The linear UO₂ group is equatorially surrounded by five O atoms belonging to dmf molecules. The uranyl distance of 1.762 (3) Å is typical for the UO₂ group and the equatorial U—O distances fall in the range 2.334 (5)–2.383 (3) Å.

Experimental. A yellow single crystal of dimensions 0.35 × 0.35 × 0.30 mm, grown during the reaction between tetrafluoroboric acid and UO₃ in aqueous dmf solution, was used for data collection on an Enraf–Nonius CAD-4 diffractometer with graphite monochromator. Lattice parameters were determined by least-squares procedure from the setting angles of 25 reflections with $\theta = 8$ –12°. Data were collected up to $2\theta = 50^\circ$ and measured for $0 \leq h \leq 25$, $0 \leq k \leq 15$, $-13 \leq l \leq 13$, with ω - 2θ scans, ω -scan width $(0.80 + 0.35 \tan \theta)^\circ$ at 1.54–10° min⁻¹. Three standard reflections measured every hour (151, 731, 423) showed a loss of intensity of 4.7% in 36 h; a linear correction was applied. Lorentz–polarization correction and an empirical absorption correction using the program *DIFABS* (Walker & Stuart, 1983), maximum and minimum transmission 0.801 and

References

- BLAKE, A. J., COCKMAN, R. W., EBSWORTH, E. A. V., HENDERSON, S. G. D., HOLLOWAY, J. H., PILKINGTON, N. J. & RANKIN, D. W. H. (1987). *Phosphorus Sulfur*, **30**, 143–146.
 BLAKE, A. J., EBSWORTH, E. A. V., HOLLOWAY, J. H. & RIELAND, M. J. (1992). *Inorg. Chem.* Submitted.
 CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
 GOULD, R. O. & TAYLOR, P. (1985). *CALC*. Program for molecular geometry calculations. Univ. of Edinburgh, Scotland.
 MALLINSON, P. D. & MUIR, K. W. (1985). *J. Appl. Cryst.* **18**, 51–53.
 SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
 WALKER, N. & STUART, D. (1983). *Acta Cryst.* **A39**, 158–166.

1.298, were applied. 2832 reflections were measured, 2626 unique ($R_{\text{int}} = 0.024$), of which 1935 with $I > 3\sigma(I)$ were used in structure solution and refinement. The structure was solved by the heavy-atom method and refined by full-matrix least-squares procedures based on F , with anisotropic thermal parameters. 178 parameters were refined; H atoms not introduced. All calculations were performed on a MicroVAX II computer with the *Enraf–Nonius Structure Determination Package* (Frenz, 1985). The U atom lies on the diad axis, as do the O and N atoms of a dmf molecule [O(2) and N(2) introduced in refinements with an occupation factor of 0.5]. Atomic scattering factors including anomalous-scattering contributions were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Minimized function: $\sum w(|F_o| - |F_c|)^2$, where $w = 4F_o/\sigma^2(I)$ with $\sigma^2(I) = [\sigma^2(I_c) + (pF_o)^2]$, $p = 0.04$. Final $R = 0.029$, $wR = 0.034$, $S = 3.60$, $(\Delta/\sigma)_{\text{max}} = 0.01$, highest peak in the final difference Fourier map = $0.499 \text{ e } \text{Å}^{-3}$. The final atomic coordinates and equivalent isotropic temperature factors are given in Table 1.* Bond lengths and angles are listed in Table 2. A perspective view of the molecular structure with the atom-numbering scheme is shown in Fig. 1.

* Lists of structure factors, anisotropic thermal parameters, and bond distances and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55132 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: DU0327]

Table 1. Atomic coordinates and equivalent isotropic thermal parameters (\AA^2)
$$B_{eq} = (4/3) \sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

	x	y	z	B_{eq}
U	0.000	0.05533 (3)	0.250	3.780 (7)
F(1)	0.3111 (3)	0.1681 (7)	0.3457 (7)	15.3 (2)
F(2)	0.3768 (4)	0.0437 (5)	0.3439 (7)	12.4 (3)
F(3)	0.4122 (3)	0.1984 (7)	0.3440 (9)	14.9 (3)
F(4)	0.3313 (4)	0.1365 (8)	0.1808 (6)	13.5 (3)
O(1)	0.0406 (2)	0.0556 (5)	0.1415 (4)	5.6 (1)
O(2)	0.000	0.2309 (7)	0.250	7.0 (3)
O(3)	0.0647 (3)	-0.0881 (4)	0.3514 (5)	5.4 (1)
O(4)	-0.1051 (3)	0.1092 (4)	0.0922 (5)	5.7 (1)
N(2)	0.000	0.3973 (7)	0.250	6.5 (2)
N(3)	0.1022 (3)	-0.2168 (5)	0.4864 (5)	4.6 (1)
N(4)	-0.1917 (3)	0.0942 (5)	-0.0962 (6)	5.6 (2)
C(2)	0.0285 (8)	0.311 (1)	0.308 (1)	6.2 (4)
C(3)	0.0540 (4)	-0.1663 (6)	0.4012 (7)	5.0 (2)
C(4)	-0.1258 (4)	0.0982 (7)	-0.0248 (8)	5.8 (2)
C(21)	0.0428 (7)	0.455 (2)	0.350 (1)	16.1 (6)
C(31)	0.0875 (5)	-0.3113 (7)	0.5378 (9)	8.0 (3)
C(32)	0.1738 (4)	-0.1822 (8)	0.534 (1)	7.9 (3)
C(41)	-0.2100 (5)	0.082 (1)	-0.2329 (8)	8.4 (3)
C(42)	-0.2458 (4)	0.105 (1)	-0.048 (1)	8.7 (3)
B	0.3587 (5)	0.140 (1)	0.305 (1)	6.6 (3)

Table 2. Selected bond lengths (\AA) and bond angles ($^\circ$)

Uranium coordination sphere			
U—O(1)	1.762 (3)	U—O(2)	2.334 (5)
U—O(3)	2.378 (3)	U—O(4)	2.383 (3)
O(1)—U—O(2)	89.9 (1)	O(1)—U—O(3)	90.5 (1)
O(1)—U—O(4)	91.1 (1)	O(1)—U—O(4')	88.8 (1)
dmf			
O(2)—C(2)	1.28 (1)	N(3)—C(31)	1.471 (6)
C(2)—N(2)	1.35 (1)	N(3)—C(32)	1.471 (6)
N(2)—C(21)	1.391 (8)	O(4)—C(4)	1.259 (5)
O(3)—C(3)	1.249 (5)	C(4)—N(4)	1.320 (5)
C(3)—N(3)	1.301 (5)	N(4)—C(41)	1.482 (6)
		N(4)—C(42)	1.457 (6)
U—O(2)—C(2)	146.3 (5)	U—O(3)—C(3)	136.3 (3)
U—O(4)—C(4)	128.8 (3)		
BF₄			
B—F(1)	1.311 (7)	B—F(2)	1.359 (7)
B—F(3)	1.303 (7)	B—F(4)	1.329 (7)
F(1)—B—F(2)	108.2 (6)	F(1)—B—F(3)	112.7 (7)
F(1)—B—F(4)	108.4 (6)	F(2)—B—F(3)	110.5 (6)
F(2)—B—F(4)	106.0 (6)	F(3)—B—F(4)	110.8 (6)

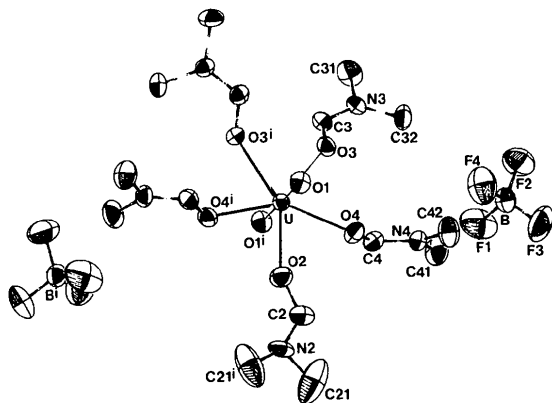
Symmetry code: (i) $-x, y, \frac{1}{2} - z$.

Fig. 1. Perspective view drawn using ORTEP (Johnson, 1976). Atoms labelled with (i) are related by the diad axis.

Related literature. This work is part of structural studies of uranyl with poorly coordinating anions in various solvents (Alcock & Esperas, 1977).

References

- ALCOCK, N. W. & ESPERAS, S. (1977). *J. Chem. Soc. Dalton Trans.* pp. 893–896.
 FRENZ, B. A. (1985). *Enraf-Nonius Structure Determination Package*. Version 3.0. Enraf-Nonius, Delft, The Netherlands.
 JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
 WALKER, N. & STUART, D. (1983). *Acta Cryst.* **A39**, 158–166.

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Structure of [1,3-Bis(diphenylphosphino)propane](η^5 -cyclopentadienyl)hydridoruthenium(II)

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Abstract. $[\text{RuH}(\text{C}_5\text{H}_5)(\text{C}_{27}\text{H}_{26}\text{P}_2)]$, $M_r = 579.6$, monoclinic, $P2_1/n$, $a = 9.188$ (2), $b = 19.307$ (3), $c = 15.545$ (3) \AA , $\beta = 102.98$ (1) $^\circ$, $V = 2687.0$ (8) \AA^3 , $Z =$

4, $D_x = 1.43$ Mg m^{-3} , $\lambda(\text{Mo K}\alpha) = 0.7107$ \AA , $\mu = 0.707$ mm^{-1} , $F(000) = 1192$, $T = 213$ K, $R = 0.039$ for 3412 observed reflections. The compound was