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Dinitratodioxobis(4-picoline *N*-oxide)uranium(VI)

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Key indicators

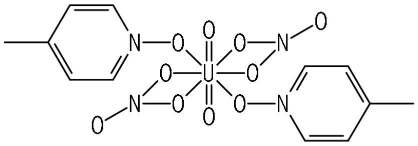
Single-crystal X-ray study
T = 203 K
Mean σ(C–C) = 0.006 Å
R factor = 0.022
wR factor = 0.052
Data-to-parameter ratio = 16.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $[UO_2(NO_3)_2(C_6H_7NO)_2]$, has a centre of symmetry. This is the first uranyl complex of a simple monodentate *N*-oxide ligand to be structurally characterized.

Comment

Although aromatic *N*-oxide complexes of uranium(VI) have been known for some time (Ahuja & Singh, 1973, and references therein), few have been structurally characterized. Those that have been structurally characterized invariably have a second donor group attached to the aromatic ring giving bidentate ligands. One such example is (2,2′-bipyridine *N*,*N*′-dioxide)dinitratodioxouranium(VI) (Alcock & Roberts, 1987) in which the uranium is eight-coordinate *via* two bidentate nitrate groups, two *N*-oxide O atoms and the two oxo ligands.



(I)

The title compound, (I), is eight-coordinate, with a hexagonal bipyramidal geometry. The U atom lies on a centre of symmetry, dictating a *trans* arrangement of the *N*-oxide ligands unlike the 2,2′-bipyridine *N*,*N*′-dioxide complex where a *cis* arrangement is dictated by ligand constraints. The NO_3 groups are bound slightly asymmetrically, with U—O bonds of 2.518 (3) and 2.544 (3) Å. These are significantly longer than the U—O (*N*-oxide) bonds of 2.376 (3) Å. These values are comparable to those found in the 2,2′-bipyridine *N*,*N*′-dioxide complex of 2.513 (18) and 2.378 (14) Å (Alcock & Roberts, 1987). The U═O distance is 1.769 (3) Å, which is typical of values found in uranyl complexes. There are no significant intermolecular interactions.

Experimental

The title complex was prepared by mixing solutions of $UO_2(NO_3)_2\cdot6H_2O$ and 4-picoline *N*-oxide in ethanol in a 1:2 molar ratio. Crystals of (I) were obtained by recrystalization from ethanol.

Crystal data

$[UO_2(NO_3)_2(C_6H_7NO)_2]$
M_r = 612.30
Monoclinic, $P2_1/c$
a = 7.9772 (2) Å
b = 13.5738 (2) Å
c = 8.2288 (2) Å
β = 91.583 (2)°
V = 890.68 (3) Å^3
Z = 2

D_x = 2.283 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6110 reflections
θ = 2–26°
μ = 9.17 mm^{-1}
T = 203 (2) K
Prism, yellow
0.32 × 0.26 × 0.22 mm

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Data collection

Siemens SMART diffractometer
Area-detector ω scans
Absorption correction: multi-scan (Blessing, 1995)
$T_{\min} = 0.088$, $T_{\max} = 0.133$
8841 measured reflections
1998 independent reflections

1750 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.020$
$\theta_{\max} = 27.5°$
$h = -10 \rightarrow 10$
$k = 0 \rightarrow 17$
$l = 0 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.022$
$wR(F^2) = 0.052$
$S = 1.24$
1998 reflections
125 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0153P)^2 + 1.8153P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.058$
$\Delta\rho_{\max} = 0.35$ e Å^{-3}
$\Delta\rho_{\min} = -0.65$ e Å^{-3}

Table 1
Selected bond lengths (Å).

U—O1	1.769 (3)	O2—N1	1.272 (5)
U—O5	2.376 (3)	O3—N1	1.269 (5)
U—O2	2.518 (3)	O4—N1	1.214 (5)
U—O3	2.544 (3)		

The data collection nominally covered over a hemisphere of reciprocal space, by a combination of three sets of exposures; each set had a different φ angle for the crystal and each exposure covered 0.3° in ω. Crystal decay was monitored by repeating the initial frames at the end of data collection and analyzing the duplicate reflections. H atoms were placed geometrically and refined with a riding model (including free rotation about the methyl C—C bonds), and with $U_{\rm iso}$ constrained to be 1.2 (1.5 for the methyl group) times $U_{\rm eq}$ of the carrier atom.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1994); software used to prepare material for publication: *SHELXL*97.

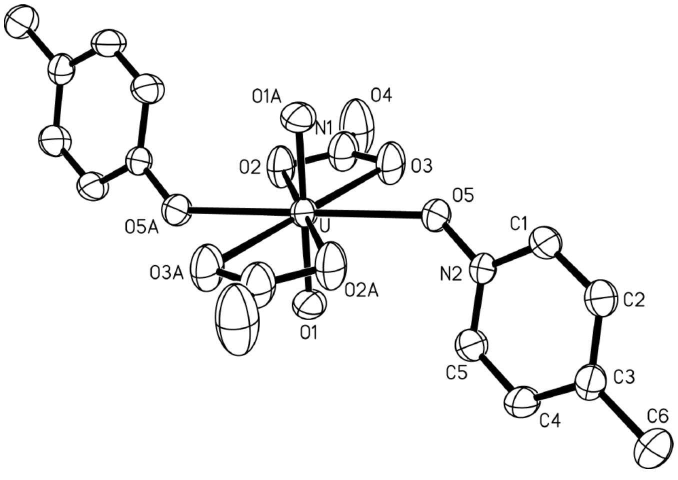

Figure 1
The structure of (I) showing 50% probability displacement ellipsoids. H atoms have been omitted.

References

Ahuja, I. S. & Singh, R. (1973). *J. Inorg. Nucl. Chem.* **35**, 561–566.
Alcock, N. W. & Roberts, M. M. (1987). *Acta Cryst.* C**43**, 476–478.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Siemens (1994). *SHELXTL*. Siemens Analytical Instruments X-ray Inc., Madison, Wisconsin, USA.
Siemens (1995). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.