

Asif.A. Tahir,^a Mazhar Hamid,^a
Muhammad Mazhar,^{a*} Matthias
Zeller^b and Allen D Hunter^b^aDepartment of Chemistry, Quaid-i-Azam
University, Islamabad 45320, Pakistan, and
^bYoungstown State University, STaRBURSTT–
Cyberdiffraction Consortium @ YSU &
Department of Chemistry, 1 University Plaza,
Youngstown, OH 44555-3663, USACorrespondence e-mail:
mazhar42pk@yahoo.com

Key indicators

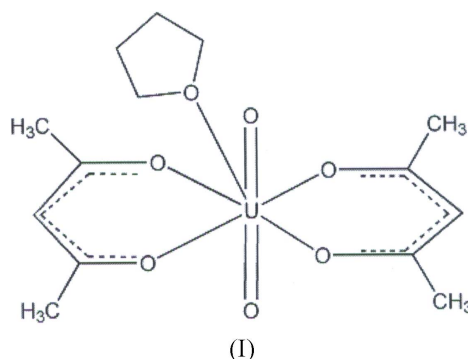
Single-crystal X-ray study
 $T = 100$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.020
 wR factor = 0.049
Data-to-parameter ratio = 20.1For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Dioxobis(pentane-2,4-dionato)(tetrahydro-
furan)uranium(VI)

The U^{VI} atom in the title complex, $[\text{UO}_2(\text{C}_5\text{H}_7\text{O}_2)_2(\text{C}_4\text{H}_8\text{O})]$, has a pentagonal–bipyramidal geometry. The U^{VI} atom is surrounded by seven O atoms, four of pentane-2,4-dione, one of the tetrahydrofuran ligand and the two uranyl O atoms.

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Comment

Compounds of uranyl β -diketonates with neutral donor ligands have been extensively studied due to their importance in the processes of separation of uranium by solvent extraction. Complexes with pentane-2,4-dione have also been reported; examples are (2-aminopentan-4-one)dioxobis(pentane-2,4-dionato)uranium(VI) (Rodgers *et al.*, 1977), aquadioxobis(pentane-2,4-dionato)uranium(VI) and 2,4,6-trimethylpyridinium dinitratodioxo(pentane-2,4-dionato)urate(VI) (Alcock & Flanders, 1987). All of them have a pentagonal–bipyramidal geometry with considerable deformation in the equatorial plane.



For the title compound, (I), the coordination geometry around the U atom closely resembles that of a standard pentagonal bipyramid. The principal bond lengths and angles are shown in Table 1. The apical U–O distances (mean 1.778 Å) are as expected for a uranium–oxygen double bond and are considerably shorter than equatorial U–O_{acac} counterparts (mean 2.351 Å). The latter are very close to but slightly shorter than the corresponding distances found for (2-*N*-methylaminopentan-4-one)dioxobis(pentane-2,4-dionato)uranium(VI) (mean 2.360 Å; Haigh *et al.*, 1976). The tetrahydrofuran (THF) molecule is less tightly bound than the chelating pentanedionate ligands and the U_{THF}–O bond length (2.507 Å) is longer than all other U–O bond lengths observed here.

The axial/equatorial O–U–O bond angles in (I) are close to the ideal pentagonal–bipyramidal angle of 90°, lying in the range 88.87 (9)–91.71 (9)°, whilst the axial/axial O–U–O angle of 178.39 (10)° is very close to the ideal angle of 180°.

The 'bite' angles of the two acac groups, O3—U1—O4 and O5—U1—O6 [71.60 (7) and 70.88 (7)°] are close to the ideal pentagonal–bipyramidal angle of 72°, and are in good agreement with the average bite angle of 70.8° in [UO₂(acac)₂(H₂O)₂] (Alcock & Flanders, 1987).

Experimental

[UO₂(acac)₂] was prepared by the reaction of [UO₂(OAc)₂]·3H₂O (1.5 g, 3.53 mmol) with pentane-2,4-dione (acacH) (0.07 g, 7.07 mmol) in toluene (5 ml). Continuous stirring for 3 h at 343 K followed by removal of all the volatiles *in vacuo* gave a dry powder. Orange block-shaped crystals suitable for single-crystal X-ray analysis were grown from a concentrated THF solution at room temperature after 1 d.

Crystal data

[UO ₂ (C ₅ H ₇ O ₂) ₂ (C ₄ H ₈ O)]	Z = 4
<i>M_r</i> = 540.35	<i>D_x</i> = 2.143 Mg m ⁻³
Monoclinic, <i>P</i> ₂ ₁ / <i>c</i>	Mo <i>K</i> α radiation
<i>a</i> = 13.2988 (8) Å	<i>μ</i> = 9.72 mm ⁻¹
<i>b</i> = 8.0155 (5) Å	<i>T</i> = 100 (2) K
<i>c</i> = 16.0928 (9) Å	Block, orange
<i>β</i> = 102.465 (1)°	0.34 × 0.22 × 0.22 mm
<i>V</i> = 1675.00 (17) Å ³	

Data collection

Bruker AXS SMART APEX CCD diffractometer	10188 measured reflections
<i>ω</i> scans	4090 independent reflections
Absorption correction: multi-scan (SADABS in S _{AINT-Plus} ; Bruker, 2003)	3830 reflections with <i>I</i> > 2σ(<i>I</i>)
<i>T</i> _{min} = 0.065, <i>T</i> _{max} = 0.118	<i>R</i> _{int} = 0.019
	<i>θ</i> _{max} = 28.3°

Refinement

Refinement on <i>F</i> ²	$w = 1/[\sigma^2(F_o^2) + (0.0231P)^2 + 1.9168P]$
$R[F^2 > 2\sigma(F^2)] = 0.020$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.049$	(Δ/σ) _{max} = 0.001
<i>S</i> = 1.10	$\Delta\rho_{\text{max}} = 1.30 \text{ e } \text{Å}^{-3}$
4090 reflections	$\Delta\rho_{\text{min}} = -0.86 \text{ e } \text{Å}^{-3}$
203 parameters	
H-atom parameters constrained	

All H atoms were positioned geometrically and were refined with $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{carrier atom})$, where $x = 1.5$ for methyl H and 1.2 for all other H atoms. The s.u. values of the cell parameters are taken from the software, recognizing that the values are unreasonably small

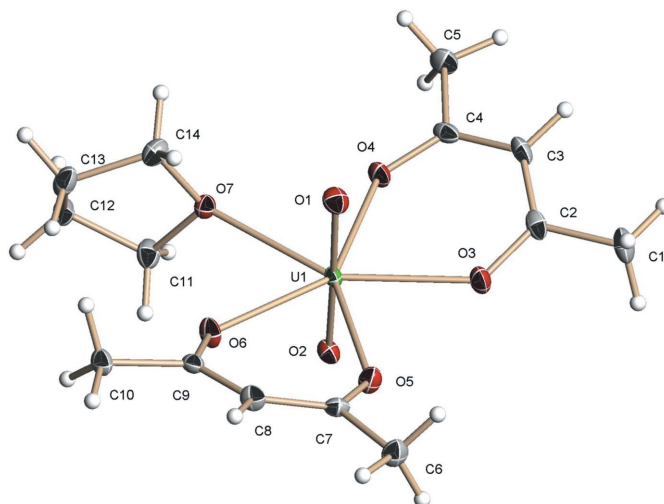


Figure 1

View of the title complex, showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

(Herbstein, 2000). The highest peak is located 0.79 Å from the uranium atom.

Data collection: *SMART* (Bruker, 2002); cell refinement: *S_{AINT-Plus}* (Bruker, 2003); data reduction: *S_{AINT-Plus}*; program(s) used to solve structure: *SHELXTL* (Bruker, 2003); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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References

- Alcock, N. W. & Flanders, D. J. (1987). *Acta Cryst.* **C43**, 1480–1483.
 Bruker (2002). *SMART for WNT/2000*. Version 5.630. Bruker AXS Inc., Madison, Wisconsin, USA.
 Bruker (2003). *S_{AINT-Plus}* (Version 6.45) and *SHELXTL* (Version 6.14). Bruker AXS Inc., Madison, Wisconsin, USA.
 Haigh, J. M., Nassimbeni, L. R., Pauptit, R. A., Rodgers, A. L. & Sheldrick, G. M. (1976). *Acta Cryst.* **B32**, 1398–1401.
 Herbstein, F. H. (2000). *Acta Cryst.* **B56**, 547–557.
 Rodgers, A. L., Nassimbeni, L. R., Pauptit, R. A., Orpen, G. & Haigh, J. M. (1977). *Acta Cryst.* **B33**, 3110–3113.