

Nuray Şenyüz,^a Çiğdem
Yüksektepe,^{b*} Hümeysra Batı,^a
Nezihe Çalışkan^b and Orhan
Büyükgüngör^b

^aDepartment of Chemistry, Arts and Sciences
Faculty, Ondokuz Mayıs University, 55139
Samsun, Turkey, and ^bDepartment of Physics,
Arts and Sciences Faculty, Ondokuz Mayıs
University, 55139 Samsun, Turkey

Correspondence e-mail: yuksekc@yahoo.com

Key indicators

Single-crystal X-ray study
T = 296 K
Mean $\sigma(\text{C}-\text{C}) = 0.016 \text{ \AA}$
R factor = 0.042
wR factor = 0.099
Data-to-parameter ratio = 15.4

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

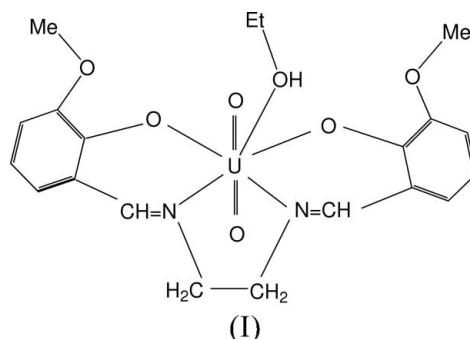
{6,6'-Dimethoxy-2,2'-[ethane-1,2-diylbis-(nitrilomethylidyne)]diphenolato}(ethanol)-dioxouranium(VI)

In the title compound, $[\text{UO}_2(\text{C}_{18}\text{H}_{18}\text{N}_2\text{O}_4)(\text{C}_2\text{H}_6\text{O})]$, the U atom has a distorted pentagonal–bipyramidal geometry with a tetradentate Schiff base and ethanol ligands in the equatorial plane and oxo atoms in the axial positions. Complex molecules are connected by $\text{O}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{O}$ interactions, forming dimers in the crystal structure.

Received 2 October 2006
Accepted 26 October 2006

Comment

Many Schiff base complexes have potential biological interest in an attempt to mimic the structural and functional features of multimetal active sites. Schiff bases have also found useful applications in the synthesis of molecular and supramolecular polymeric functional systems exhibiting specific optical or magnetic properties (Salmon *et al.*, 2004). Here, we report the synthesis and crystal structure of the title compound, (I).



The U^{VI} atom has a distorted pentagonal–bipyramidal coordination geometry, with the Schiff base and ethanol ligands in the equatorial plane (Fig. 1). The phenoxide atoms O1 and O4 are displaced by 0.066 (5) and 0.143 (5) \AA , respectively, from the plane defined by O1/N1/N2/O4/U1/O5.

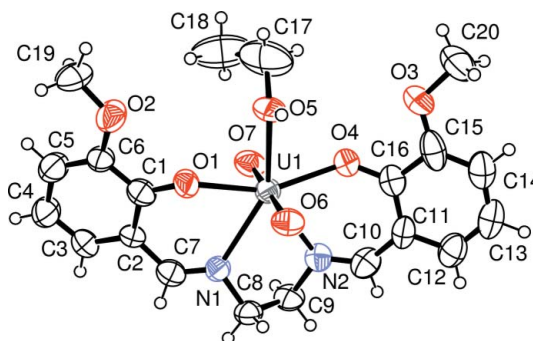
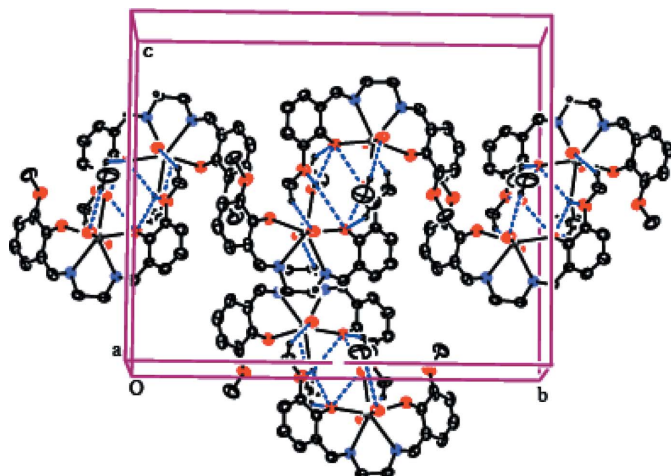


Figure 1
The molecular structure of (I), showing the atom numbering scheme and 50% probability displacement ellipsoids.


Figure 2

The crystal structure of (I). H atoms have been omitted unless they are involved in hydrogen bonds (dashed lines).

The oxo atoms O6 and O7 occupy the axial positions. The U atom is displaced by 0.053 (3) Å from the equatorial plane towards atom O7. The U1–O6 and U1–O7 bond distances (Table 1) are typical for U=O double bonds, close to those reported previously [1.778 (4) and 1.794 (5) Å; Masci *et al.*, 2005].

In the crystal structure, molecules related by an inversion centre are connected by O5–H5A···O1ⁱ, O5–H5A···O2ⁱ, C17–H17B···O1ⁱ and C19–H19A···O6ⁱ interactions (Table 1), forming dimers having two intersecting R₂²(10) and R₂²(16) rings (Fig. 2). Dimers are interlinked along the *c* axis via C8–H8B···O7ⁱⁱ interactions.

Experimental

A solution of UO₂(CH₃COO)₂·2H₂O (1 mmol, 0.424 g) in hot ethanol (10 ml) was added to a hot ethanol solution (15 ml) of *N,N'*-ethylenebis(3-methoxysalicylideneimine) (1 mmol, 0.328 g). The mixture was refluxed for 4 h, with stirring. The mixture was then cooled to room temperature and a red powder was precipitated. The product was filtered and washed with ethanol. Crystals of (I) suitable for X-ray diffraction were grown from an acetone/ethanol/*n*-hexane (1:1:1 v/v/v) solution by slow evaporation over a period of 2 d (yield 79%).

Crystal data

[U(C ₁₈ H ₁₈ N ₂ O ₄)O ₂ (C ₂ H ₆ O)]	<i>Z</i> = 8
<i>M_r</i> = 642.44	<i>D_x</i> = 1.984 Mg m ⁻³
Orthorhombic, <i>Pbca</i>	Mo <i>K</i> α radiation
<i>a</i> = 9.2132 (4) Å	<i>μ</i> = 7.60 mm ⁻¹
<i>b</i> = 23.6595 (10) Å	<i>T</i> = 296 K
<i>c</i> = 19.7075 (10) Å	Plate, red
<i>V</i> = 4295.8 (3) Å ³	0.43 × 0.23 × 0.05 mm

Data collection

Stoe IPDS-2 diffractometer	57109 measured reflections
<i>ω</i> scans	4219 independent reflections
Absorption correction: integration (<i>X-RED32</i> ; Stoe & Cie, 2002)	2789 reflections with <i>I</i> > 2σ(<i>I</i>)
<i>T</i> _{min} = 0.104, <i>T</i> _{max} = 0.597	<i>R</i> _{int} = 0.104
	<i>θ</i> _{max} = 26.0°

Refinement

Refinement on *F*²
 $R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.099$
 $S = 1.10$
 4219 reflections
 274 parameters
 H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0393P)^2 + 9.9627P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 1.26 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.68 \text{ e } \text{Å}^{-3}$

Table 1

Selected geometric parameters (Å, °).

U1–O7	1.752 (7)	U1–O5	2.471 (6)
U1–O6	1.763 (7)	U1–N1	2.540 (7)
U1–O4	2.253 (6)	U1–N2	2.542 (8)
U1–O1	2.265 (6)		
O7–U1–O6	177.3 (3)	O7–U1–O5	96.1 (3)
O7–U1–O4	87.8 (3)	O6–U1–O5	86.5 (3)
O6–U1–O4	91.7 (3)	O4–U1–N1	135.6 (2)
O7–U1–O1	90.3 (3)	O1–U1–N1	70.1 (2)
O6–U1–O1	91.3 (3)	O5–U1–N1	146.0 (2)
O4–U1–O1	154.2 (2)	O4–U1–N2	69.9 (2)

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
O5–H5A···O1 ⁱ	0.83 (6)	2.53 (15)	3.059 (9)	124 (15)
O5–H5A···O2 ⁱ	0.83 (6)	2.19 (8)	2.952 (11)	154 (16)
C8–H8B···O7 ⁱⁱ	0.97	2.50	3.183 (11)	127
C17–H17B···O1 ⁱ	0.97	2.45	3.163 (19)	130
C18–H18C···O7	0.96	2.57	3.382 (19)	142
C19–H19A···O6 ⁱ	0.96	2.59	3.423 (13)	145

Symmetry codes: (i) $-x + 1, -y + 1, -z$; (ii) $x + \frac{1}{2}, y, -z + \frac{1}{2}$.

The hydroxy H atom was located in a difference map and refined with an O–H distance restraint of 0.83 (6) Å [$U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$]. All other H atoms were positioned geometrically and treated as riding, with C–H = 0.93–0.97 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$. The highest residual electron density peak is located 1.21 Å from atom U1.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

References

- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
 Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
 Masci, B., Levi Mortera, S. & Thuéry, P. (2005). *Acta Cryst.* **C61**, m482–m484.
 Salmon, L., Thuéry, P. & Ephritikhine, M. (2004). *J. Chem. Soc. Dalton Trans.* pp. 1635–1643.
 Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
 Stoe & Cie (2002). *X-AREA* (Version 1.18) and *X-RED32* (Version 1.04). Stoe & Cie, Darmstadt, Germany.