Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis(2-benzoyl-1-phenylethenolato-κ^2O,O')(ethanol-κO)dioxidouranium(VI)

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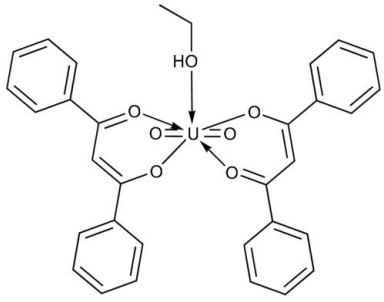
Received 21 November 2007; accepted 27 November 2007

Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.009 Å; R factor = 0.043; wR factor = 0.083; data-to-parameter ratio = 17.8.

In the title compound, $[U(C_{15}H_{11}O_2)_2O_2(C_2H_6O)]$, the U^{VI} atom has a pentagonal–bipyramidal coordination geometry. The two so-called '-yl' O atoms occupy the axial positions whereas four O atoms from the two chelating dibenzoylmethanate ligands and the O atom from the ethanol molecule are situated in the equatorial plane. Intermolecular hydrogen bonds between one of the '-yl' O atoms and the ethanol OH group assemble molecules into a centrosymmetric dimer.

Related literature

For literature on the structural chemistry of uranyl(VI) complexes with dibenzoylmethanate and unidentate ligands, see: Alagar *et al.* (2003, 2004); Fun, Kannan, Chantrapromma *et al.* (2002); Fun, Kannan, Usman *et al.* (2002); Kannan & Gerguson (1997); Kannan *et al.* (1995, 1997, 2000); Linert *et al.* (2001); Mizuoka & Ikeda (2004); Rajagopal *et al.* (2002).



Experimental

Crystal data

$[U(C_{15}H_{11}O_2)_2O_2(C_2H_6O)]$
M_r = 762.57
Monoclinic, $P2_1/c$
a = 9.088 (5) Å
b = 12.141 (7) Å
c = 25.878 (13) Å
β = 99.126 (16)°
V = 2819 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 5.81 mm^{-1}
T = 173 (2) K
0.40 × 0.30 × 0.10 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: numerical (Higashi, 1999)
T_{min} = 0.205, T_{max} = 0.594
26257 measured reflections
6442 independent reflections
4539 reflections with $I > 2\sigma(I)$
R_{int} = 0.097

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.083
S = 1.01
6442 reflections
362 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.05 e Å^{-3}
$\Delta\rho_{min}$ = −0.69 e Å^{-3}

Table 1
Selected bond lengths (Å).

U1—O1	1.762 (4)	U1—O5	2.347 (4)
U1—O2	1.787 (4)	U1—O3	2.365 (4)
U1—O4	2.288 (4)	U1—O7	2.464 (4)
U1—O6	2.339 (4)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O7—H6···O2^{i}	0.86	1.94	2.765 (5)	162

Symmetry code: (i) $-x+1, -y+1, -z+1$.

Data collection: *PROCESS-AUTO* (Rigaku, 2006); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (MSC/Rigaku, 2006); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999) and *DIRDIF99* (Beurskens *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2123).

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supplementary materials

Acta Cryst. (2008). E**64**, m219-m220 [**doi:10.1107/S1600536807063799**]

Bis(2-benzoyl-1-phenylethenolato-κ^2O,O')(ethanol-κO)dioxidouranium(VI)

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Comment

Structural chemistry of uranyl(VI) complexes with dibenzoylmethanate (dbm) and unidentate ligands (*L*) has been extensively explored (Kannan *et al.*, 1997*a,b*, 1995, 2000; Rajagopal *et al.*, 2002; Fun, Kannan, S., Chantrapromma *et al.* 2002;Fun, Kannan, S., Usman *et al.* 2002 Alagar *et al.*, 2003, 2004). Generally, two dbm and one *L* are placed in an equatorial plane of the uranyl(VI) ion (UO_2^{2+}). This results in a $UO_2(dbm)_2L$ complex with a pentagonal-bipyramidal geometry around the uranium atom. In our previous study, we also used $UO_2(dbm)_2DMSO$ complex (DMSO = dimethyl sulfoxide) as a precursor of a corresponding uranyl(V) complex, $[U^VO_2(dbm)_2DMSO]^-$ (Mizuoka & Ikeda, 2004). In our recent experiment, we obtained crystals of the title compound, $UO_2(dbm)_2EtOH$ (**I**), suitable for single-crystal X-ray analysis. In this paper, we report results of the structure determination of **I** to accumulate more structural data in a series of $UO_2(dbm)_2L$ complexes.

The molecular structure of **I** is shown in Fig. 1. The uranium atom in **I** is surrounded by seven O atoms; two O are at the axial positions, and the remaining five O from dbm and EtOH in the equatorial plane. As a consequence, the coordination geometry around U in **I** is pentagonal bipyramidal. The deviations of the O atoms in dbm and EtOH from the equatorial plane are within 0.1 Å.

Bond lengths in **I** are listed in Table 1, and are similar to the structural parameters of other $UO_2(dbm)_2L$ complexes reported previously. As an exception, the bond length between U and O of EtOH [U1–O7 = 2.464 (4) Å] seems to be slightly longer than the corresponding bond lengths in the $UO_2(dbm)_2L$ complexes (*L* = di-substituted sulfoxides, dibenzoylacetone, and triphenylphosphine oxide), while shorter than those with H_2O, malonanilide, and camphor. It is likely that the bond length between U and O of *L* ($U–O_L$) depends on donicity of *L* (Linert *et al.* 2001). In this discussion, the steric effect of *L* should also be taken into account. However, it is not the case of **I**, because there seems to be no significant steric hindrance due to EtOH in its molecular structure shown in Fig. 1.

Intermolecular hydrogen bond between OH group of ethanol and the -yl oxygen, $O7–H6\cdots O2^i$ [symmetry operation: (i) 1 - *x*, 1 - *y*, 1 - *z*], was observed between the neighboring complex molecules (Table 2). This results in a dimeric aggregate of **I** as shown in Fig. 2.

Experimental

Solution of uranyl nitrate hexahydrate (1.20 g) in 2 ml of ethanol was added to a hot solution (10 ml) of dibenzoylmethane (Hdbm, 0.523 g) in 10 ml of ethanol with vigorous stirring. After addition of 1 N NaOH (3 ml), the solution was concentrated by heating, and then cooled to room temperature. Deposited crystals of the title compound were filtered off, washed with ethanol, and dried under the ambient atmosphere.

Refinement

The structure was solved by direct methods, *SIR97* (Altomare *et al.*, 1999) and expanded using Fourier techniques (Beurskens *et al.*, 1999). The H atom from the OH group was located from a difference map and the remaining H atoms were placed at calculated positions. All H atoms were refined as riding on their parent atoms with $U_{iso}(H) = 1.2U_{eq}(C,O)$.

Figures

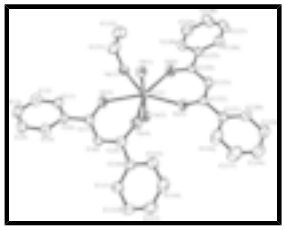

Fig. 1. : Molecular structure of $UO_2(dbm)_2EtOH$ (**I**) with displacement elipsoids shown at the 50% probability level. Hydrogen atoms are omitted for clarity.

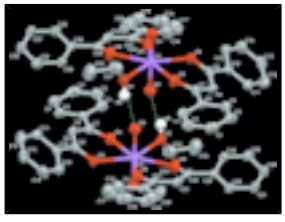

Fig. 2. : Structure of the dimeric aggregate of **I** [molecules are related by the symmetry operation (i) 1 - *x*, 1 - *y*, 1 - *z*]. Dashed lines indicate intermolecular –OH···O_{yl} hydrogen bonds between neighboring molecules. Hydrogen atoms except for the OH group are omitted for clarity.

Bis(2-benzoyl-1-phenylethenolato-κ^2*O*,*O*')(ethanol-κ*O*)dioxidouranium(VI)

Crystal data

$[U(C_{15}H_{11}O_2)_2O_2(C_2H_6O)]$	F_{000} = 1472
M_r = 762.57	D_x = 1.797 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71075 Å
Hall symbol: -P 2ybc	Cell parameters from 22955 reflections
a = 9.088 (5) Å	θ = 3.0–27.6º
b = 12.141 (7) Å	μ = 5.81 mm^{-1}
c = 25.878 (13) Å	*T* = 173 (2) K
β = 99.126 (16)º	Platelet, orange
V = 2819 (3) Å^3	0.40 × 0.30 × 0.10 mm
Z = 4	

Data collection

Rigaku R-AXIS RAPID diffractometer	6442 independent reflections
Radiation source: fine-focus sealed tube	4539 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.097
Detector resolution: 10.00 pixels mm^{-1}	θ_{max} = 27.5º
T = 173(2) K	θ_{min} = 3.0º

ω scans

Absorption correction: numerical (Higashi, 1999)

T_{min} = 0.205, T_{max} = 0.594

26257 measured reflections

$h = -11 \rightarrow 11$

$k = -15 \rightarrow 15$

$l = -33 \rightarrow 33$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.043$

$wR(F^2) = 0.083$

$S = 1.01$

6442 reflections

362 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0288P)^2 + 2.9796P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 1.05$ e Å^{-3}

$\Delta\rho_{min} = -0.69$ e Å^{-3}

Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
U1	0.56596 (2)	0.415650 (18)	0.597530 (8)	0.03064 (7)
O1	0.4826 (4)	0.3391 (4)	0.64300 (17)	0.0415 (10)
O2	0.6473 (4)	0.4912 (4)	0.54993 (16)	0.0403 (10)
O3	0.4712 (4)	0.2914 (3)	0.52999 (16)	0.0396 (10)
O4	0.7334 (4)	0.2763 (3)	0.59376 (15)	0.0382 (10)
O5	0.7648 (4)	0.4744 (4)	0.66069 (17)	0.0419 (11)
O6	0.4991 (5)	0.5764 (4)	0.63790 (18)	0.0491 (12)
O7	0.3209 (4)	0.4808 (3)	0.55377 (15)	0.0372 (10)
H6	0.3287	0.5044	0.5230	0.031*
C1	0.2955 (7)	0.2313 (5)	0.4362 (2)	0.0387 (14)
H1	0.2766	0.3017	0.4498	0.046*
C2	0.1947 (7)	0.1867 (6)	0.3973 (3)	0.0450 (16)
H2	0.1063	0.2260	0.3839	0.054*

C3	0.2210 (7)	0.0843 (6)	0.3772 (2)	0.0469 (16)
H3	0.1506	0.0535	0.3500	0.056*
C4	0.3477 (7)	0.0271 (6)	0.3962 (2)	0.0453 (16)
H4	0.3646	−0.0433	0.3822	0.054*
C5	0.4521 (7)	0.0717 (5)	0.4360 (2)	0.0387 (14)
H5	0.5404	0.0321	0.4491	0.046*
C6	0.4260 (6)	0.1754 (5)	0.4566 (2)	0.0312 (13)
C7	0.5299 (6)	0.2243 (5)	0.5012 (2)	0.0327 (13)
C8	0.6823 (6)	0.1955 (5)	0.5101 (2)	0.0330 (13)
H8	0.7219	0.1563	0.4837	0.040*
C9	0.7770 (6)	0.2221 (5)	0.5562 (2)	0.0322 (13)
C10	0.9351 (6)	0.1858 (5)	0.5657 (2)	0.0319 (13)
C11	1.0087 (7)	0.1396 (5)	0.5280 (3)	0.0390 (15)
H11	0.9558	0.1268	0.4938	0.047*
C12	1.1592 (7)	0.1116 (5)	0.5393 (3)	0.0461 (17)
H12	1.2081	0.0805	0.5130	0.055*
C13	1.2362 (7)	0.1291 (5)	0.5885 (3)	0.0472 (17)
H13	1.3386	0.1099	0.5963	0.057*
C14	1.1658 (7)	0.1747 (6)	0.6271 (3)	0.0483 (17)
H14	1.2199	0.1873	0.6611	0.058*
C15	1.0173 (7)	0.2017 (5)	0.6160 (3)	0.0417 (15)
H15	0.9691	0.2316	0.6429	0.050*
C16	1.0565 (8)	0.4803 (6)	0.7029 (3)	0.0546 (18)
H16	1.0176	0.4199	0.6817	0.066*
C17	1.2082 (8)	0.4842 (8)	0.7233 (3)	0.068 (2)
H17	1.2722	0.4269	0.7152	0.082*
C18	1.2655 (8)	0.5694 (8)	0.7546 (3)	0.061 (2)
H18	1.3684	0.5705	0.7688	0.073*
C19	1.1746 (8)	0.6530 (8)	0.7653 (3)	0.067 (2)
H19	1.2137	0.7121	0.7873	0.080*
C20	1.0220 (7)	0.6511 (7)	0.7435 (3)	0.064 (2)
H20	0.9597	0.7108	0.7499	0.076*
C21	0.9625 (6)	0.5643 (5)	0.7134 (2)	0.0352 (14)
C22	0.8030 (6)	0.5595 (5)	0.6885 (2)	0.0326 (14)
C23	0.7037 (7)	0.6423 (5)	0.6961 (2)	0.0348 (14)
H23	0.7374	0.6988	0.7206	0.042*
C24	0.5560 (6)	0.6478 (5)	0.6700 (2)	0.0313 (13)
C25	0.4577 (6)	0.7408 (5)	0.6784 (2)	0.0309 (12)
C26	0.4787 (7)	0.8051 (5)	0.7236 (2)	0.0416 (15)
H26	0.5600	0.7897	0.7505	0.050*
C27	0.3832 (8)	0.8908 (5)	0.7299 (3)	0.0504 (18)
H27	0.3987	0.9332	0.7611	0.060*
C28	0.2655 (8)	0.9151 (6)	0.6909 (3)	0.0522 (17)
H28	0.2001	0.9742	0.6954	0.063*
C29	0.2423 (7)	0.8526 (6)	0.6448 (3)	0.0452 (16)
H29	0.1625	0.8697	0.6177	0.054*
C30	0.3369 (7)	0.7660 (5)	0.6394 (2)	0.0374 (14)
H30	0.3197	0.7224	0.6085	0.045*
C31	0.1778 (7)	0.4503 (6)	0.5666 (3)	0.054 (2)

H31A	0.1922	0.4165	0.6018	0.065*
H31B	0.1316	0.3944	0.5412	0.065*
C32	0.0755 (8)	0.5455 (7)	0.5659 (4)	0.069 (2)
H32A	−0.0198	0.5207	0.5750	0.083*
H32B	0.0586	0.5782	0.5308	0.083*
H32C	0.1199	0.6006	0.5913	0.083*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
U1	0.02771 (11)	0.03283 (12)	0.03087 (11)	0.00087 (12)	0.00310 (8)	−0.00756 (11)
O1	0.032 (2)	0.051 (3)	0.040 (2)	−0.004 (2)	0.002 (2)	−0.006 (2)
O2	0.032 (2)	0.046 (3)	0.042 (2)	−0.002 (2)	0.001 (2)	0.002 (2)
O3	0.034 (2)	0.040 (3)	0.042 (2)	−0.0005 (19)	−0.004 (2)	−0.014 (2)
O4	0.042 (2)	0.039 (3)	0.032 (2)	0.007 (2)	0.001 (2)	−0.0085 (19)
O5	0.030 (2)	0.050 (3)	0.045 (3)	−0.001 (2)	0.002 (2)	−0.018 (2)
O6	0.039 (2)	0.048 (3)	0.058 (3)	0.006 (2)	0.001 (2)	−0.023 (2)
O7	0.034 (2)	0.046 (3)	0.032 (2)	0.006 (2)	0.0067 (19)	−0.0010 (19)
C1	0.039 (3)	0.034 (4)	0.042 (3)	−0.010 (3)	0.004 (3)	−0.005 (3)
C2	0.043 (4)	0.044 (4)	0.047 (4)	−0.004 (3)	0.002 (3)	0.004 (3)
C3	0.044 (3)	0.058 (4)	0.037 (3)	−0.018 (4)	0.000 (3)	−0.006 (4)
C4	0.050 (4)	0.047 (4)	0.036 (3)	−0.010 (3)	−0.001 (3)	−0.008 (3)
C5	0.044 (3)	0.036 (4)	0.034 (3)	−0.002 (3)	0.000 (3)	−0.004 (3)
C6	0.036 (3)	0.031 (3)	0.025 (3)	−0.008 (3)	0.002 (3)	−0.002 (2)
C7	0.034 (3)	0.029 (3)	0.035 (3)	−0.002 (3)	0.005 (3)	−0.002 (3)
C8	0.029 (3)	0.041 (4)	0.028 (3)	0.003 (3)	0.002 (3)	−0.006 (3)
C9	0.033 (3)	0.024 (3)	0.041 (3)	0.000 (2)	0.006 (3)	0.003 (3)
C10	0.034 (3)	0.027 (3)	0.035 (3)	−0.002 (3)	0.010 (3)	0.003 (3)
C11	0.043 (3)	0.034 (4)	0.041 (4)	−0.002 (3)	0.010 (3)	−0.001 (3)
C12	0.034 (3)	0.049 (5)	0.059 (4)	0.006 (3)	0.020 (4)	0.002 (3)
C13	0.037 (3)	0.040 (4)	0.065 (5)	0.005 (3)	0.009 (4)	0.000 (3)
C14	0.040 (4)	0.049 (4)	0.050 (4)	0.007 (3)	−0.013 (3)	−0.003 (3)
C15	0.039 (3)	0.041 (4)	0.042 (4)	0.006 (3)	0.000 (3)	−0.005 (3)
C16	0.045 (4)	0.051 (5)	0.061 (5)	0.002 (4)	−0.013 (4)	0.000 (4)
C17	0.047 (4)	0.074 (6)	0.080 (6)	0.006 (4)	−0.004 (4)	0.005 (5)
C18	0.039 (4)	0.097 (7)	0.045 (4)	−0.018 (5)	0.000 (4)	0.009 (4)
C19	0.043 (4)	0.105 (7)	0.052 (5)	−0.031 (5)	0.008 (4)	−0.022 (5)
C20	0.033 (4)	0.088 (6)	0.071 (6)	−0.015 (4)	0.014 (4)	−0.037 (5)
C21	0.032 (3)	0.047 (4)	0.027 (3)	−0.010 (3)	0.004 (3)	0.000 (3)
C22	0.035 (3)	0.037 (4)	0.029 (3)	−0.005 (3)	0.014 (3)	0.000 (2)
C23	0.040 (3)	0.033 (3)	0.032 (3)	−0.011 (3)	0.008 (3)	−0.007 (3)
C24	0.035 (3)	0.031 (3)	0.031 (3)	−0.006 (3)	0.018 (3)	−0.002 (2)
C25	0.038 (3)	0.027 (3)	0.030 (3)	−0.005 (3)	0.011 (3)	−0.004 (2)
C26	0.049 (4)	0.043 (4)	0.032 (3)	0.003 (3)	0.003 (3)	−0.008 (3)
C27	0.069 (5)	0.040 (4)	0.042 (4)	0.010 (3)	0.010 (4)	−0.014 (3)
C28	0.068 (5)	0.041 (4)	0.048 (4)	0.017 (4)	0.011 (4)	−0.003 (4)
C29	0.044 (4)	0.051 (4)	0.041 (4)	0.005 (3)	0.006 (3)	0.003 (3)
C30	0.045 (3)	0.036 (4)	0.033 (3)	0.002 (3)	0.008 (3)	−0.006 (3)

C31	0.035 (3)	0.068 (5)	0.057 (5)	−0.005 (3)	0.000 (4)	0.006 (4)
C32	0.042 (4)	0.082 (6)	0.084 (6)	0.009 (4)	0.009 (4)	−0.016 (5)

Geometric parameters (Å, °)

U1—O1	1.762 (4)	C14—C15	1.374 (9)
U1—O2	1.787 (4)	C14—H14	0.9500
U1—O4	2.288 (4)	C15—H15	0.9500
U1—O6	2.339 (4)	C16—C21	1.384 (9)
U1—O5	2.347 (4)	C16—C17	1.396 (10)
U1—O3	2.365 (4)	C16—H16	0.9500
U1—O7	2.464 (4)	C17—C18	1.365 (11)
O3—C7	1.278 (6)	C17—H17	0.9500
O4—C9	1.288 (6)	C18—C19	1.365 (11)
O5—C22	1.276 (7)	C18—H18	0.9500
O6—C24	1.253 (7)	C19—C20	1.411 (10)
O7—C31	1.440 (7)	C19—H19	0.9500
O7—H6	0.8596	C20—C21	1.370 (9)
C1—C2	1.362 (9)	C20—H20	0.9500
C1—C6	1.395 (8)	C21—C22	1.492 (8)
C1—H1	0.9500	C22—C23	1.385 (8)
C2—C3	1.383 (9)	C23—C24	1.405 (8)
C2—H2	0.9500	C23—H23	0.9500
C3—C4	1.368 (9)	C24—C25	1.477 (8)
C3—H3	0.9500	C25—C26	1.395 (8)
C4—C5	1.395 (9)	C25—C30	1.403 (8)
C4—H4	0.9500	C26—C27	1.381 (8)
C5—C6	1.403 (8)	C26—H26	0.9500
C5—H5	0.9500	C27—C28	1.380 (10)
C6—C7	1.492 (8)	C27—H27	0.9500
C7—C8	1.411 (8)	C28—C29	1.401 (9)
C8—C9	1.392 (8)	C28—H28	0.9500
C8—H8	0.9500	C29—C30	1.380 (8)
C9—C10	1.486 (8)	C29—H29	0.9500
C10—C11	1.386 (8)	C30—H30	0.9500
C10—C15	1.408 (9)	C31—C32	1.481 (10)
C11—C12	1.394 (9)	C31—H31A	0.9900
C11—H11	0.9500	C31—H31B	0.9900
C12—C13	1.368 (10)	C32—H32A	0.9800
C12—H12	0.9500	C32—H32B	0.9800
C13—C14	1.383 (9)	C32—H32C	0.9800
C13—H13	0.9500		
O1—U1—O2	178.36 (19)	C12—C13—H13	119.8
O1—U1—O4	89.86 (18)	C14—C13—H13	119.8
O2—U1—O4	90.00 (17)	C15—C14—C13	119.8 (7)
O1—U1—O6	88.42 (19)	C15—C14—H14	120.1
O2—U1—O6	92.55 (19)	C13—C14—H14	120.1
O4—U1—O6	149.05 (15)	C14—C15—C10	121.2 (6)
O1—U1—O5	93.46 (18)	C14—C15—H15	119.4

O2—U1—O5	88.12 (18)	C10—C15—H15	119.4
O4—U1—O5	79.09 (15)	C21—C16—C17	120.3 (7)
O6—U1—O5	70.19 (15)	C21—C16—H16	119.9
O1—U1—O3	90.71 (18)	C17—C16—H16	119.9
O2—U1—O3	87.70 (18)	C18—C17—C16	120.6 (8)
O4—U1—O3	70.35 (14)	C18—C17—H17	119.7
O6—U1—O3	140.55 (15)	C16—C17—H17	119.7
O5—U1—O3	149.14 (14)	C17—C18—C19	119.9 (7)
O1—U1—O7	91.33 (17)	C17—C18—H18	120.0
O2—U1—O7	87.75 (16)	C19—C18—H18	120.0
O4—U1—O7	140.97 (14)	C18—C19—C20	119.6 (7)
O6—U1—O7	69.97 (15)	C18—C19—H19	120.2
O5—U1—O7	139.70 (14)	C20—C19—H19	120.2
O3—U1—O7	70.62 (14)	C21—C20—C19	120.9 (7)
C7—O3—U1	134.5 (4)	C21—C20—H20	119.5
C9—O4—U1	134.1 (4)	C19—C20—H20	119.5
C22—O5—U1	138.5 (4)	C20—C21—C16	118.6 (6)
C24—O6—U1	139.8 (4)	C20—C21—C22	123.0 (6)
C31—O7—U1	126.5 (4)	C16—C21—C22	118.3 (6)
C31—O7—H6	120.6	O5—C22—C23	122.9 (6)
U1—O7—H6	109.3	O5—C22—C21	115.7 (5)
C2—C1—C6	121.2 (6)	C23—C22—C21	121.4 (5)
C2—C1—H1	119.4	C22—C23—C24	124.0 (6)
C6—C1—H1	119.4	C22—C23—H23	118.0
C1—C2—C3	119.9 (7)	C24—C23—H23	118.0
C1—C2—H2	120.0	O6—C24—C23	123.3 (5)
C3—C2—H2	120.0	O6—C24—C25	115.5 (5)
C4—C3—C2	120.6 (6)	C23—C24—C25	121.2 (5)
C4—C3—H3	119.7	C26—C25—C30	117.9 (6)
C2—C3—H3	119.7	C26—C25—C24	123.1 (6)
C3—C4—C5	120.2 (6)	C30—C25—C24	118.9 (5)
C3—C4—H4	119.9	C27—C26—C25	121.1 (6)
C5—C4—H4	119.9	C27—C26—H26	119.4
C4—C5—C6	119.5 (6)	C25—C26—H26	119.4
C4—C5—H5	120.2	C28—C27—C26	120.2 (6)
C6—C5—H5	120.2	C28—C27—H27	119.9
C1—C6—C5	118.5 (6)	C26—C27—H27	119.9
C1—C6—C7	119.7 (5)	C27—C28—C29	120.0 (6)
C5—C6—C7	121.7 (5)	C27—C28—H28	120.0
O3—C7—C8	123.5 (5)	C29—C28—H28	120.0
O3—C7—C6	115.6 (5)	C30—C29—C28	119.3 (6)
C8—C7—C6	120.9 (5)	C30—C29—H29	120.4
C9—C8—C7	122.7 (5)	C28—C29—H29	120.4
C9—C8—H8	118.6	C29—C30—C25	121.5 (6)
C7—C8—H8	118.6	C29—C30—H30	119.3
O4—C9—C8	122.9 (5)	C25—C30—H30	119.3
O4—C9—C10	115.6 (5)	O7—C31—C32	112.6 (6)
C8—C9—C10	121.6 (5)	O7—C31—H31A	109.1
C11—C10—C15	117.6 (6)	C32—C31—H31A	109.1

C11—C10—C9	124.4 (6)	O7—C31—H31B	109.1
C15—C10—C9	118.0 (5)	C32—C31—H31B	109.1
C10—C11—C12	121.1 (7)	H31A—C31—H31B	107.8
C10—C11—H11	119.5	C31—C32—H32A	109.5
C12—C11—H11	119.5	C31—C32—H32B	109.5
C13—C12—C11	119.9 (6)	H32A—C32—H32B	109.5
C13—C12—H12	120.1	C31—C32—H32C	109.5
C11—C12—H12	120.1	H32A—C32—H32C	109.5
C12—C13—C14	120.4 (6)	H32B—C32—H32C	109.5
O1—U1—O3—C7	−115.6 (5)	C7—C8—C9—O4	−1.0 (9)
O2—U1—O3—C7	64.8 (5)	C7—C8—C9—C10	176.9 (5)
O4—U1—O3—C7	−26.0 (5)	O4—C9—C10—C11	−171.1 (5)
O6—U1—O3—C7	156.0 (5)	C8—C9—C10—C11	10.8 (9)
O5—U1—O3—C7	−17.7 (7)	O4—C9—C10—C15	7.3 (8)
O7—U1—O3—C7	153.2 (6)	C8—C9—C10—C15	−170.7 (6)
O1—U1—O4—C9	129.1 (5)	C15—C10—C11—C12	−1.0 (9)
O2—U1—O4—C9	−49.3 (5)	C9—C10—C11—C12	177.4 (6)
O6—U1—O4—C9	−144.2 (5)	C10—C11—C12—C13	0.4 (10)
O5—U1—O4—C9	−137.3 (5)	C11—C12—C13—C14	−0.2 (10)
O3—U1—O4—C9	38.3 (5)	C12—C13—C14—C15	0.6 (11)
O7—U1—O4—C9	37.2 (6)	C13—C14—C15—C10	−1.2 (10)
O1—U1—O5—C22	−99.8 (6)	C11—C10—C15—C14	1.4 (9)
O2—U1—O5—C22	80.7 (6)	C9—C10—C15—C14	−177.1 (6)
O4—U1—O5—C22	171.0 (6)	C21—C16—C17—C18	1.2 (12)
O6—U1—O5—C22	−12.7 (5)	C16—C17—C18—C19	−1.3 (12)
O3—U1—O5—C22	163.0 (5)	C17—C18—C19—C20	−0.6 (12)
O7—U1—O5—C22	−3.6 (7)	C18—C19—C20—C21	2.6 (13)
O1—U1—O6—C24	100.7 (7)	C19—C20—C21—C16	−2.7 (11)
O2—U1—O6—C24	−80.7 (7)	C19—C20—C21—C22	−178.5 (7)
O4—U1—O6—C24	13.6 (8)	C17—C16—C21—C20	0.8 (11)
O5—U1—O6—C24	6.4 (6)	C17—C16—C21—C22	176.8 (6)
O3—U1—O6—C24	−170.1 (6)	U1—O5—C22—C23	16.0 (9)
O7—U1—O6—C24	−167.3 (7)	U1—O5—C22—C21	−164.9 (4)
O1—U1—O7—C31	−2.7 (5)	C20—C21—C22—O5	178.6 (6)
O2—U1—O7—C31	175.9 (5)	C16—C21—C22—O5	2.7 (8)
O4—U1—O7—C31	88.7 (5)	C20—C21—C22—C23	−2.3 (9)
O6—U1—O7—C31	−90.6 (5)	C16—C21—C22—C23	−178.2 (6)
O5—U1—O7—C31	−99.7 (5)	O5—C22—C23—C24	−7.2 (9)
O3—U1—O7—C31	87.6 (5)	C21—C22—C23—C24	173.8 (5)
C6—C1—C2—C3	−0.1 (9)	U1—O6—C24—C23	−3.9 (10)
C1—C2—C3—C4	0.1 (10)	U1—O6—C24—C25	175.3 (4)
C2—C3—C4—C5	−0.2 (10)	C22—C23—C24—O6	1.6 (9)
C3—C4—C5—C6	0.3 (9)	C22—C23—C24—C25	−177.5 (5)
C2—C1—C6—C5	0.2 (9)	O6—C24—C25—C26	156.8 (6)
C2—C1—C6—C7	−176.9 (5)	C23—C24—C25—C26	−24.0 (8)
C4—C5—C6—C1	−0.3 (8)	O6—C24—C25—C30	−22.9 (8)
C4—C5—C6—C7	176.7 (5)	C23—C24—C25—C30	156.3 (5)
U1—O3—C7—C8	10.9 (9)	C30—C25—C26—C27	0.2 (9)
U1—O3—C7—C6	−169.8 (4)	C24—C25—C26—C27	−179.5 (6)

C1—C6—C7—O3	24.7 (8)	C25—C26—C27—C28	−0.7 (10)
C5—C6—C7—O3	−152.3 (5)	C26—C27—C28—C29	0.1 (11)
C1—C6—C7—C8	−156.1 (6)	C27—C28—C29—C30	1.1 (10)
C5—C6—C7—C8	26.9 (8)	C28—C29—C30—C25	−1.6 (10)
O3—C7—C8—C9	11.7 (10)	C26—C25—C30—C29	1.0 (9)
C6—C7—C8—C9	−167.5 (5)	C24—C25—C30—C29	−179.3 (5)
U1—O4—C9—C8	−34.8 (8)	U1—O7—C31—C32	137.6 (5)
U1—O4—C9—C10	147.1 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O7—H6···O2^{i}	0.86	1.94	2.765 (5)	162

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1.

Fig. 1

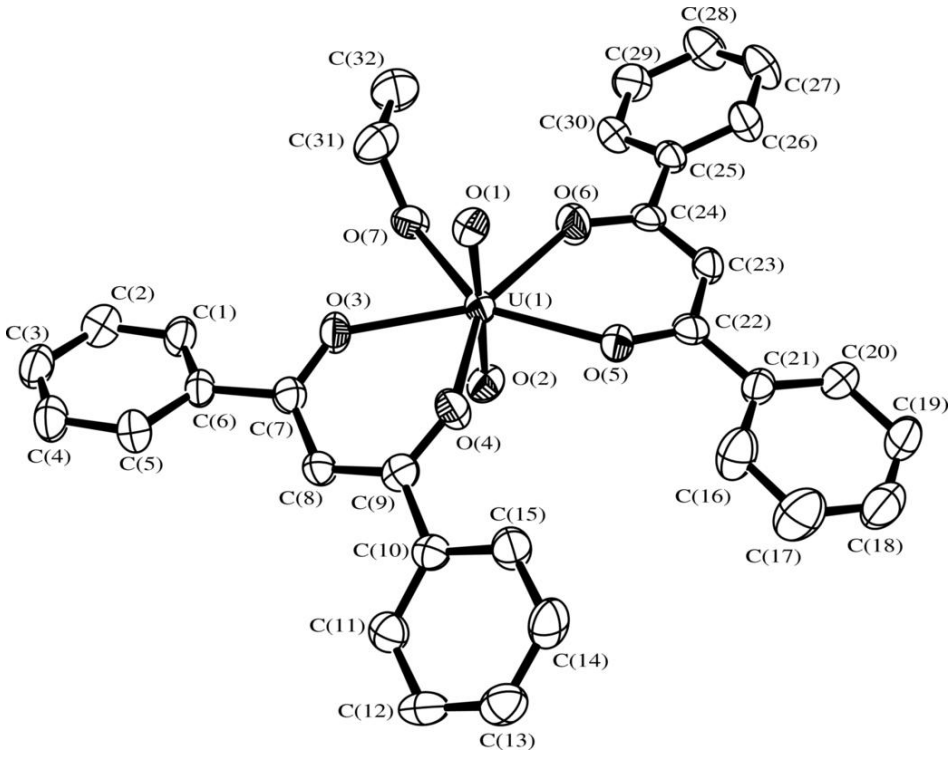
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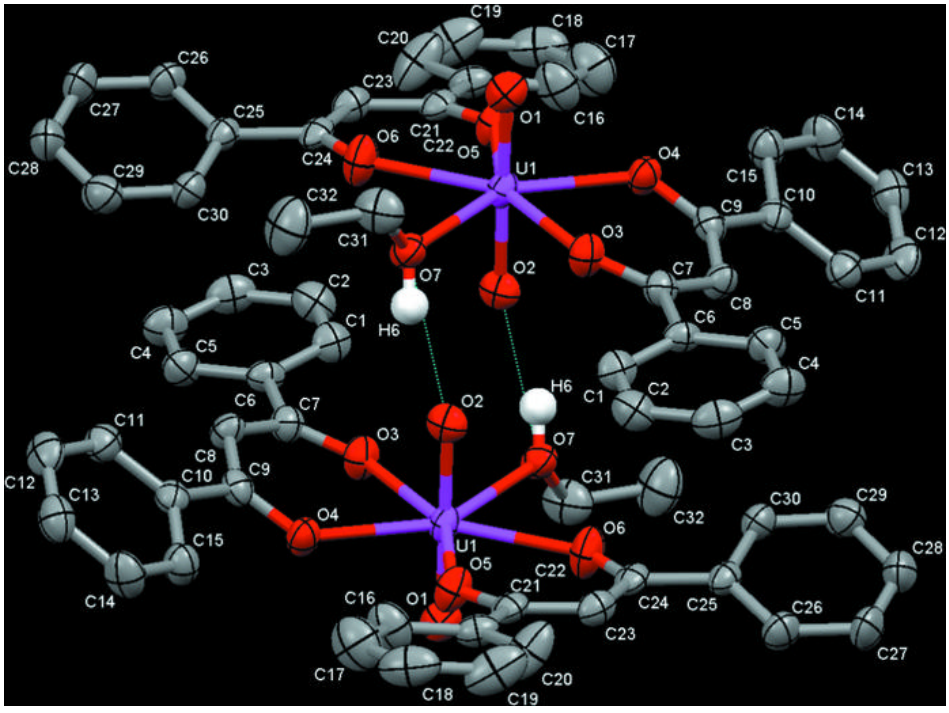
Fig. 2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