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Crystal structure and Hirshfeld surface analysis of bis(benzoylacetonato)(ethanol)dioxidouranium(VI)

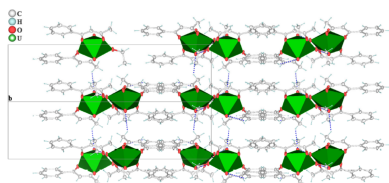
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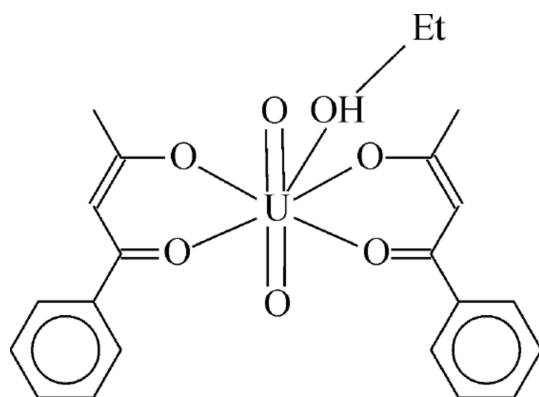
A new uranium metal–organic complex salt, $[\text{U}(\text{C}_{10}\text{H}_9\text{O}_2)_2\text{O}_2(\text{C}_2\text{H}_6\text{O})]$, with benzoyl acetone, namely, bis(benzoylacetonato)(ethanol)dioxidouranium(VI), was synthesized. The compound has monoclinic $P2_1/n$ symmetry. The geometry of the seven-coordinate U atom is pentagonal bipyramidal, with the uranyl oxygen atoms in apical positions. In the complex, the ligands bind to the metal through oxygen atoms. Additional weak $\text{O} \cdots \text{H} \cdots \text{O}$ contacts between the cations and anions consolidate the three-dimensional arrangement of the structure. On the Hirshfeld surface, the largest contributions come from the short contacts such as van der Waals forces, including $\text{H} \cdots \text{H}$, $\text{O} \cdots \text{H}$ and $\text{C} \cdots \text{H}$. Interactions including $\text{C} \cdots \text{C}$ and $\text{O} \cdots \text{C}$ contacts were also observed; however, their contribution to the overall cohesion of the crystal structure is minor. A packing analysis was performed to check the strength of the crystal packing.

1. Chemical context

A greater understanding of the coordination chemistry of uranium is important for the development of new technologies for the safe reprocessing and long-term immobilization of irradiated nuclear fuel. One of the main reasons for the renewed interest in uranium compounds is their remarkable structural versatility. In the oxidation states +III or +IV, eight- or nine-coordinate uranium environments are typically found, similar to those observed in lanthanide complexes (Enriquez *et al.*, 2005; Oldham *et al.* 2002). Uranium oxo compounds with oxidation state +VI form approximately linear triatomic uranyl ions, UO_2^{2+} . Although this cation can bind additional ligands perpendicular to the uranium axis to form five-, six-, seven-, and eight-coordinate metal centres, seven-coordinate is particularly common for hexavalent uranium oxo compounds (Hernandez *et al.*, 2022; Almond & Albrecht-Schmitt, 2003; Arndt *et al.* 2002). Heptacoordinated uranium centers can exhibit pentagonal–bipyramidal, capped-octahedral, and trigonal–prismatic coordination geometries. The specific geometry depends on steric requirements caused by ligand–ligand repulsion, weaker bonds, and generally reduced crystal field stability. Despite the abundance of layered structures for U^{VI} -oxo compounds (Chakraborty *et al.*, 2006; Hughes & Burns, 2003; Neu *et al.*, 2001), one-dimensional topology or multidimensional framework structural studies of



uranyl compounds are rather sparse (Bean *et al.*, 2001; Sykora & Albrecht-Schmitt, 2003). The anti-inflammatory, analgesic, anti-microbial, anti-convulsant, anti-cancer, anti-tubercular, antioxidant, antidepressant, antiglycation, antihelminthic, antifungal, anti-tumour, antibiotic and anti-allergic effects of the ligand have been studied (Şahin & Dege, 2021).



The present work was undertaken to study the effect of oxo-ligands on the metal coordination geometry and explore the possibility of any supramolecular architecture in the resulting uranyl compounds. We isolated the title metal–organic

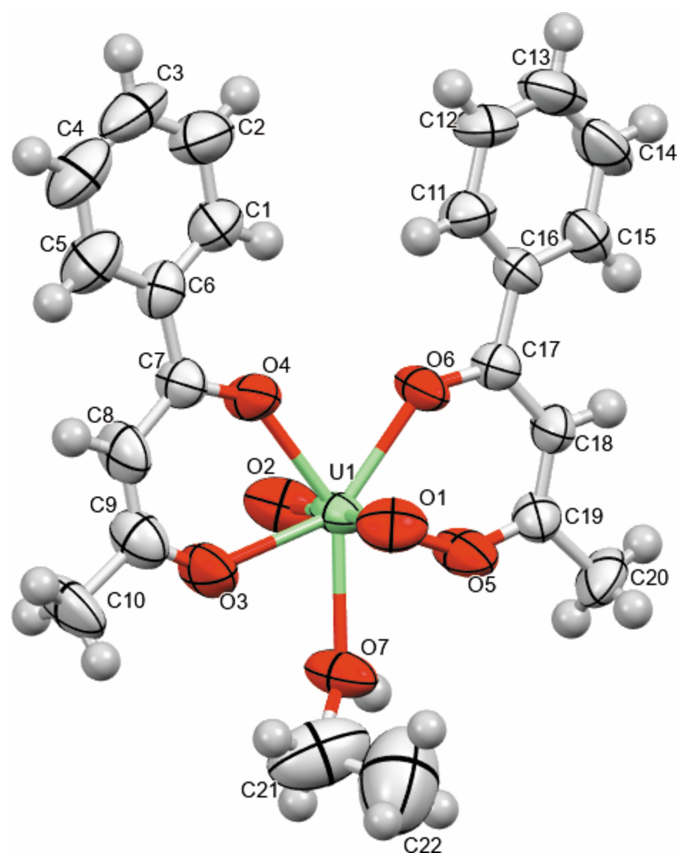


Figure 1
The molecular structure of title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are displayed as small spheres of arbitrary radii.

Table 1
Selected bond lengths (Å).

U1—O6	2.317 (5)	U1—O1	1.759 (7)
U1—O7	2.458 (5)	U1—O3	2.358 (7)
U1—O4	2.308 (5)	U1—O5	2.369 (7)
U1—O2	1.779 (8)		

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>
O7—H7···O2 ⁱ	0.86 (6)	2.44 (5)	3.246 (10)	158 (3)

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

complex uranium salt, [C₂₂H₂₄O₇U] and report here its crystal structure and Hirshfeld surface analysis.

2. Structural commentary

The single-crystal structure of bis(benzoylacetonato)(ethanol)dioxidouranium(VI) crystallizes in the monoclinic space group *P*2₁/*n*. The molecular structure is shown in Fig. 1. The molecule is almost planar with an r.m.s. deviation of 0.0593 Å from planarity. In the compound, the coordination geometry around the uranium atom includes seven oxygen donors from one ethanol, two oxido, and two bidentate benzoylacetonato ligands. It is approximately pentagonal bipyramidal. The U—O uranyl bond distances [1.759 (7) and 2.358 (7) Å; Table 1] agree well with the previously reported values for dioxouranium (VI) complexes (Hernandez *et al.*, 2022; Takao & Ikeda, 2008; Chakraborty *et al.*, 2006; Gatto *et al.*, 2004; Kannan *et al.*, 2004). The distortion of the metal coordination geometry from an ideal pentagonal bipyramidal arrangement (Fig. 2) is revealed by the O—U—O bond angles for the pentagon, which range between 70.6 (2) and 177.4 (3)°.

3. Supramolecular features

In the complex, the crystal packing exhibits one intermolecular O7—H7···O2(1 - *x*, 1 - *y*, 1 - *z*) hydrogen-bonding interactions (Fig. 3, Table 2). In additional π – π stacking (Fig. 3) occurs between the aromatic rings of neighbouring molecules with centroid–centroid distances Cg1···Cg2(–1 + *x*, *y*, *z*) = 3.900 (6) Å, with a ring slippage of

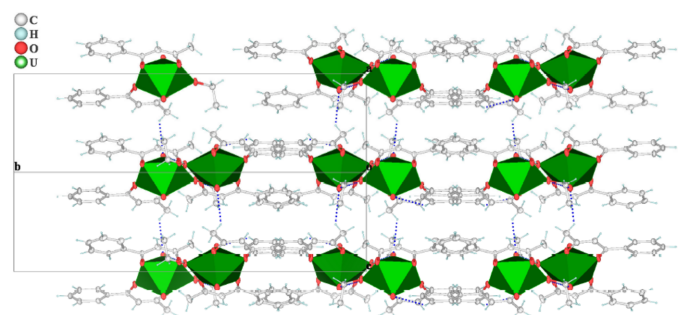


Figure 2
A view of the molecular packing showing the pentagonal-bipyramidal structure extending along the *b*-axis direction.

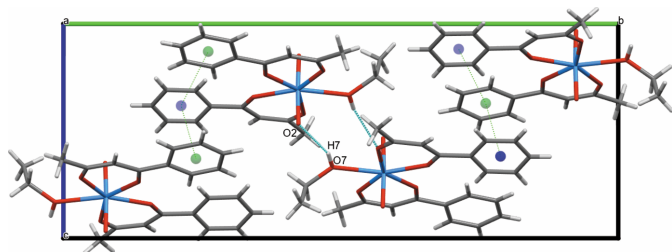


Figure 3
View of the crystal structure of the title compound, showing the O–H...O hydrogen bond and π – π interactions as green dotted lines.

1.577 Å, and $Cg2 \cdots Cg1(\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z) = 3.765$ (6) Å, with a ring slippage of 1.035 Å where Cg1 and Cg2 are the centroids of the C1–C6 and C11–C16 rings, respectively.

4. Hirshfeld surface analysis

To further investigate the intermolecular interactions present in the title compound, a Hirshfeld surface (HS) analysis was performed, and the two-dimensional fingerprint plots were generated with *CrystalExplorer17.5* (Spackman *et al.*, 2021). The HS mapped with d_{norm} , curviness and shape-index are given in Fig. 4. The white surface indicates contacts with distances equal to the sum of van der Waals radii, and the red and blue colours indicate distances shorter or longer than the van der Waals radii, respectively. The bright-red points in the d_{norm} surface of the molecule are located near atoms O2 and O7/H7, consistent with the O7–H7...O2 hydrogen-bonding interaction, highlighted in Fig. 3. From the Hirshfeld surfaces, it is also evident that the molecules are related to one another by π – π stacking interactions, as can be inferred from inspection of the adjacent red and blue triangles (highlighted by yellow circles) on the shape-index surface (Fig. 4). The presence of π – π stacking is also evident in the flat region toward the top of both sides of the molecules and is clearly visible on the curviness surface (Fig. 4): the shape of the blue outline on the curviness surface unambiguously delineates the contacting patches of the molecules.

The two-dimensional (2D) fingerprint plots (McKinnon *et al.*, 2007) are shown in Fig. 5. On the HS, the largest contributions (53.2%, 23.4%, 13.8%) come from short contacts such as van der Waals forces, H...H, O...H and C...H contacts. C...C (8.6%), O...C (0.8%) and O...O (0.1%) contacts are also observed. The classical O–H...O hydrogen bonds

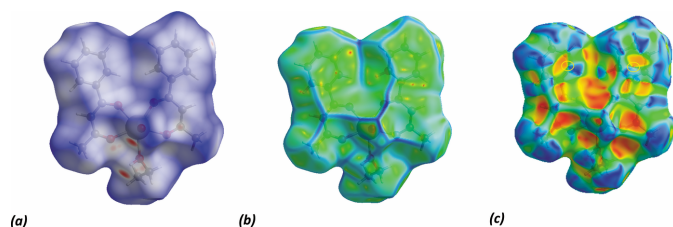


Figure 4
Hirshfeld surfaces of the title complex mapped with (a) d_{norm} , (b) curviness and (c) shape-index.

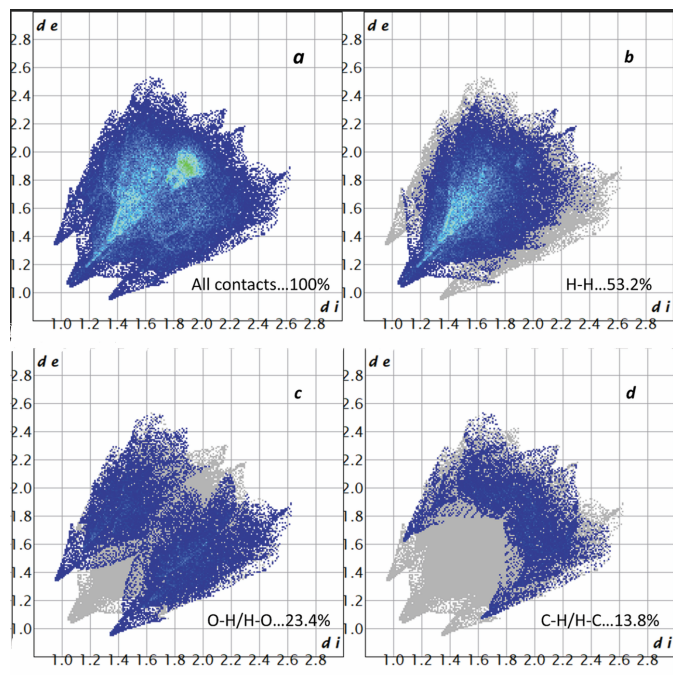


Figure 5
Two-dimensional fingerprint plots for the title compound, showing (a) all interactions, and decomposed into (b) H...H, (c) O...H/H...O and (d) C...H/H...C interactions.

correspond to O...H/H...O contacts (23.4% contribution) in Fig. 5 and show up as a pair of spikes. The scattered points in the breakdown of the fingerprint plot show that the π – π stacking interactions C...C comprise 8.6% of the total Hirshfeld surface of the molecule displayed as a region of blue/green colour.

5. Database survey

A search in the Cambridge Structural Database (CSD, version 5.43, update of November 2022; Groom *et al.*, 2016) revealed 25 hits with the β -diketonate ligand moiety. Among these, one structure contains tin (AGESUA: Pettinari *et al.*, 2002), two structures contain zinc (BZACZN: Belford *et al.*, 1969; NEYBID: Dang *et al.*, 2006), one structure contains uranium(VI) (CAZMEV: Haider *et al.*, 1983), one structure contains platinum(II) (CBZACP: Okeya *et al.*, 1976), one structure contains iron(III) (ARUMOR: Zou *et al.*, 2016), one structure contains manganese(II), one structure contains cadmium (HICRAP and HICRET: Yang, 2018*a,b*), one structure contains vanadium (KIJPAV: Xing *et al.*, 2007), four structures contain copper (CUBEAC: Hon *et al.*, 1966; LEZVAO: Lennartson *et al.*, 2007; NINFIC, NINFOI: Chen *et al.*, 2018), one structure contains lithium (UCIMAU: Jung *et al.*, 1998), nine structures contain manganese(II) (NENNAX: Cvrtila *et al.*, 2012; PIDPOJ, PIDPUP, PIDQAW, PIDQEA, PIDQIE, PIDQOK, PIDQUQ, PIDRAX: Cvrtila *et al.*, 2013), and two structures contain cobalt(II) (POJBUN: Perdih, 2014; YADKUJ: Döring *et al.*, 1992). A search for the uranyl moiety returned five hits with pentagonal–bipyramidal geometries similar to that in the title structure. These include: aqua-

Table 3
Experimental details.

Crystal data	
Chemical formula	[U(C ₁₀ H ₉ O ₂) ₂ O ₂ (C ₂ H ₆ O)]
<i>M_r</i>	638.44
Crystal system, space group	Monoclinic, <i>P2₁/n</i>
Temperature (K)	293
<i>a</i> , <i>b</i> , <i>c</i> (Å)	8.55214 (16), 26.0026 (4), 10.3057 (2)
β (°)	102.8291 (19)
<i>V</i> (Å ³)	2234.56 (7)
<i>Z</i>	4
Radiation type	Cu <i>K</i> α
μ (mm ⁻¹)	20.79
Crystal size (mm)	0.28 × 0.26 × 0.18
Data collection	
Diffraction	XtaLAB Synergy, Single source at home/near, HyPix3000
Absorption correction	Multi-scan (<i>CrysAlis PRO</i> ; Rigaku OD, 2020)
<i>T_{min}</i> , <i>T_{max}</i>	0.266, 1.000
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	22840, 4331, 3260
<i>R_{int}</i>	0.085
(sin θ/λ) _{max} (Å ⁻¹)	0.615
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.041, 0.115, 1.05
No. of reflections	4331
No. of parameters	277
No. of restraints	4
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	1.49, -1.95

Computer programs: *CrysAlis PRO* (Rigaku OD, 2020), *SHELXT2018/2* (Sheldrick, 2015a), *SHELXL2019/3* (Sheldrick, 2015b), *PLATON* (Spek, 2020) and *publCIF* (Westrip, 2010).

bis(benzoylacetato)dioxouranium(VI) monohydrate (CAZMEV: Haider *et al.*, 1983), uranyl(VI) complexes containing the β-diketonatephenol ligands derived from 1-(2-hydroxyphenyl)-1,3-butanedione and 1-(2-hydroxyphenyl)-3-phenyl-1,3-propanedione (GIYXAN, GIYXER: Ainscough *et al.*, 1998), a uranyl β-diketonate complex [UO₂(tfa)₂(*L*)] [*L* = H₂O, OHCH₂CH₃; tfa = deprotonated 4,4,4-trifluoro-1-(2-furyl)-1,3-butanedione] with a well-described 3D supra-molecular structure and electronic absorption spectroscopy (IVEDIX: Al-Anber *et al.*, 2011), and bis(2-benzoyl-1-phenylethenolato-κ²O,O')(ethanol-κO)dioxidouranium(VI) (RISVAR: Takao & Ikeda, 2008).

6. Synthesis and crystallization

Benzoylacetone (BNA) (0.0324 g, 0.200 mmol) dissolved in 5 ml of ethanol and uranyl acetate (0.0388 g, 0.100 mmol) dissolved in 5 ml of ethanol were mixed under constant stirring until the colour of the solution turned to orange–red. The stirring continued for an hour, then the solution was left to stand overnight. The orange–red crystalline solid was filtered off and dried under vacuum. The solid was dissolved in ethanol and slow evaporation of the solution yielded diffraction-quality single-crystals of the title compound. Selected IR bands (KBr pellet, cm⁻¹): 1589 (C=O), 1340 (C–O), 471

(U–O_{ligand}), 380 (U–O_{eth} Raman spectroscopy), 908 (O=U=O).

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. C-bound H atoms were positioned geometrically (C–H = 0.93–0.97 Å) and treated as riding on their parent atoms, with C–H = 0.95 Å (aromatic) and were refined with *U*_{iso}(H) = 1.2*U*_{eq}(C).

Acknowledgements

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Computing details

Bis(benzoylacetato)(ethanol)dioxidouranium(VI)

Crystal data

$[\text{U}(\text{C}_{10}\text{H}_9\text{O}_2)_2\text{O}_2(\text{C}_2\text{H}_6\text{O})]$

$M_r = 638.44$

Monoclinic, $P2_1/n$

$a = 8.55214$ (16) Å

$b = 26.0026$ (4) Å

$c = 10.3057$ (2) Å

$\beta = 102.8291$ (19)°

$V = 2234.56$ (7) Å³

$Z = 4$

$F(000) = 1216$

$D_x = 1.898$ Mg m⁻³

Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å

Cell parameters from 7001 reflections

$\theta = 3.4\text{--}70.9^\circ$

$\mu = 20.79$ mm⁻¹

$T = 293$ K

Block, orange

$0.28 \times 0.26 \times 0.18$ mm

Data collection

XtaLAB Synergy, Single source at home/near,

HyPix3000

diffractometer

Radiation source: micro-focus sealed X-ray

tube, PhotonJet (Cu) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm⁻¹

ω scans

Absorption correction: multi-scan

(CrysAlisPro; Rigaku OD, 2020)

$T_{\min} = 0.266$, $T_{\max} = 1.000$

22840 measured reflections

4331 independent reflections

3260 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.085$

$\theta_{\max} = 71.6^\circ$, $\theta_{\min} = 3.4^\circ$

$h = -10 \rightarrow 10$

$k = -31 \rightarrow 31$

$l = -12 \rightarrow 12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.041$

$wR(F^2) = 0.115$

$S = 1.05$

4331 reflections

277 parameters

4 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent

and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0559P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 1.49$ e Å⁻³

$\Delta\rho_{\min} = -1.94$ e Å⁻³

Special details

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

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
U1	0.35139 (3)	0.42260 (2)	0.30596 (3)	0.06079 (13)
O6	0.4734 (6)	0.3427 (2)	0.3413 (7)	0.0814 (19)
O7	0.4126 (8)	0.5149 (2)	0.3315 (7)	0.089 (2)
H7	0.492 (7)	0.5227 (10)	0.395 (6)	0.133*
O4	0.1461 (6)	0.3644 (2)	0.2364 (7)	0.0834 (18)
O2	0.3043 (11)	0.4237 (2)	0.4656 (7)	0.097 (2)
O1	0.4002 (10)	0.4245 (2)	0.1490 (7)	0.086 (2)
O3	0.1132 (8)	0.4682 (3)	0.2177 (7)	0.094 (2)
O5	0.6224 (8)	0.4331 (2)	0.4195 (8)	0.109 (3)
C17	0.6112 (8)	0.3228 (3)	0.3875 (7)	0.0546 (17)
C16	0.6196 (9)	0.2656 (3)	0.3843 (7)	0.0553 (17)
C19	0.7469 (10)	0.4058 (3)	0.4512 (8)	0.0612 (19)
C18	0.7443 (8)	0.3533 (3)	0.4370 (8)	0.065 (2)
H18	0.842014	0.336419	0.463399	0.077*
C6	-0.0712 (9)	0.3112 (3)	0.1567 (8)	0.065 (2)
C7	0.0003 (9)	0.3631 (3)	0.1833 (8)	0.063 (2)
C11	0.4925 (11)	0.2381 (3)	0.3120 (9)	0.073 (2)
H11	0.402282	0.255445	0.265396	0.088*
C9	-0.0310 (12)	0.4571 (4)	0.1714 (10)	0.083 (3)
C20	0.8965 (10)	0.4343 (4)	0.5093 (11)	0.088 (3)
H20A	0.939075	0.449118	0.439123	0.132*
H20B	0.973720	0.411031	0.560045	0.132*
H20C	0.873495	0.461083	0.566368	0.132*
C15	0.7523 (10)	0.2390 (3)	0.4547 (9)	0.074 (2)
H15	0.840231	0.256908	0.503191	0.088*
C8	-0.0906 (11)	0.4086 (4)	0.1504 (10)	0.084 (3)
H8	-0.199147	0.405010	0.111488	0.101*
C5	-0.2247 (11)	0.3018 (5)	0.0822 (10)	0.096 (3)
H5	-0.290652	0.329367	0.048809	0.116*
C12	0.4969 (13)	0.1855 (3)	0.3079 (12)	0.094 (3)
H12	0.411937	0.167293	0.255928	0.113*
C1	0.0190 (11)	0.2687 (4)	0.2006 (10)	0.080 (2)
H1	0.123451	0.273124	0.249479	0.096*
C14	0.7535 (13)	0.1862 (4)	0.4526 (11)	0.097 (3)
H14	0.841828	0.168385	0.500949	0.117*
C13	0.6271 (15)	0.1598 (4)	0.3807 (12)	0.096 (3)
H13	0.628639	0.123990	0.380680	0.116*
C4	-0.2814 (15)	0.2524 (6)	0.0566 (12)	0.110 (4)
H4	-0.384491	0.247047	0.005943	0.132*

C2	-0.0393 (14)	0.2198 (4)	0.1749 (12)	0.104 (3)
H2	0.025503	0.191739	0.206548	0.125*
C3	-0.1889 (15)	0.2122 (5)	0.1044 (12)	0.103 (4)
H3	-0.228585	0.178916	0.088717	0.123*
C10	-0.1408 (12)	0.5024 (4)	0.1353 (11)	0.115 (4)
H10A	-0.184658	0.511753	0.210066	0.172*
H10B	-0.226310	0.493619	0.061287	0.172*
H10C	-0.081541	0.530940	0.111446	0.172*
C21	0.369 (2)	0.5632 (6)	0.2438 (15)	0.164 (7)
H21A	0.264632	0.558775	0.184657	0.196*
H21B	0.364662	0.592990	0.299405	0.196*
C22	0.492 (2)	0.5711 (7)	0.165 (2)	0.226 (11)
H22A	0.454473	0.595840	0.095993	0.339*
H22B	0.513178	0.539042	0.125400	0.339*
H22C	0.589497	0.583392	0.221952	0.339*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
U1	0.0615 (2)	0.04146 (17)	0.0673 (2)	0.00267 (10)	-0.01179 (13)	-0.00574 (11)
O6	0.059 (3)	0.045 (3)	0.122 (5)	0.009 (2)	-0.018 (3)	-0.019 (3)
O7	0.111 (5)	0.051 (3)	0.085 (4)	0.005 (3)	-0.023 (3)	-0.006 (3)
O4	0.056 (3)	0.055 (3)	0.121 (5)	-0.006 (2)	-0.022 (3)	0.006 (3)
O2	0.143 (7)	0.072 (5)	0.064 (4)	0.009 (4)	-0.002 (4)	-0.002 (3)
O1	0.117 (6)	0.073 (4)	0.061 (4)	-0.010 (3)	0.009 (4)	-0.015 (3)
O3	0.075 (4)	0.068 (4)	0.122 (6)	0.016 (3)	-0.013 (4)	0.009 (4)
O5	0.074 (4)	0.056 (4)	0.166 (8)	0.002 (3)	-0.038 (4)	-0.030 (4)
C17	0.058 (4)	0.053 (4)	0.048 (4)	0.002 (3)	0.002 (3)	-0.001 (3)
C16	0.059 (4)	0.048 (4)	0.058 (4)	0.009 (3)	0.011 (3)	-0.001 (3)
C19	0.056 (5)	0.062 (5)	0.063 (5)	0.000 (4)	0.007 (4)	-0.014 (4)
C18	0.044 (4)	0.058 (5)	0.086 (6)	0.000 (3)	0.002 (4)	-0.007 (4)
C6	0.056 (5)	0.083 (6)	0.053 (4)	-0.004 (4)	0.009 (3)	-0.004 (4)
C7	0.058 (5)	0.061 (5)	0.063 (5)	0.001 (4)	-0.003 (4)	-0.002 (4)
C11	0.073 (5)	0.057 (5)	0.085 (6)	-0.008 (4)	0.007 (4)	-0.001 (4)
C9	0.082 (7)	0.080 (7)	0.077 (6)	0.017 (5)	-0.003 (5)	-0.003 (5)
C20	0.054 (5)	0.097 (7)	0.109 (8)	-0.025 (5)	0.013 (5)	-0.029 (6)
C15	0.068 (5)	0.066 (5)	0.086 (6)	0.017 (4)	0.013 (4)	0.005 (5)
C8	0.059 (5)	0.088 (7)	0.088 (7)	0.020 (5)	-0.019 (5)	-0.002 (6)
C5	0.078 (7)	0.114 (9)	0.090 (7)	-0.029 (6)	0.003 (5)	-0.003 (6)
C12	0.098 (7)	0.048 (5)	0.134 (9)	-0.016 (5)	0.022 (6)	-0.015 (6)
C1	0.069 (5)	0.074 (6)	0.093 (6)	-0.011 (5)	0.011 (5)	-0.010 (5)
C14	0.106 (8)	0.069 (7)	0.118 (9)	0.041 (6)	0.027 (7)	0.027 (6)
C13	0.134 (9)	0.047 (5)	0.119 (9)	0.012 (6)	0.050 (7)	0.012 (6)
C4	0.088 (8)	0.139 (11)	0.098 (8)	-0.050 (8)	0.009 (6)	-0.018 (8)
C2	0.107 (8)	0.078 (7)	0.132 (10)	-0.023 (6)	0.039 (7)	-0.011 (7)
C3	0.116 (9)	0.102 (9)	0.101 (9)	-0.061 (7)	0.049 (7)	-0.036 (7)
C10	0.102 (7)	0.086 (7)	0.132 (9)	0.051 (6)	-0.026 (7)	-0.011 (7)
C21	0.24 (2)	0.151 (14)	0.102 (11)	-0.078 (13)	0.038 (12)	-0.012 (10)

C22	0.154 (19)	0.30 (3)	0.23 (2)	-0.022 (15)	0.046 (17)	-0.070 (19)
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Geometric parameters (Å, °)

U1—O6	2.317 (5)	C20—H20B	0.9600
U1—O7	2.458 (5)	C20—H20C	0.9600
U1—O4	2.308 (5)	C15—H15	0.9300
U1—O2	1.779 (8)	C15—C14	1.374 (12)
U1—O1	1.759 (7)	C8—H8	0.9300
U1—O3	2.358 (7)	C5—H5	0.9300
U1—O5	2.369 (7)	C5—C4	1.376 (15)
O6—C17	1.279 (8)	C12—H12	0.9300
O7—H7	0.854 (10)	C12—C13	1.371 (14)
O7—C21	1.544 (15)	C1—H1	0.9300
O4—C7	1.246 (8)	C1—C2	1.371 (12)
O3—C9	1.253 (10)	C14—H14	0.9300
O5—C19	1.260 (10)	C14—C13	1.354 (14)
C17—C16	1.490 (10)	C13—H13	0.9300
C17—C18	1.389 (10)	C4—H4	0.9300
C16—C11	1.374 (11)	C4—C3	1.339 (16)
C16—C15	1.388 (10)	C2—H2	0.9300
C19—C18	1.373 (11)	C2—C3	1.338 (15)
C19—C20	1.484 (11)	C3—H3	0.9300
C18—H18	0.9300	C10—H10A	0.9600
C6—C7	1.481 (11)	C10—H10B	0.9600
C6—C5	1.388 (11)	C10—H10C	0.9600
C6—C1	1.366 (12)	C21—H21A	0.9700
C7—C8	1.414 (12)	C21—H21B	0.9700
C11—H11	0.9300	C21—C22	1.482 (9)
C11—C12	1.370 (12)	C22—H22A	0.9600
C9—C8	1.360 (14)	C22—H22B	0.9600
C9—C10	1.502 (12)	C22—H22C	0.9600
C20—H20A	0.9600		
O6—U1—O7	141.3 (2)	C19—C20—H20B	109.5
O6—U1—O3	146.3 (2)	C19—C20—H20C	109.5
O6—U1—O5	70.61 (19)	H20A—C20—H20B	109.5
O4—U1—O6	75.28 (18)	H20A—C20—H20C	109.5
O4—U1—O7	143.4 (2)	H20B—C20—H20C	109.5
O4—U1—O3	71.2 (2)	C16—C15—H15	120.1
O4—U1—O5	145.3 (2)	C14—C15—C16	119.8 (9)
O2—U1—O6	93.2 (3)	C14—C15—H15	120.1
O2—U1—O7	88.4 (3)	C7—C8—H8	117.6
O2—U1—O4	89.1 (3)	C9—C8—C7	124.8 (8)
O2—U1—O3	89.7 (3)	C9—C8—H8	117.6
O2—U1—O5	86.5 (4)	C6—C5—H5	119.3
O1—U1—O6	88.8 (3)	C4—C5—C6	121.4 (11)
O1—U1—O7	89.0 (2)	C4—C5—H5	119.3

O1—U1—O4	93.0 (3)	C11—C12—H12	120.2
O1—U1—O2	177.4 (3)	C11—C12—C13	119.7 (9)
O1—U1—O3	89.6 (3)	C13—C12—H12	120.2
O1—U1—O5	92.6 (3)	C6—C1—H1	118.9
O3—U1—O7	72.3 (2)	C6—C1—C2	122.2 (9)
O3—U1—O5	143.1 (2)	C2—C1—H1	118.9
O5—U1—O7	70.9 (2)	C15—C14—H14	119.7
C17—O6—U1	140.2 (5)	C13—C14—C15	120.5 (9)
U1—O7—H7	115.4 (16)	C13—C14—H14	119.7
C21—O7—U1	135.6 (7)	C12—C13—H13	119.8
C21—O7—H7	107.4 (17)	C14—C13—C12	120.3 (9)
C7—O4—U1	140.5 (6)	C14—C13—H13	119.8
C9—O3—U1	136.3 (6)	C5—C4—H4	119.9
C19—O5—U1	137.9 (5)	C3—C4—C5	120.3 (11)
O6—C17—C16	116.3 (6)	C3—C4—H4	119.9
O6—C17—C18	121.1 (7)	C1—C2—H2	119.8
C18—C17—C16	122.6 (7)	C3—C2—C1	120.3 (12)
C11—C16—C17	119.6 (7)	C3—C2—H2	119.8
C11—C16—C15	118.8 (7)	C4—C3—H3	120.0
C15—C16—C17	121.6 (7)	C2—C3—C4	120.0 (11)
O5—C19—C18	122.6 (8)	C2—C3—H3	120.0
O5—C19—C20	115.3 (8)	C9—C10—H10A	109.5
C18—C19—C20	122.0 (8)	C9—C10—H10B	109.5
C17—C18—H18	116.5	C9—C10—H10C	109.5
C19—C18—C17	126.9 (7)	H10A—C10—H10B	109.5
C19—C18—H18	116.5	H10A—C10—H10C	109.5
C5—C6—C7	124.4 (9)	H10B—C10—H10C	109.5
C1—C6—C7	119.7 (7)	O7—C21—H21A	109.9
C1—C6—C5	115.8 (9)	O7—C21—H21B	109.9
O4—C7—C6	116.0 (7)	H21A—C21—H21B	108.3
O4—C7—C8	121.8 (8)	C22—C21—O7	109.0 (17)
C8—C7—C6	122.3 (7)	C22—C21—H21A	109.9
C16—C11—H11	119.6	C22—C21—H21B	109.9
C12—C11—C16	120.8 (9)	C21—C22—H22A	109.5
C12—C11—H11	119.6	C21—C22—H22B	109.5
O3—C9—C8	125.3 (9)	C21—C22—H22C	109.5
O3—C9—C10	114.9 (9)	H22A—C22—H22B	109.5
C8—C9—C10	119.8 (9)	H22A—C22—H22C	109.5
C19—C20—H20A	109.5	H22B—C22—H22C	109.5
U1—O6—C17—C16	-179.6 (6)	C18—C17—C16—C15	13.4 (12)
U1—O6—C17—C18	0.3 (13)	C6—C7—C8—C9	-179.4 (9)
U1—O7—C21—C22	-90.4 (15)	C6—C5—C4—C3	0.4 (18)
U1—O4—C7—C6	177.7 (6)	C6—C1—C2—C3	0.2 (16)
U1—O4—C7—C8	-2.2 (16)	C7—C6—C5—C4	177.2 (9)
U1—O3—C9—C8	-5.2 (17)	C7—C6—C1—C2	-177.7 (9)
U1—O3—C9—C10	176.2 (7)	C11—C16—C15—C14	-0.9 (13)
U1—O5—C19—C18	-9.9 (17)	C11—C12—C13—C14	-2.4 (17)

U1—O5—C19—C20	172.1 (8)	C20—C19—C18—C17	178.2 (8)
O6—C17—C16—C11	12.1 (11)	C15—C16—C11—C12	-0.8 (13)
O6—C17—C16—C15	-166.8 (7)	C15—C14—C13—C12	0.7 (17)
O6—C17—C18—C19	4.2 (14)	C5—C6—C7—O4	-170.7 (8)
O4—C7—C8—C9	0.5 (17)	C5—C6—C7—C8	9.2 (14)
O3—C9—C8—C7	3.0 (19)	C5—C6—C1—C2	-1.2 (14)
O5—C19—C18—C17	0.3 (15)	C5—C4—C3—C2	-1.5 (19)
C17—C16—C11—C12	-179.7 (9)	C1—C6—C7—O4	5.5 (12)
C17—C16—C15—C14	178.0 (8)	C1—C6—C7—C8	-174.5 (9)
C16—C17—C18—C19	-176.0 (8)	C1—C6—C5—C4	0.9 (14)
C16—C11—C12—C13	2.5 (15)	C1—C2—C3—C4	1.2 (18)
C16—C15—C14—C13	1.0 (15)	C10—C9—C8—C7	-178.4 (10)
C18—C17—C16—C11	-167.8 (8)		

Hydrogen-bond geometry (Å, °)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O7—H7 \cdots O2 ⁱ	0.86 (6)	2.44 (5)	3.246 (10)	158 (3)

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