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## Structure Reports

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# (Butan-2-ol- $\kappa$ O)[2-({(ethylsulfanyl)-[2-(2-oxidobenzylidene- $\kappa$ O)hydrazinylidene- $\kappa$ N<sup>2</sup>]methyl]iminomethyl)-phenolato- $\kappa$ O]dioxidouranium(VI)

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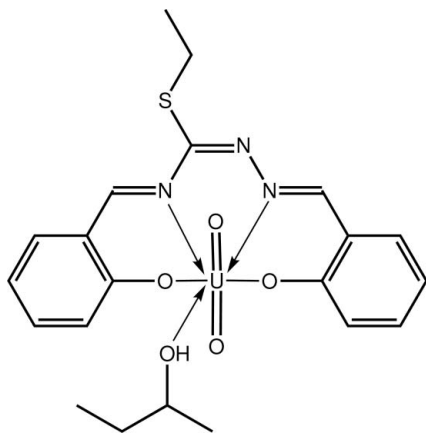
Received 28 January 2012; accepted 1 February 2012

Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.009$  Å; disorder in main residue;  $R$  factor = 0.038;  $wR$  factor = 0.078; data-to-parameter ratio = 17.4.

The U atom in the title complex,  $[\text{U}(\text{C}_{17}\text{H}_{15}\text{N}_3\text{O}_2\text{S})\text{O}_2(\text{C}_4\text{H}_{10}\text{O})]$ , exists within a distorted pentagonal-bipyramidal geometry where the oxide O atoms occupy axial positions [ $\text{O}-\text{U}-\text{O} = 179.61$  ( $18^\circ$ )] and the pentagonal plane is defined by the  $\text{N}_2\text{O}_2$  atoms of the tetradentate Schiff base ligand and the O atom of the butan-2-ol molecule. In the crystal, centrosymmetric aggregates are formed *via* pairs of hydroxy-phenoxide  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds. The azomethine  $\text{C}=\text{N}$  atoms, the ethylthioly group and the butyl group of the butan-2-ol molecule are disordered over two positions in a 0.668 (3):0.332 (3) ratio.

## Related literature

For background to uranyl Schiff base complexes, see: Şahin *et al.* (2010); Özdemir *et al.* (2011). For a related structure, see: Takjoo *et al.* (2012).


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## Experimental

## Crystal data

$[\text{U}(\text{C}_{17}\text{H}_{15}\text{N}_3\text{O}_2\text{S})\text{O}_2(\text{C}_4\text{H}_{10}\text{O})]$   
 $M_r = 669.53$   
 Monoclinic,  $P2_1/c$   
 $a = 11.3803$  (2) Å  
 $b = 14.3999$  (3) Å  
 $c = 14.0264$  (4) Å  
 $\beta = 97.326$  ( $2^\circ$ )

$V = 2279.81$  (9) Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 7.25$  mm<sup>-1</sup>  
 $T = 100$  K  
 $0.25 \times 0.10 \times 0.05$  mm

## Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector  
 Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)  
 $T_{\min} = 0.265$ ,  $T_{\max} = 0.713$

15878 measured reflections  
 5262 independent reflections  
 4493 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.040$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.038$   
 $wR(F^2) = 0.078$   
 $S = 1.16$   
 5262 reflections  
 302 parameters  
 8 restraints

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\max} = 1.16$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.81$  e Å<sup>-3</sup>

Table 1

Selected bond lengths (Å).

U—O1	2.291 (4)	U—O5	2.415 (4)
U—O2	2.229 (4)	U—N1	2.562 (5)
U—O3	1.779 (4)	U—N3	2.579 (5)
U—O4	1.776 (4)		

Table 2

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O5}-\text{H1o}\cdots\text{O1}^i$	0.84 (1)	1.83 (2)	2.648 (6)	166 (7)

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

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We gratefully acknowledge financial support of this study by Ferdowsi University of Mashhad, and thank the Ministry of Higher Education (Malaysia) for funding structural studies through the High-Impact Research scheme (UM.C/HIR/MOHE/SC/12).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6617).

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## supporting information

*Acta Cryst.* (2012). E68, m255–m256 [doi:10.1107/S160053681200431X]

**(Butan-2-ol- $\kappa$ O)[2-((ethylsulfanyl)[2-(2-oxidobenzylidene- $\kappa$ O)hydrazinylidene- $\kappa$ N<sup>2</sup>]methyl)iminomethyl)phenolato- $\kappa$ O]dioxidouranium(VI)**

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### S1. Comment

Tetradentate ligands with N<sub>2</sub>O<sub>2</sub> donor sets and their metal complexes attract attention as they provide synthetic models for the metal-containing sites in metallo-proteins and metallo-enzymes, and display extensive catalytic and bioactive applications. In this connection, recent studies of uranyl Schiff base complexes (Şahin *et al.*, 2010; Özdemir *et al.*, 2011) motivated the synthesis of the title complex, (I), in continuation of related studies (Takjoo *et al.*, 2012).

The U atom in (I), Fig. 1, exists within a distorted pentagonal bipyramidal geometry with the axial positions occupied by the oxido-O atoms, O3—U—O4 = 179.61 (18)°. The pentagonal plane is defined by the N<sub>2</sub>O<sub>2</sub> atoms, derived from the tetradentate Schiff base ligand, and the O atom of the butan-2-ol molecule, Table 1. The Schiff base ligand is somewhat twisted with the dihedral angle between the terminal benzene rings being 31.7 (3)°.

In the crystal structure, centrosymmetric pairs of molecules are linked *via* O—H...O hydrogen bonds formed between the hydroxyl and O1-phenoxide atoms, Fig. 2 and Table 2. The dimeric aggregates stack into columns parallel to *c*, Fig. 3.

### S2. Experimental

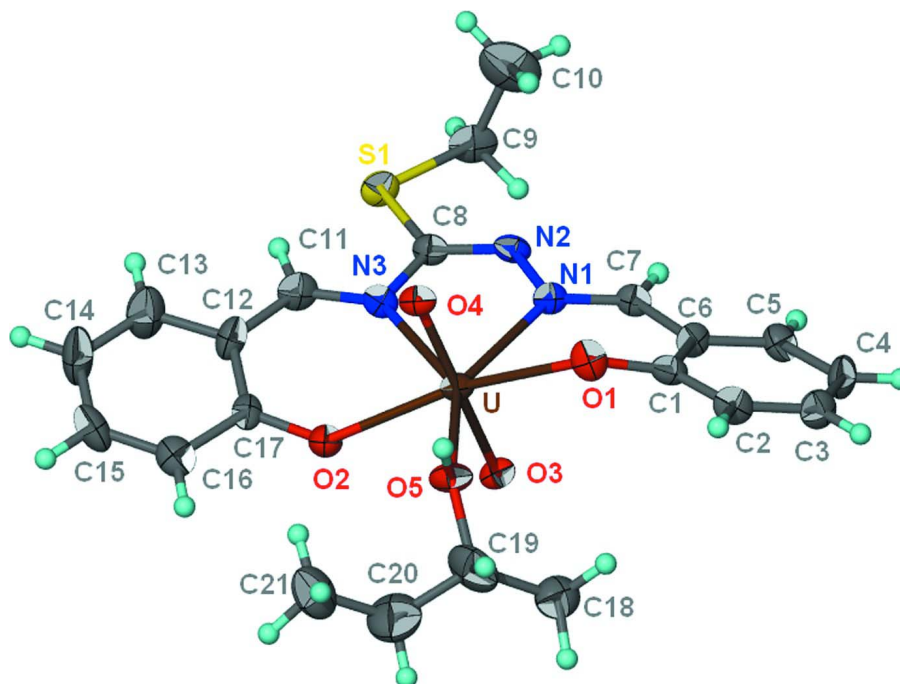
UO<sub>2</sub>(OAc)<sub>2</sub>·2H<sub>2</sub>O (0.42 g, 1.0 mmol) was added to a butanol solution (20 cm<sup>3</sup>) of salicylaldehyde mono-*S*-ethylisothiosemicarbazone hydrobromide (0.32 g, 1.0 mmol) and salicylaldehyde (0.12 g, 1.0 mmol). The red solution was heated under reflux for 1 h at 70 °C. Red prisms precipitated after four days, which were collected by filtration, washed with diethyl ether and dried in air. *M.pt.* 503 K. Yield: 25%.

### S3. Refinement

Carbon-bound H-atoms were placed in calculated positions [C—H = 0.95 to 0.99 Å,  $U_{\text{iso}}(\text{H}) = 1.2$  to  $1.5U_{\text{eq}}(\text{C})$ ] and were included in the refinement in the riding model approximation. The hydroxyl H-atom was located in a difference Fourier map, and was refined with a distance restraint of O—H 0.84±0.01 Å and with  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$ .

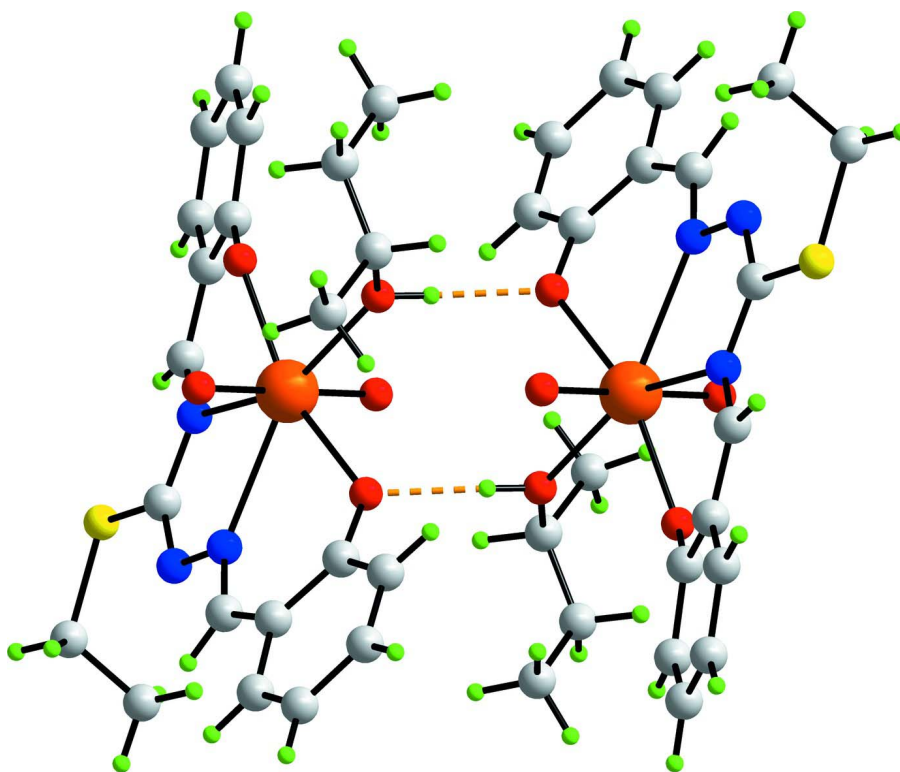
The ethylthioly unit is disordered over two positions; the minor component refined to a site occupancy of 0.332 (3). The  $U_{\text{iso}}$  parameters of the atoms of the minor component were constrained to be equal to  $U_{\text{eq}}$  of the major component. Pairs of S—C and C—C distances were restrained to within 0.01 Å of each other. The azomethine C=N unit is also disordered; the positions and anisotropic displacement parameters of the primed atoms were set to those of the unprimed ones. The butan-2-ol molecule is also disordered over two positions with respect to the butyl portion only, and the occupancies were set to those of the ethylthioly unit. Pairs of C—O and C—C distances were restrained to within 0.01 Å.

The final difference Fourier map had a peak at 0.88 Å from C19 and a hole at 0.75 Å from U.



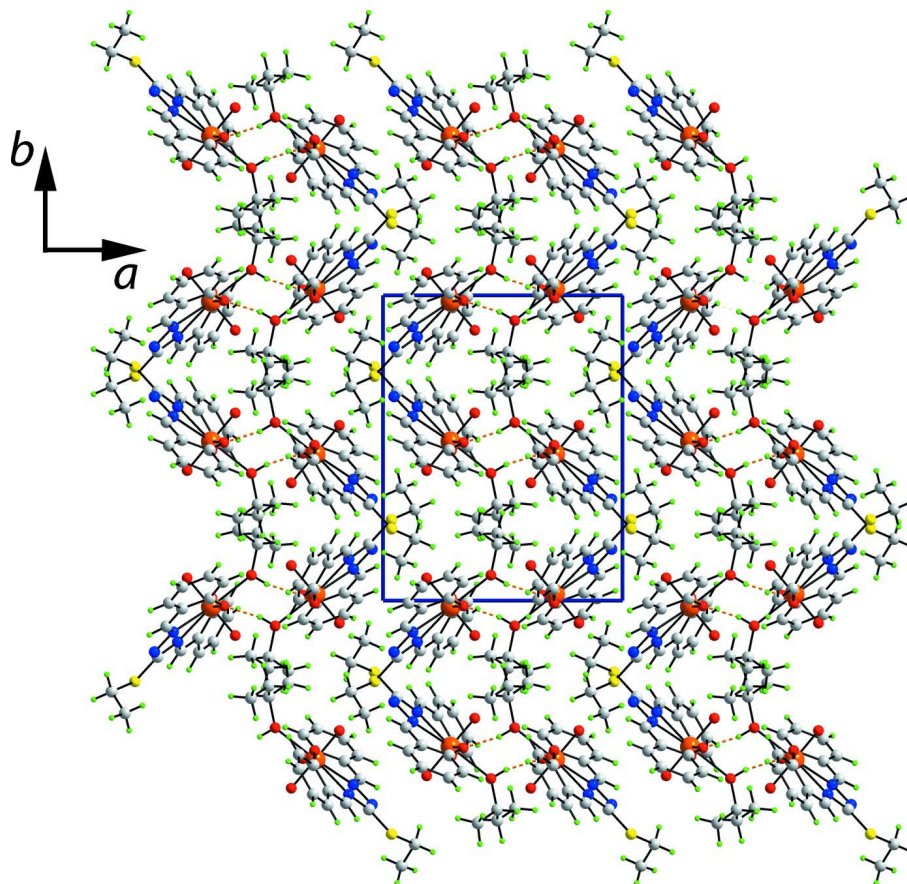
**Figure 1**

The molecular structure of (I) showing displacement ellipsoids at the 70% probability level. Only the major component of the disordered residues is shown.



**Figure 2**

A view of the centrosymmetric aggregate in (I). The O—H···O hydrogen bonds are shown as dashed lines.

**Figure 3**

A view in projection down the *c* axis of the unit-cell contents of (I).

**(Butan-2-ol- $\kappa$ O)[2-((ethylsulfanyl)[2-(2-oxidobenzylidene- $\kappa$ O)hydrazinylidene- $\kappa$ N<sup>2</sup>]methyl]iminomethyl)phenolato- $\kappa$ O]dioxidouranium(VI)**

*Crystal data*

[U(C<sub>17</sub>H<sub>15</sub>N<sub>3</sub>O<sub>2</sub>S)O<sub>2</sub>(C<sub>4</sub>H<sub>10</sub>O)]

$M_r = 669.53$

Monoclinic,  $P2_1/c$

Hall symbol: -P 2ybc

$a = 11.3803$  (2) Å

$b = 14.3999$  (3) Å

$c = 14.0264$  (4) Å

$\beta = 97.326$  (2)°

$V = 2279.81$  (9) Å<sup>3</sup>

$Z = 4$

$F(000) = 1280$

$D_x = 1.951$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 8576 reflections

$\theta = 2.2$ – $27.5$ °

$\mu = 7.25$  mm<sup>-1</sup>

$T = 100$  K

Prism, red

$0.25 \times 0.10 \times 0.05$  mm

Data collection

Agilent SuperNova Dual  
diffractometer with an Atlas detector  
Radiation source: SuperNova (Mo) X-ray  
Source  
Mirror monochromator  
Detector resolution: 10.4041 pixels mm<sup>-1</sup>  
 $\omega$  scan  
Absorption correction: multi-scan  
(*CrysAlis PRO*; Agilent, 2010)

$T_{\min} = 0.265$ ,  $T_{\max} = 0.713$   
15878 measured reflections  
5262 independent reflections  
4493 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.040$   
 $\theta_{\max} = 27.6^\circ$ ,  $\theta_{\min} = 2.3^\circ$   
 $h = -14 \rightarrow 14$   
 $k = -18 \rightarrow 18$   
 $l = -18 \rightarrow 12$

Refinement

Refinement on  $F^2$   
Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.038$   
 $wR(F^2) = 0.078$   
 $S = 1.16$   
5262 reflections  
302 parameters  
8 restraints  
Primary atom site location: structure-invariant  
direct methods

Secondary atom site location: difference Fourier  
map  
Hydrogen site location: inferred from  
neighbouring sites  
H atoms treated by a mixture of independent  
and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.0199P)^2 + 8.1201P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 1.16 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.81 \text{ e } \text{\AA}^{-3}$

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
U	0.717677 (17)	0.479390 (14)	0.617403 (15)	0.01623 (7)	
S1	1.0356 (2)	0.23488 (16)	0.73678 (17)	0.0243 (6)	0.668 (3)
S1'	1.0688 (4)	0.2786 (3)	0.5310 (3)	0.024*	0.332 (3)
O1	0.6650 (3)	0.4824 (3)	0.4542 (3)	0.0239 (9)	
O2	0.7142 (4)	0.5085 (3)	0.7731 (3)	0.0228 (9)	
O3	0.8179 (3)	0.5740 (3)	0.6153 (3)	0.0190 (9)	
O4	0.6169 (3)	0.3854 (3)	0.6190 (3)	0.0195 (9)	
O5	0.5497 (3)	0.5832 (3)	0.6033 (3)	0.0237 (9)	
H1o	0.486 (3)	0.554 (4)	0.590 (5)	0.036*	
N1	0.8758 (4)	0.3968 (3)	0.5338 (3)	0.0186 (10)	
N2	0.9541 (4)	0.3318 (3)	0.5841 (4)	0.0194 (11)	0.668 (3)
C8'	0.9541 (4)	0.3318 (3)	0.5841 (4)	0.0194 (11)	0.33
N3	0.8570 (4)	0.3646 (3)	0.7175 (3)	0.0194 (10)	
C1	0.7233 (5)	0.5070 (4)	0.3817 (4)	0.0215 (13)	
C2	0.6682 (5)	0.5611 (4)	0.3058 (4)	0.0244 (13)	
H2	0.5910	0.5850	0.3088	0.029*	
C3	0.7248 (5)	0.5801 (4)	0.2268 (4)	0.0262 (14)	
H3	0.6865	0.6174	0.1763	0.031*	
C4	0.8382 (6)	0.5450 (4)	0.2201 (4)	0.0296 (15)	
H4	0.8764	0.5576	0.1651	0.036*	
C5	0.8933 (5)	0.4923 (4)	0.2941 (4)	0.0246 (14)	
H5	0.9699	0.4677	0.2894	0.030*	
C6	0.8393 (5)	0.4738 (4)	0.3765 (4)	0.0192 (12)	
C7	0.9043 (5)	0.4154 (4)	0.4491 (4)	0.0187 (12)	

H7	0.9751	0.3878	0.4331	0.022*	
C8	0.9416 (4)	0.3157 (4)	0.6716 (4)	0.0198 (12)	0.668 (3)
N2'	0.9416 (4)	0.3157 (4)	0.6716 (4)	0.0198 (12)	0.33
C9	1.1329 (10)	0.2017 (7)	0.6476 (9)	0.029 (3)	0.668 (3)
H9A	1.1528	0.2578	0.6121	0.035*	0.668 (3)
H9B	1.2076	0.1762	0.6813	0.035*	0.668 (3)
C9'	1.117 (2)	0.1895 (15)	0.621 (2)	0.029*	0.332 (3)
H9'A	1.1998	0.1722	0.6162	0.035*	0.332 (3)
H9'B	1.1135	0.2155	0.6862	0.035*	0.332 (3)
C10	1.0774 (11)	0.1312 (9)	0.5773 (9)	0.053 (4)	0.668 (3)
H10A	1.1324	0.1162	0.5312	0.080*	0.668 (3)
H10B	1.0040	0.1565	0.5429	0.080*	0.668 (3)
H10C	1.0595	0.0748	0.6118	0.080*	0.668 (3)
C10'	1.041 (3)	0.1044 (19)	0.609 (2)	0.053*	0.332 (3)
H10D	1.0712	0.0576	0.6562	0.080*	0.332 (3)
H10E	1.0417	0.0795	0.5438	0.080*	0.332 (3)
H10F	0.9591	0.1205	0.6179	0.080*	0.332 (3)
C11	0.8453 (5)	0.3368 (4)	0.8038 (4)	0.0254 (14)	
H11	0.8910	0.2843	0.8265	0.031*	
C12	0.7716 (5)	0.3761 (4)	0.8691 (4)	0.0259 (14)	
C13	0.7682 (6)	0.3306 (5)	0.9574 (5)	0.0398 (18)	
H13	0.8097	0.2737	0.9689	0.048*	
C14	0.7077 (7)	0.3653 (6)	1.0269 (5)	0.0422 (19)	
H14	0.7068	0.3329	1.0858	0.051*	
C15	0.6474 (6)	0.4485 (5)	1.0108 (5)	0.0320 (15)	
H15	0.6047	0.4731	1.0591	0.038*	
C16	0.6485 (5)	0.4960 (4)	0.9258 (5)	0.0277 (14)	
H16	0.6073	0.5534	0.9165	0.033*	
C17	0.7097 (5)	0.4610 (4)	0.8523 (4)	0.0198 (13)	
C18	0.6045 (6)	0.7293 (5)	0.5245 (5)	0.0350 (16)	
H18A	0.5949	0.7969	0.5244	0.053*	
H18B	0.5833	0.7054	0.4591	0.053*	
H18C	0.6872	0.7135	0.5471	0.053*	
C19	0.5234 (10)	0.6853 (7)	0.5916 (8)	0.044 (3)	0.668 (3)
H19	0.4405	0.6907	0.5585	0.053*	0.668 (3)
C19'	0.574 (2)	0.6858 (9)	0.6188 (10)	0.044*	0.332 (3)
H19'	0.6420	0.6942	0.6704	0.053*	0.332 (3)
C20	0.5226 (12)	0.7204 (9)	0.6886 (9)	0.051 (3)	0.668 (3)
H20A	0.6052	0.7169	0.7207	0.062*	0.668 (3)
H20B	0.5022	0.7872	0.6827	0.062*	0.668 (3)
C20'	0.468 (2)	0.725 (2)	0.650 (2)	0.051*	0.332 (3)
H20C	0.4803	0.7928	0.6609	0.062*	0.332 (3)
H20D	0.4010	0.7179	0.5981	0.062*	0.332 (3)
C21	0.4487 (16)	0.6807 (19)	0.7538 (10)	0.042 (4)	0.668 (3)
H21A	0.4618	0.7135	0.8154	0.063*	0.668 (3)
H21B	0.4686	0.6149	0.7639	0.063*	0.668 (3)
H21C	0.3654	0.6865	0.7265	0.063*	0.668 (3)
C21'	0.434 (4)	0.685 (4)	0.737 (3)	0.042*	0.332 (3)

H21D	0.3639	0.7177	0.7544	0.063*	0.332 (3)
H21E	0.4992	0.6910	0.7889	0.063*	0.332 (3)
H21F	0.4146	0.6193	0.7256	0.063*	0.332 (3)

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
U	0.01237 (10)	0.01495 (11)	0.02069 (11)	0.00117 (9)	−0.00049 (8)	0.00006 (10)
S1	0.0231 (11)	0.0219 (12)	0.0267 (13)	0.0079 (9)	−0.0014 (10)	0.0046 (10)
O1	0.0144 (19)	0.033 (2)	0.024 (2)	0.0056 (18)	−0.0006 (17)	0.0000 (19)
O2	0.026 (2)	0.020 (2)	0.022 (2)	−0.0008 (17)	0.0038 (18)	−0.0006 (17)
O3	0.0125 (18)	0.018 (2)	0.028 (2)	0.0010 (16)	0.0060 (17)	0.0025 (17)
O4	0.0133 (19)	0.018 (2)	0.028 (2)	0.0001 (16)	0.0037 (17)	−0.0015 (17)
O5	0.018 (2)	0.016 (2)	0.037 (3)	0.0032 (17)	0.004 (2)	−0.0016 (19)
N1	0.016 (2)	0.019 (2)	0.019 (3)	0.002 (2)	−0.005 (2)	0.001 (2)
N2	0.018 (3)	0.016 (3)	0.023 (3)	0.006 (2)	−0.001 (2)	−0.003 (2)
C8'	0.018 (3)	0.016 (3)	0.023 (3)	0.006 (2)	−0.001 (2)	−0.003 (2)
N3	0.015 (2)	0.022 (3)	0.021 (3)	0.002 (2)	0.000 (2)	−0.002 (2)
C1	0.023 (3)	0.018 (3)	0.022 (3)	0.001 (2)	−0.001 (3)	−0.003 (2)
C2	0.018 (3)	0.027 (3)	0.025 (3)	0.007 (3)	−0.005 (3)	0.000 (3)
C3	0.026 (3)	0.028 (3)	0.024 (3)	0.007 (3)	−0.001 (3)	0.005 (3)
C4	0.038 (4)	0.034 (4)	0.016 (3)	−0.001 (3)	0.003 (3)	0.007 (3)
C5	0.023 (3)	0.030 (4)	0.021 (3)	0.000 (3)	0.002 (3)	−0.003 (3)
C6	0.015 (3)	0.022 (3)	0.019 (3)	0.003 (2)	−0.004 (2)	0.003 (3)
C7	0.012 (3)	0.021 (3)	0.024 (3)	0.002 (2)	0.003 (2)	−0.002 (2)
C8	0.016 (3)	0.019 (3)	0.024 (3)	0.001 (2)	0.002 (2)	−0.002 (2)
N2'	0.016 (3)	0.019 (3)	0.024 (3)	0.001 (2)	0.002 (2)	−0.002 (2)
C9	0.018 (6)	0.025 (6)	0.042 (8)	0.012 (4)	−0.004 (5)	0.002 (5)
C10	0.044 (8)	0.051 (9)	0.062 (9)	0.027 (7)	−0.002 (6)	−0.009 (7)
C11	0.020 (3)	0.026 (3)	0.029 (3)	0.008 (3)	−0.003 (3)	0.004 (3)
C12	0.023 (3)	0.032 (4)	0.023 (3)	0.001 (3)	0.004 (3)	0.010 (3)
C13	0.041 (4)	0.047 (5)	0.033 (4)	0.020 (4)	0.010 (3)	0.013 (3)
C14	0.045 (4)	0.061 (5)	0.022 (4)	0.012 (4)	0.009 (3)	0.014 (3)
C15	0.028 (3)	0.047 (4)	0.022 (3)	0.007 (3)	0.004 (3)	−0.009 (3)
C16	0.020 (3)	0.034 (4)	0.029 (3)	−0.001 (3)	0.003 (3)	−0.003 (3)
C17	0.017 (3)	0.026 (3)	0.016 (3)	−0.008 (2)	−0.001 (2)	0.000 (2)
C18	0.033 (4)	0.035 (4)	0.035 (4)	−0.002 (3)	−0.002 (3)	0.000 (3)
C19	0.038 (7)	0.049 (7)	0.050 (7)	0.005 (6)	0.020 (6)	−0.016 (6)
C20	0.051 (8)	0.046 (7)	0.057 (9)	−0.018 (6)	0.005 (6)	−0.002 (6)
C21	0.041 (8)	0.056 (8)	0.026 (7)	0.010 (6)	−0.008 (7)	−0.005 (7)

*Geometric parameters (Å, °)*

U—O1	2.291 (4)	C10—H10B	0.9800
U—O2	2.229 (4)	C10—H10C	0.9800
U—O3	1.779 (4)	C10'—H10D	0.9800
U—O4	1.776 (4)	C10'—H10E	0.9800
U—O5	2.415 (4)	C10'—H10F	0.9800



U—N1	2.562 (5)	C11—C12	1.435 (9)
U—N3	2.579 (5)	C11—H11	0.9500
S1—C8	1.757 (5)	C12—C13	1.405 (9)
S1—C9	1.836 (12)	C12—C17	1.416 (8)
S1'—C9'	1.835 (15)	C13—C14	1.357 (10)
O1—C1	1.331 (7)	C13—H13	0.9500
O2—C17	1.311 (7)	C14—C15	1.385 (10)
O5—C19	1.506 (10)	C14—H14	0.9500
O5—C19'	1.513 (13)	C15—C16	1.376 (9)
O5—H1o	0.839 (10)	C15—H15	0.9500
N1—C7	1.299 (7)	C16—C17	1.409 (8)
N1—N2	1.417 (6)	C16—H16	0.9500
N2—C8	1.274 (7)	C18—C19	1.537 (11)
N3—C11	1.298 (8)	C18—C19'	1.544 (13)
N3—C8	1.412 (7)	C18—H18A	0.9800
C1—C2	1.402 (8)	C18—H18B	0.9800
C1—C6	1.414 (8)	C18—H18C	0.9800
C2—C3	1.378 (9)	C19—C20	1.453 (13)
C2—H2	0.9500	C19—H19	1.0000
C3—C4	1.399 (9)	C19'—C20'	1.455 (15)
C3—H3	0.9500	C19'—H19'	1.0000
C4—C5	1.371 (8)	C20—C21	1.438 (14)
C4—H4	0.9500	C20—H20A	0.9900
C5—C6	1.402 (8)	C20—H20B	0.9900
C5—H5	0.9500	C20'—C21'	1.439 (16)
C6—C7	1.449 (8)	C20'—H20C	0.9900
C7—H7	0.9500	C20'—H20D	0.9900
C9—C10	1.498 (13)	C21—H21A	0.9800
C9—H9A	0.9900	C21—H21B	0.9800
C9—H9B	0.9900	C21—H21C	0.9800
C9'—C10'	1.497 (15)	C21'—H21D	0.9800
C9'—H9'A	0.9900	C21'—H21E	0.9800
C9'—H9'B	0.9900	C21'—H21F	0.9800
C10—H10A	0.9800		
O4—U—O3	179.61 (18)	H10A—C10—H10B	109.5
O4—U—O2	92.23 (16)	C9—C10—H10C	109.5
O3—U—O2	87.92 (16)	H10A—C10—H10C	109.5
O4—U—O1	86.55 (17)	H10B—C10—H10C	109.5
O3—U—O1	93.18 (16)	C9'—C10'—H10D	109.5
O2—U—O1	160.07 (14)	C9'—C10'—H10E	109.5
O4—U—O5	88.12 (15)	H10D—C10'—H10E	109.5
O3—U—O5	91.55 (15)	C9'—C10'—H10F	109.5
O2—U—O5	81.44 (15)	H10D—C10'—H10F	109.5
O1—U—O5	78.64 (14)	H10E—C10'—H10F	109.5
O4—U—N1	98.29 (16)	N3—C11—C12	127.8 (5)
O3—U—N1	81.88 (16)	N3—C11—H11	116.1
O2—U—N1	129.55 (14)	C12—C11—H11	116.1

O1—U—N1	70.20 (14)	C13—C12—C17	118.7 (6)
O5—U—N1	147.64 (15)	C13—C12—C11	117.6 (6)
O4—U—N3	81.81 (16)	C17—C12—C11	123.5 (5)
O3—U—N3	98.57 (16)	C14—C13—C12	122.3 (7)
O2—U—N3	70.97 (15)	C14—C13—H13	118.8
O1—U—N3	128.32 (15)	C12—C13—H13	118.8
O5—U—N3	150.10 (15)	C13—C14—C15	119.2 (6)
N1—U—N3	62.11 (15)	C13—C14—H14	120.4
C8—S1—C9	101.4 (4)	C15—C14—H14	120.4
C1—O1—U	132.5 (4)	C16—C15—C14	120.8 (6)
C17—O2—U	137.7 (4)	C16—C15—H15	119.6
C19—O5—U	139.3 (5)	C14—C15—H15	119.6
C19'—O5—U	117.8 (9)	C15—C16—C17	121.1 (6)
C19—O5—H1 <sub>o</sub>	108 (5)	C15—C16—H16	119.5
C19'—O5—H1 <sub>o</sub>	131 (5)	C17—C16—H16	119.5
U—O5—H1 <sub>o</sub>	111 (5)	O2—C17—C16	120.6 (5)
C7—N1—N2	112.1 (5)	O2—C17—C12	121.3 (5)
C7—N1—U	126.5 (4)	C16—C17—C12	118.0 (5)
N2—N1—U	120.9 (3)	C19—C18—H18A	109.5
C8—N2—N1	117.3 (4)	C19—C18—H18B	109.5
C11—N3—C8	115.4 (5)	H18A—C18—H18B	109.5
C11—N3—U	125.1 (4)	C19—C18—H18C	109.5
C8—N3—U	118.6 (3)	H18A—C18—H18C	109.5
O1—C1—C2	120.7 (5)	H18B—C18—H18C	109.5
O1—C1—C6	120.6 (5)	C20—C19—O5	105.2 (9)
C2—C1—C6	118.6 (6)	C20—C19—C18	120.4 (9)
C3—C2—C1	120.7 (6)	O5—C19—C18	110.0 (6)
C3—C2—H2	119.6	C20—C19—H19	106.8
C1—C2—H2	119.6	O5—C19—H19	106.8
C2—C3—C4	120.8 (6)	C18—C19—H19	106.8
C2—C3—H3	119.6	C20'—C19'—O5	106.5 (17)
C4—C3—H3	119.6	C20'—C19'—C18	113.0 (17)
C5—C4—C3	119.1 (6)	O5—C19'—C18	109.2 (8)
C5—C4—H4	120.5	C20'—C19'—H19'	109.3
C3—C4—H4	120.5	O5—C19'—H19'	109.3
C4—C5—C6	121.5 (6)	C18—C19'—H19'	109.3
C4—C5—H5	119.3	C21—C20—C19	122.1 (13)
C6—C5—H5	119.3	C21—C20—H20A	106.8
C5—C6—C1	119.3 (5)	C19—C20—H20A	106.8
C5—C6—C7	116.8 (5)	C21—C20—H20B	106.8
C1—C6—C7	123.8 (5)	C19—C20—H20B	106.8
N1—C7—C6	126.7 (5)	H20A—C20—H20B	106.7
N1—C7—H7	116.7	C21'—C20'—C19'	115 (3)
C6—C7—H7	116.7	C21'—C20'—H20C	108.6
N2—C8—N3	121.0 (4)	C19'—C20'—H20C	108.6
N2—C8—S1	119.1 (4)	C21'—C20'—H20D	108.6
N3—C8—S1	119.9 (4)	C19'—C20'—H20D	108.6
C10—C9—S1	112.6 (9)	H20C—C20'—H20D	107.5

C10—C9—H9A	109.1	C20—C21—H21A	109.5
S1—C9—H9A	109.1	C20—C21—H21B	109.5
C10—C9—H9B	109.1	H21A—C21—H21B	109.5
S1—C9—H9B	109.1	C20—C21—H21C	109.5
H9A—C9—H9B	107.8	H21A—C21—H21C	109.5
C10'—C9'—S1'	112.0 (17)	H21B—C21—H21C	109.5
C10'—C9'—H9'A	109.2	C20'—C21'—H21D	109.5
S1'—C9'—H9'A	109.2	C20'—C21'—H21E	109.5
C10'—C9'—H9'B	109.2	H21D—C21'—H21E	109.5
S1'—C9'—H9'B	109.2	C20'—C21'—H21F	109.5
H9'A—C9'—H9'B	107.9	H21D—C21'—H21F	109.5
C9—C10—H10A	109.5	H21E—C21'—H21F	109.5
C9—C10—H10B	109.5		
O4—U—O1—C1	-150.2 (5)	C2—C3—C4—C5	-0.9 (10)
O3—U—O1—C1	30.1 (5)	C3—C4—C5—C6	-0.8 (10)
O2—U—O1—C1	122.8 (5)	C4—C5—C6—C1	2.7 (9)
O5—U—O1—C1	121.0 (5)	C4—C5—C6—C7	178.5 (6)
N1—U—O1—C1	-50.1 (5)	O1—C1—C6—C5	172.9 (5)
N3—U—O1—C1	-73.5 (5)	C2—C1—C6—C5	-2.9 (8)
O4—U—O2—C17	37.7 (5)	O1—C1—C6—C7	-2.6 (9)
O3—U—O2—C17	-142.6 (5)	C2—C1—C6—C7	-178.4 (5)
O1—U—O2—C17	123.7 (6)	N2—N1—C7—C6	175.5 (5)
O5—U—O2—C17	125.5 (5)	U—N1—C7—C6	-12.5 (8)
N1—U—O2—C17	-65.0 (6)	C5—C6—C7—N1	173.2 (6)
N3—U—O2—C17	-42.8 (5)	C1—C6—C7—N1	-11.2 (9)
O4—U—O5—C19	-176.8 (7)	N1—N2—C8—N3	-3.1 (8)
O3—U—O5—C19	3.0 (7)	N1—N2—C8—S1	179.5 (4)
O2—U—O5—C19	90.7 (7)	C11—N3—C8—N2	174.3 (5)
O1—U—O5—C19	-89.9 (7)	U—N3—C8—N2	4.1 (7)
N1—U—O5—C19	-74.2 (8)	C11—N3—C8—S1	-8.3 (7)
N3—U—O5—C19	113.3 (7)	U—N3—C8—S1	-178.5 (2)
O4—U—O5—C19'	167.3 (6)	C9—S1—C8—N2	2.3 (6)
O3—U—O5—C19'	-12.9 (6)	C9—S1—C8—N3	-175.1 (5)
O2—U—O5—C19'	74.8 (6)	C8—S1—C9—C10	-80.6 (9)
O1—U—O5—C19'	-105.8 (6)	C8—N3—C11—C12	176.4 (6)
N1—U—O5—C19'	-90.1 (6)	U—N3—C11—C12	-14.1 (9)
N3—U—O5—C19'	97.4 (6)	N3—C11—C12—C13	176.4 (7)
O4—U—N1—C7	113.7 (5)	N3—C11—C12—C17	-8.4 (10)
O3—U—N1—C7	-66.0 (5)	C17—C12—C13—C14	0.0 (11)
O2—U—N1—C7	-146.5 (4)	C11—C12—C13—C14	175.5 (7)
O1—U—N1—C7	30.4 (4)	C12—C13—C14—C15	-0.2 (12)
O5—U—N1—C7	14.0 (6)	C13—C14—C15—C16	-0.2 (11)
N3—U—N1—C7	-170.2 (5)	C14—C15—C16—C17	0.8 (10)
O4—U—N1—N2	-75.1 (4)	U—O2—C17—C16	-145.9 (5)
O3—U—N1—N2	105.3 (4)	U—O2—C17—C12	37.9 (8)
O2—U—N1—N2	24.8 (4)	C15—C16—C17—O2	-177.3 (6)
O1—U—N1—N2	-158.4 (4)	C15—C16—C17—C12	-1.0 (9)

O5—U—N1—N2	-174.8 (3)	C13—C12—C17—O2	176.9 (6)
N3—U—N1—N2	1.0 (3)	C11—C12—C17—O2	1.7 (9)
C7—N1—N2—C8	173.0 (5)	C13—C12—C17—C16	0.6 (9)
U—N1—N2—C8	0.6 (6)	C11—C12—C17—C16	-174.6 (6)
O4—U—N3—C11	-67.7 (5)	C19'—O5—C19—C20	-58.1 (15)
O3—U—N3—C11	112.3 (5)	U—O5—C19—C20	-93.5 (9)
O2—U—N3—C11	27.6 (5)	C19'—O5—C19—C18	73.0 (11)
O1—U—N3—C11	-146.7 (4)	U—O5—C19—C18	37.6 (12)
O5—U—N3—C11	3.9 (6)	C19'—C18—C19—C20	49.7 (16)
N1—U—N3—C11	-171.6 (5)	C19'—C18—C19—O5	-72.9 (11)
O4—U—N3—C8	101.5 (4)	C19—O5—C19'—C20'	51.0 (19)
O3—U—N3—C8	-78.5 (4)	U—O5—C19'—C20'	-154.3 (14)
O2—U—N3—C8	-163.3 (4)	C19—O5—C19'—C18	-71.3 (11)
O1—U—N3—C8	22.5 (4)	U—O5—C19'—C18	83.4 (14)
O5—U—N3—C8	173.0 (3)	C19—C18—C19'—C20'	-47 (2)
N1—U—N3—C8	-2.5 (3)	C19—C18—C19'—O5	71.2 (11)
U—O1—C1—C2	-136.4 (5)	O5—C19—C20—C21	-54.6 (18)
U—O1—C1—C6	47.9 (8)	C18—C19—C20—C21	-179.5 (14)
O1—C1—C2—C3	-174.5 (6)	O5—C19'—C20'—C21'	58 (4)
C6—C1—C2—C3	1.3 (9)	C18—C19'—C20'—C21'	178 (3)
C1—C2—C3—C4	0.6 (10)		

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O5—H1 $o$ —O1 <sup>i</sup>	0.84 (1)	1.83 (2)	2.648 (6)	166 (7)

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