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$(C_7H_{20}N_2)[(UO_2)_2(SO_4)_3(H_2O)]$: an organically templated uranium sulfate with a novel layer topology

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.011 Å
R factor = 0.037
wR factor = 0.097
Data-to-parameter ratio = 15.7

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

The title compound, poly[*N*,*N*,*N*′,*N*′-tetramethylpropane-1,3-diaminium [aquatetraoxotri-μ_3-sulfatodiuranate(VI)]], $(C_7H_{20}N_2)[(UO_2)_2(SO_4)_3(H_2O)]_n$, contains two-dimensional $[(UO_2)_2(SO_4)_3(H_2O)]^{2-}$ layers that are unprecedented in uranium chemistry. The layers in this compound are separated by $C_7H_{20}N_2^{2+}$ cations, forming the basis of an extensive hydrogen-bonding network. An independent intralayer hydrogen-bonding network is also observed, involving water molecules bound directly to a uranium centre.

Comment

Hydrothermal actinide chemistry has been the focus of intense interest in recent years. Great structural diversity is observed in actinide sulfates (Doran *et al.*, 2002; Norquist *et al.*, 2002; Thomas *et al.*, 2003), phosphates (Doran, Stuart *et al.*, 2004; Burns *et al.*, 2004), iodates (Bean *et al.*, 2004), selenites (Almond *et al.*, 2004), carbonates (Kubatko & Burns, 2004), molybdates (Krivovichev *et al.*, 2005) and chromates (Sykora *et al.*, 2004). These studies have led to the formation of a host of novel inorganic structures. Several strategies are employed for the formation of new structure types, including the use of alternate coordination polyhedra (Kubatko & Burns, 2004; Sykora *et al.*, 2004), inclusion of organic components into the framework (Kim *et al.*, 2003), systematic exploration of reaction conditions (Norquist *et al.*, 2003) and use of varied organic amines (Doran, Norquist *et al.*, 2004). The last approach is utilized in this study for the formation of an organically templated uranium sulfate with a novel layer

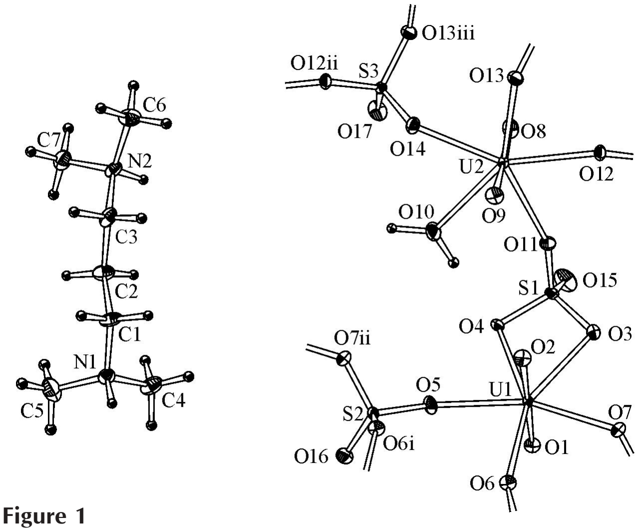


Figure 1
View of the title compound, with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) $2 - x, 1 - y, 1 - z$; (ii) $x - 1, y, z$; (iii) $1 - x, 1 - y, 2 - z$.]

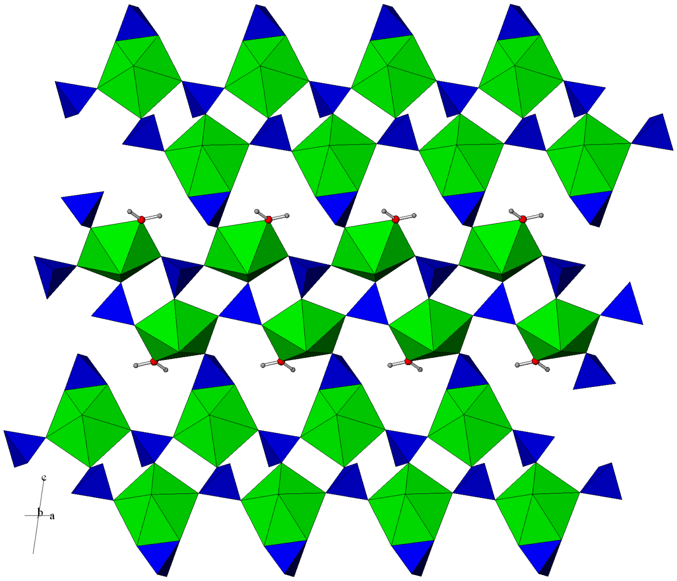
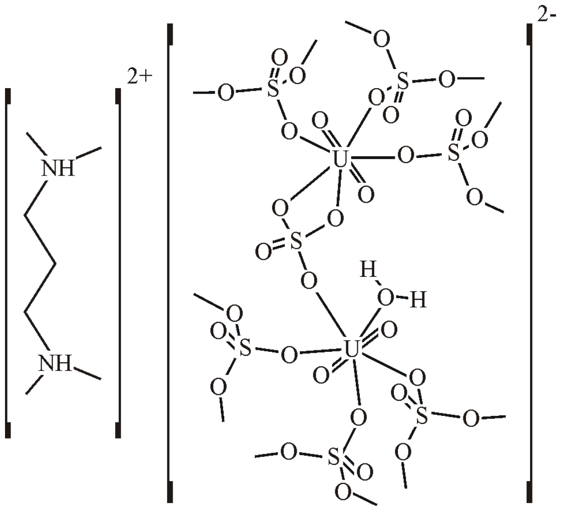


Figure 2
Layer structure in USO-30. Green pentagonal bipyramids and blue tetrahedra represent $[UO_7]$ and $[SO_4]$, respectively.

topology. This compound, $[C_7H_{20}N_2][(UO_2)_2(SO_4)_3(H_2O)]$, (I), is denoted USO-30 (uranium sulfate from Oxford).



(I)

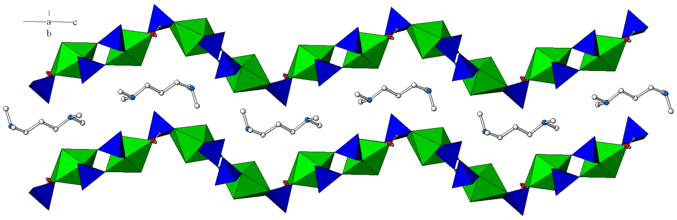


Figure 3
Three-dimensional packing of USO-30. Green pentagonal bipyramids and blue tetrahedra represent $[UO_7]$ and $[SO_4]$, respectively. Template H atoms have been omitted for clarity.

Two unique uranium centres are present in USO-30. Both U1 and U2 are seven-coordinate (Fig. 1), in a pentagonal bipyramidal geometry (Table 1). Two short 'uranyl' bonds to axial oxide ligands are observed for each uranium environment, with distances that range from 1.754 (5) to 1.770 (5) Å, which are close to the average reported value of 1.758 (3) Å (Burns *et al.*, 1997). The O1—U1—O2 and O8—U2—O9 bond angles are close to 180°, with values of 179.4 (2) and 178.5 (2)°. Each of the five equatorial coordination sites around U1 is occupied by O atoms that are bonded to sulfur centres. Three of these O atoms, O5, O6 and O7, bridge to different $[SO_4]$ tetrahedra, while O3 and O4 bridge to the same sulfur centre, creating a shared edge between the $[SO_4]$ and $[UO_7]$ polyhedra. The result of this shared edge is an elongation of the U1—O3 and U1—O4 bond lengths [2.470 (5) and 2.441 (5) Å] with respect to the U1—O5, U1—O6 and U1—O7 lengths [2.333 (5)–2.373 (5) Å]. A contraction of the O3—U1—O4 bond angle, with respect to the other equatorial bond angles, is also observed. The O3—U1—O4 angle is 57.28 (16)°, while the other four O_{eq}—U1—O_{eq} bond angles range between 72.00 (16) and 78.96 (17)°. The equatorial coordination sites of the second unique uranium environment, U2, are occupied by four bridging O atoms, with distances ranging between 2.337 (5) and 2.367 (5) Å, and a bound water molecule, which exhibits a U—O_{water} distance of 2.522 (5) Å. The assignment of the bound water molecule was based on hydrogen-bonding interactions. Three distinct sulfur environments are observed in USO-30, each of which is at the centre of an $[SO_4]$ tetrahedron. Each sulfur centre is bound to one terminal O atom and three O atoms that bridge to uranium centres. The S—$O_{bridging}$ distances range between 1.461 (5) and 1.509 (5) Å, while the S—$O_{terminal}$ distances range from 1.437 (6) to 1.440 (5) Å.

The presence of numerous shared O atoms, between $[UO_7]$ and $[SO_4]$ polyhedra, results in the formation of an inorganic structure that extends in two dimensions (Fig. 2). This layer topology is, to the best of our knowledge, unprecendented in uranium chemistry. Two distinct one-dimensional chains are observed, both of which contain a $[UO_2(SO_4)_{3/3}]$ backbone. This chain structure is well known in uranium chemistry (Brandeburg & Loopstra, 1973; Zalkin *et al.*, 1978; Serezhkin *et al.*, 1981; Doran *et al.*, 2003). In each chain, three of the five equatorial coordination sites surrounding both U1 and U2 participate in chain construction. The other two equatorial sites contain O atoms that are not involved in chain propagation. The two non-backbone sites in the chain containing U1 are occupied by a single $[SO_4]$ tetrahedron, containing S1, which shares a common edge with the U1 $[UO_7]$ pentagonal bipyramid. The same coordination sites on the chain that contains U2 are occupied by one bound water molecule and one O atom that is part of the aforementioned S1 sulfate tetrahedron; the result is the formation of the layers shown in Fig. 2.

The bound water molecule on U2 acts as a hydrogen-bond donor in USO-30. The acceptors are O3 and O4, resulting in intralayer hydrogen bonding. The orientations of successive bound water molecules adopt an 'up–down–up' motif. The interlayer spacing is occupied by the protonated amines, which also act as hydrogen-bond donors. The orientation of these cations is shown in Fig. 3. Hydrogen bonding details are listed in Table 2.

Experimental

$UO_2(CH_3CO_2)_2\cdot 2H_2O$ (0.2767 g, 0.653×10^{-3} mol), H_2SO_4 (0.3394 g, 3.46×10^{-3} mol), *N*,*N*,*N*′*N*′-tetramethyl-1,3-propanediamine (0.1317 g, 1.01×10^{-3} mol) and water (1.0557g, 58.7×10^{-3} mol) were placed in a 23 ml Teflon-lined autoclave. The autoclave was heated to 453 K for 24 h, and then slowly cooled to 297 K over an additional period of 24 h. The autoclave was opened in air and the products recovered by filtration. A yield of 87%, based on uranium, was observed. USO-30 was characterized using several techniques. Through the use of IR spectroscopy, bands from both the organic and the inorganic components of USO-30 were observed. A broad band centred at 3400 cm^{-1}, corresponding to the symmetric stretch of the bound water, was present. Bands at 3154 and 1620 cm^{-1} correspond to the N—H stretching and bending modes, while C—H bands were observed at 1464 and 1482 cm^{-1}. C—N stretching modes were observed around 1230 cm^{-1}, while S—O and uranyl bands were observed at 1100, and 930 and 942 cm^{-1}, respectively. Analysis found: N 2.87, C 8.69, H 2.15, S 9.87, U 48.13%; calculated: N 2.86, C 8.59, H 2.25, S 9.82, U 48.67%. The thermal stability of USO-30 was probed using thermogravimetric analysis. A 1.0% weight loss was observed between 443 and 503 K, which corresponds to loss of the bound water molecules (calculated 1.8%). Template decomposition begins at 573 K, and is soon followed by the breakdown of the inorganic layers. The material calcines to UO_2 by 1273 K, as determined using powder X-ray diffraction, with a total weight loss of 43.4% (calculated 44.8%). Structural analysis was conducted at 150 K.

Crystal data

$(C_7H_{20}N_2)[U_2O_4(SO_4)_3(H_2O)]$
$M_r = 978.51$
Triclinic, $P\overline{1}$
$a = 6.7861\ (1)$ Å
$b = 8.5143\ (1)$ Å
$c = 19.0442\ (3)$ Å
$\alpha = 88.6230\ (9)^\circ$
$\beta = 81.6364\ (8)^\circ$
$\gamma = 84.8577\ (6)^\circ$
$V = 1084.20\ (3)$ Å^3
$Z = 2$
$D_x = 2.997$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4635 reflections
$\theta = 5$–27°
$\mu = 15.29$ mm^{-1}
$T = 150$ K
Block, yellow
$0.35 \times 0.18 \times 0.18$ mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (*DENZO*/*SCALEPACK*; Otwinowski & Minor, 1997)
$T_{\min} = 0.053$, $T_{\max} = 0.064$
9107 measured reflections
4907 independent reflections
4422 reflections with $I > 3\sigma(I)$
$R_{\rm int} = 0.03$
$\theta_{\max} = 27.5^\circ$
$h = -8 \rightarrow 8$
$k = -11 \rightarrow 11$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.097$
$S = 0.91$
4422 reflections
281 parameters
H-atom parameters constrained
Chebychev polynomial (Watkin, 1994; Prince, 1982) with five parameters 61.5, 99.0, 63.5, 27.7, 8.43
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 3.72$ e Å^{-3}
$\Delta\rho_{\min} = -3.36$ e Å^{-3}
Extinction correction: Larson (1970), equation 22
Extinction coefficient: 70 (3)

Table 1
Selected geometric parameters (Å, °).

U1—O1	1.758 (5)	S1—O15	1.437 (6)
U1—O2	1.754 (5)	S2—O5	1.469 (5)
U1—O3	2.470 (5)	S2—O6^{i}	1.497 (5)
U1—O4	2.441 (5)	S2—O7^{ii}	1.487 (5)
U1—O5	2.333 (5)	S2—O16	1.438 (5)
U1—O6	2.360 (5)	S3—O12^{ii}	1.482 (5)
U1—O7	2.373 (5)	S3—O13^{iii}	1.480 (5)
U2—O8	1.765 (5)	S3—O14	1.492 (5)
U2—O9	1.770 (5)	S3—O17	1.440 (5)
U2—O10	2.522 (5)	N1—C1	1.512 (10)
U2—O11	2.361 (5)	N1—C4	1.501 (10)
U2—O12	2.342 (5)	N1—C5	1.485 (10)
U2—O13	2.367 (5)	N2—C3	1.507 (9)
U2—O14	2.337 (5)	N2—C6	1.507 (10)
S1—O3	1.496 (5)	N2—C7	1.484 (10)
S1—O4	1.509 (5)	C1—C2	1.515 (9)
S1—O11	1.461 (5)	C2—C3	1.541 (10)
O1—U1—O2	179.4 (2)	O11—U2—O14	138.30 (18)
O1—U1—O3	92.8 (2)	O12—U2—O14	149.75 (17)
O2—U1—O3	87.4 (2)	O13—U2—O14	75.94 (17)
O1—U1—O4	88.92 (19)	O3—S1—O4	103.1 (3)
O2—U1—O4	90.76 (19)	O3—S1—O11	108.2 (3)
O3—U1—O4	57.28 (16)	O4—S1—O11	107.9 (3)
O1—U1—O5	89.3 (2)	O3—S1—O15	112.4 (3)
O2—U1—O5	90.2 (2)	O4—S1—O15	112.2 (3)
O3—U1—O5	129.16 (17)	O11—S1—O15	112.5 (4)
O4—U1—O5	72.00 (16)	O5—S2—O6^{i}	108.2 (3)
O1—U1—O6	87.57 (19)	O5—S2—O7^{ii}	107.4 (3)
O2—U1—O6	92.5 (2)	O6^{i}—S2—O7^{ii}	106.3 (3)
O3—U1—O6	151.87 (16)	O5—S2—O16	110.8 (3)
O4—U1—O6	150.78 (16)	O6^{i}—S2—O16	111.9 (3)
O5—U1—O6	78.96 (17)	O7^{ii}—S2—O16	112.0 (3)
O1—U1—O7	90.7 (2)	O12^{ii}—S3—O13^{iii}	108.6 (3)
O2—U1—O7	89.9 (2)	O12^{ii}—S3—O14	104.6 (3)
O3—U1—O7	73.82 (17)	O13^{iii}—S3—O14	108.5 (3)
O4—U1—O7	131.00 (16)	O12^{ii}—S3—O17	113.1 (3)
O5—U1—O7	156.99 (17)	O13^{iii}—S3—O17	110.3 (3)
O6—U1—O7	78.05 (17)	O14—S3—O17	111.5 (3)
O8—U2—O9	178.5 (2)	U1—O3—S1	98.8 (2)
O8—U2—O10	92.3 (2)	U1—O4—S1	99.7 (2)
O9—U2—O10	87.5 (2)	U1—O5—S2	155.4 (3)
O8—U2—O11	90.5 (2)	U1—O6—S2^{i}	134.6 (3)
O9—U2—O11	90.7 (2)	U1—O7—S2^{iv}	133.7 (3)
O10—U2—O11	68.36 (18)	U2—O11—S1	155.0 (3)
O8—U2—O12	88.8 (2)	U2—O12—S3^{iv}	144.1 (3)
O9—U2—O12	92.3 (2)	U2—O13—S3^{iii}	143.7 (3)
O10—U2—O12	140.21 (17)	U2—O14—S3	131.3 (3)
O11—U2—O12	71.85 (17)	C1—N1—C4	111.5 (6)
O8—U2—O13	92.8 (2)	C1—N1—C5	114.8 (6)
O9—U2—O13	86.6 (2)	C4—N1—C5	110.0 (7)
O10—U2—O13	145.43 (17)	C3—N2—C6	110.0 (6)
O11—U2—O13	145.69 (17)	C3—N2—C7	112.9 (6)
O12—U2—O13	74.09 (16)	C6—N2—C7	110.7 (6)
O8—U2—O14	88.6 (2)	N1—C1—C2	113.0 (6)
O9—U2—O14	90.0 (2)	C1—C2—C3	107.7 (6)
O10—U2—O14	70.02 (18)	N2—C3—C2	110.2 (6)

Symmetry codes: (i) $-x+2, -y+1, -z+1$; (ii) $x-1, y, z$; (iii) $-x+1, -y+1, -z+2$; (iv) $x+1, y, z$.

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

D—H···A	D—H	H···A	D···A	D—H···A
O10—H1···O3^{ii}	1.00	2.01	2.877 (7)	143
O10—H2···O4	1.00	1.84	2.788 (7)	158
N1—H21···O16^{v}	1.00	2.12	2.850 (8)	128
N2—H22···O15^{vi}	1.00	2.34	2.935 (8)	117
N2—H22···O17^{vii}	1.00	2.21	2.889 (8)	123

Symmetry codes: (ii) $x-1, y, z$; (v) $-x+1, -y+1, -z+1$; (vi) $x-1, y-1, z$; (vii) $x, y-1, z$.

Acta Cryst. (2005). E**61**, m807–m810 Norquist *et al.* • $(C_7H_{20}N_2)[U_2O_4(SO_4)_3(H_2O)]$ **m809**

The C- and N-bound H atoms were positioned in idealized locations. The water H atoms were positioned geometrically to make plausible H$\cdots$O hydrogen bonds. All H atoms were refined as riding on their carrier atoms [C—H, N—H and O—H = 1.00 Å and U_{iso}(H) = 1.2U_{eq} (carrier atom)]. The highest peak is 1.30 Å from U1 and the deepest hole is 0.99 Å from U1.

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO*/*SCALEPACK*; program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ATOMS* (Dowty, 2000); software used to prepare material for publication: *CRYSTALS*.

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$(C_7H_{20}N_2)[(UO_2)_2(SO_4)_3(H_2O)]$: an organically templated uranium sulfate with a novel layer topology

Alexander J. Norquist, Michael B. Doran and Dermot O'Hare

Poly[*N*,*N*,*N*′,*N*′-tetramethylpropane-1,3-diaminium [aquatetraoxotri-μ_3-sulfato-diuranate(VI)]]

Crystal data

$(C_7H_{20}N_2)[U_2O_4(SO_4)_3(H_2O)]$
$M_r = 978.51$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 6.7861$ (1) Å
$b = 8.5143$ (1) Å
$c = 19.0442$ (3) Å
$\alpha = 88.6230$ (9)°
$\beta = 81.6364$ (8)°
$\gamma = 84.8577$ (6)°
$V = 1084.20$ (3) Å^3

$Z = 2$
$F(000) = 892$
$D_x = 2.997$ Mg m^{-3}
Melting point: not measured K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4635 reflections
$\theta = 5$–27°
$\mu = 15.29$ mm^{-1}
$T = 150$ K
Block, yellow
0.35 × 0.18 × 0.18 mm

Data collection

Nonius Kappa CCD diffractometer
Graphite monochromator
ω scans
Absorption correction: multi-scan (Denzo/Scalepack; Otwinowski & Minor, 1997)
$T_{min} = 0.053$, $T_{max} = 0.064$
9107 measured reflections

4907 independent reflections
4422 reflections with $I > 3u(I)$
$R_{int} = 0.03$
$\theta_{max} = 27.5°$, $\theta_{min} = 5.2°$
$h = -8 \rightarrow 8$
$k = -11 \rightarrow 11$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.097$
$S = 0.91$
4422 reflections
281 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
Chebychev polynomial (Watkin, 1994, Prince, 1982) with five parameters 61.5, 99.0, 63.5, 27.7, 8.43
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 3.72$ e Å^{-3}
$\Delta\rho_{min} = -3.36$ e Å^{-3}
Extinction correction: Larson 1970 Crystallographic Computing eq 22
Extinction coefficient: 70 (3)

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

	x	y	z	$U_{\rm iso}*/U_{\rm eq}$
U1	1.16861 (3)	0.66755 (3)	0.601618 (11)	0.0075
U2	0.77237 (3)	0.56677 (3)	0.884025 (11)	0.0086
S1	1.0637 (2)	0.81558 (19)	0.74968 (8)	0.0110
S2	0.6892 (2)	0.61770 (18)	0.53520 (8)	0.0086
S3	0.2551 (2)	0.61767 (18)	0.93931 (8)	0.0083
O1	1.2072 (7)	0.8486 (6)	0.5577 (3)	0.0138
O2	1.1276 (7)	0.4873 (6)	0.6454 (3)	0.0140
O3	1.2667 (7)	0.7596 (6)	0.7125 (3)	0.0154
O4	0.9322 (7)	0.8080 (6)	0.6927 (2)	0.0112
O5	0.8521 (8)	0.6785 (6)	0.5667 (3)	0.0152
O6	1.2441 (7)	0.5466 (5)	0.4896 (2)	0.0120
O7	1.5188 (7)	0.6041 (6)	0.5931 (3)	0.0151
O8	0.7119 (7)	0.7377 (6)	0.9362 (3)	0.0150
O9	0.8266 (8)	0.3941 (6)	0.8324 (3)	0.0163
O10	0.6138 (8)	0.6932 (8)	0.7824 (3)	0.0286
O11	1.0037 (8)	0.7000 (7)	0.8048 (3)	0.0226
O12	1.0821 (7)	0.5345 (6)	0.9255 (3)	0.0123
O13	0.7285 (7)	0.3965 (6)	0.9841 (3)	0.0132
O14	0.4323 (7)	0.5273 (6)	0.8989 (3)	0.0140
O15	1.0571 (9)	0.9719 (7)	0.7773 (3)	0.0256
O16	0.6347 (8)	0.7192 (6)	0.4784 (3)	0.0156
O17	0.2416 (8)	0.7810 (6)	0.9177 (3)	0.0184
N1	0.6377 (10)	0.1872 (7)	0.6186 (3)	0.0153
N2	0.3177 (10)	0.0966 (7)	0.8688 (3)	0.0153
C1	0.4853 (12)	0.2416 (9)	0.6811 (4)	0.0184
C2	0.5008 (12)	0.1413 (9)	0.7472 (4)	0.0186
C3	0.3397 (12)	0.2099 (8)	0.8067 (4)	0.0174
C4	0.6097 (13)	0.0232 (9)	0.5970 (4)	0.0222
C5	0.8490 (13)	0.1985 (12)	0.6284 (5)	0.0301
C6	0.1330 (13)	0.1472 (9)	0.9198 (4)	0.0228
C7	0.4965 (12)	0.0786 (10)	0.9058 (4)	0.0212
H1	0.4760	0.6785	0.7723	0.0352*
H2	0.7096	0.7609	0.7537	0.0352*
H3	0.3474	0.2382	0.6680	0.0212*
H4	0.5050	0.3533	0.6921	0.0212*
H5	0.4766	0.0294	0.7378	0.0223*
H6	0.6362	0.1432	0.7614	0.0223*
H7	0.2088	0.2290	0.7881	0.0215*
H8	0.3798	0.3120	0.8226	0.0215*
H9	0.4665	0.0162	0.5905	0.0269*
H10	0.6981	−0.0046	0.5517	0.0269*
H11	0.6435	−0.0538	0.6352	0.0269*
H12	0.8850	0.1236	0.6667	0.0381*
H13	0.9396	0.1728	0.5832	0.0381*
H14	0.8662	0.3091	0.6425	0.0381*

H15	0.0122	0.1489	0.8950	0.0262*
H16	0.1184	0.0717	0.9610	0.0262*
H17	0.1439	0.2554	0.9376	0.0262*
H18	0.4747	0.0019	0.9463	0.0264*
H19	0.6164	0.0384	0.8719	0.0264*
H20	0.5204	0.1831	0.9244	0.0264*
H21	0.6123	0.2621	0.5788	0.0183*
H22	0.3047	−0.0101	0.8496	0.0183*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
U1	0.00769 (15)	0.00765 (15)	0.00742 (15)	−0.00129 (9)	−0.00130 (9)	−0.00145 (9)
U2	0.00748 (15)	0.01087 (16)	0.00785 (15)	−0.00169 (9)	−0.00139 (9)	−0.00041 (9)
S1	0.0126 (7)	0.0139 (8)	0.0071 (7)	−0.0034 (6)	−0.0019 (5)	−0.0009 (5)
S2	0.0080 (7)	0.0100 (7)	0.0084 (7)	−0.0009 (5)	−0.0021 (5)	−0.0036 (5)
S3	0.0079 (7)	0.0080 (7)	0.0094 (7)	−0.0023 (5)	−0.0016 (5)	0.0021 (5)
O1	0.015 (2)	0.013 (2)	0.013 (2)	−0.0009 (18)	−0.0012 (18)	0.0000 (18)
O2	0.017 (2)	0.009 (2)	0.016 (2)	0.0013 (18)	−0.0018 (19)	−0.0005 (17)
O3	0.012 (2)	0.022 (3)	0.013 (2)	−0.0031 (19)	−0.0032 (18)	−0.0012 (19)
O4	0.011 (2)	0.016 (2)	0.007 (2)	0.0000 (17)	−0.0021 (16)	−0.0011 (17)
O5	0.014 (2)	0.017 (2)	0.016 (2)	−0.0009 (19)	−0.0063 (19)	−0.0076 (19)
O6	0.016 (2)	0.007 (2)	0.013 (2)	−0.0008 (17)	−0.0008 (18)	−0.0040 (17)
O7	0.010 (2)	0.021 (3)	0.014 (2)	−0.0032 (19)	0.0002 (18)	0.0008 (19)
O8	0.016 (2)	0.010 (2)	0.019 (2)	0.0016 (18)	−0.0041 (19)	−0.0096 (18)
O9	0.017 (2)	0.017 (2)	0.014 (2)	0.0063 (19)	0.0000 (18)	−0.0118 (19)
O10	0.018 (3)	0.047 (4)	0.023 (3)	−0.011 (3)	−0.010 (2)	0.020 (3)
O11	0.017 (3)	0.038 (3)	0.014 (2)	−0.011 (2)	−0.004 (2)	0.015 (2)
O12	0.009 (2)	0.015 (2)	0.014 (2)	−0.0035 (17)	−0.0039 (18)	0.0022 (18)
O13	0.016 (2)	0.014 (2)	0.011 (2)	−0.0037 (18)	−0.0029 (18)	0.0007 (17)
O14	0.010 (2)	0.016 (2)	0.016 (2)	−0.0003 (18)	−0.0014 (18)	−0.0045 (19)
O15	0.036 (3)	0.019 (3)	0.023 (3)	0.001 (2)	−0.010 (2)	−0.011 (2)
O16	0.017 (2)	0.016 (2)	0.013 (2)	−0.0001 (19)	−0.0029 (19)	−0.0012 (18)
O17	0.025 (3)	0.009 (2)	0.023 (3)	−0.0035 (19)	−0.009 (2)	0.0064 (19)
N1	0.018 (3)	0.015 (3)	0.013 (3)	−0.001 (2)	−0.004 (2)	0.002 (2)
N2	0.024 (3)	0.011 (3)	0.011 (3)	−0.002 (2)	−0.004 (2)	−0.002 (2)
C1	0.027 (4)	0.017 (4)	0.009 (3)	0.005 (3)	−0.002 (3)	0.004 (3)
C2	0.025 (4)	0.015 (3)	0.014 (3)	0.003 (3)	−0.001 (3)	0.004 (3)
C3	0.027 (4)	0.009 (3)	0.018 (3)	−0.001 (3)	−0.008 (3)	−0.003 (2)
C4	0.035 (4)	0.016 (4)	0.016 (3)	0.001 (3)	−0.004 (3)	−0.003 (3)
C5	0.024 (4)	0.036 (5)	0.034 (5)	−0.012 (4)	−0.011 (3)	0.015 (4)
C6	0.029 (4)	0.016 (4)	0.020 (4)	0.001 (3)	0.004 (3)	−0.004 (3)
C7	0.025 (4)	0.020 (4)	0.020 (4)	−0.004 (3)	−0.008 (3)	0.001 (3)

Geometric parameters (Å, °)

U1—O1	1.758 (5)	N1—C1	1.512 (10)
U1—O2	1.754 (5)	N1—C4	1.501 (10)

U1—O3	2.470 (5)	N1—C5	1.485 (10)
U1—O4	2.441 (5)	N1—H21	1.001
U1—O5	2.333 (5)	N2—C3	1.507 (9)
U1—O6	2.360 (5)	N2—C6	1.507 (10)
U1—O7	2.373 (5)	N2—C7	1.484 (10)
U2—O8	1.765 (5)	N2—H22	1.003
U2—O9	1.770 (5)	C1—C2	1.515 (9)
U2—O10	2.522 (5)	C1—H3	1.006
U2—O11	2.361 (5)	C1—H4	1.003
U2—O12	2.342 (5)	C2—C3	1.541 (10)
U2—O13	2.367 (5)	C2—H5	1.006
U2—O14	2.337 (5)	C2—H6	0.997
S1—O3	1.496 (5)	C3—H7	1.001
S1—O4	1.509 (5)	C3—H8	1.000
S1—O11	1.461 (5)	C4—H9	1.004
S1—O15	1.437 (6)	C4—H10	0.997
S2—O5	1.469 (5)	C4—H11	1.005
S2—O6^{i}	1.497 (5)	C5—H12	1.000
S2—O7^{ii}	1.487 (5)	C5—H13	0.999
S2—O16	1.438 (5)	C5—H14	1.006
S3—O12^{ii}	1.482 (5)	C6—H15	1.002
S3—O13^{iii}	1.480 (5)	C6—H16	1.001
S3—O14	1.492 (5)	C6—H17	1.001
S3—O17	1.440 (5)	C7—H18	1.002
O10—H1	1.001	C7—H19	1.002
O10—H2	1.001	C7—H20	1.000

O1—U1—O2	179.4 (2)	U1—O6—S2^{i}	134.6 (3)
O1—U1—O3	92.8 (2)	U1—O7—S2^{iv}	133.7 (3)
O2—U1—O3	87.4 (2)	U2—O10—H1	125.4
O1—U1—O4	88.92 (19)	U2—O10—H2	109.6
O2—U1—O4	90.76 (19)	H1—O10—H2	125.0
O3—U1—O4	57.28 (16)	U2—O11—S1	155.0 (3)
O1—U1—O5	89.3 (2)	U2—O12—S3^{iv}	144.1 (3)
O2—U1—O5	90.2 (2)	U2—O13—S3^{iii}	143.7 (3)
O3—U1—O5	129.16 (17)	U2—O14—S3	131.3 (3)
O4—U1—O5	72.00 (16)	C1—N1—C4	111.5 (6)
O1—U1—O6	87.57 (19)	C1—N1—C5	114.8 (6)
O2—U1—O6	92.5 (2)	C4—N1—C5	110.0 (7)
O3—U1—O6	151.87 (16)	C1—N1—H21	105.3
O4—U1—O6	150.78 (16)	C4—N1—H21	108.9
O5—U1—O6	78.96 (17)	C5—N1—H21	105.9
O1—U1—O7	90.7 (2)	C3—N2—C6	110.0 (6)
O2—U1—O7	89.9 (2)	C3—N2—C7	112.9 (6)
O3—U1—O7	73.82 (17)	C6—N2—C7	110.7 (6)
O4—U1—O7	131.00 (16)	C3—N2—H22	107.4
O5—U1—O7	156.99 (17)	C6—N2—H22	109.7
O6—U1—O7	78.05 (17)	C7—N2—H22	106.1

O8—U2—O9	178.5 (2)	N1—C1—C2	113.0 (6)
O8—U2—O10	92.3 (2)	N1—C1—H3	108.8
O9—U2—O10	87.5 (2)	C2—C1—H3	108.7
O8—U2—O11	90.5 (2)	N1—C1—H4	108.8
O9—U2—O11	90.7 (2)	C2—C1—H4	108.6
O10—U2—O11	68.36 (18)	H3—C1—H4	108.8
O8—U2—O12	88.8 (2)	C1—C2—C3	107.7 (6)
O9—U2—O12	92.3 (2)	C1—C2—H5	109.8
O10—U2—O12	140.21 (17)	C3—C2—H5	109.7
O11—U2—O12	71.85 (17)	C1—C2—H6	110.1
O8—U2—O13	92.8 (2)	C3—C2—H6	110.2
O9—U2—O13	86.6 (2)	H5—C2—H6	109.3
O10—U2—O13	145.43 (17)	N2—C3—C2	110.2 (6)
O11—U2—O13	145.69 (17)	N2—C3—H7	109.3
O12—U2—O13	74.09 (16)	C2—C3—H7	109.4
O8—U2—O14	88.6 (2)	N2—C3—H8	109.3
O9—U2—O14	90.0 (2)	C2—C3—H8	109.2
O10—U2—O14	70.02 (18)	H7—C3—H8	109.3
O11—U2—O14	138.30 (18)	N1—C4—H9	109.7
O12—U2—O14	149.75 (17)	N1—C4—H10	110.2
O13—U2—O14	75.94 (17)	H9—C4—H10	109.3
O3—S1—O4	103.1 (3)	N1—C4—H11	109.5
O3—S1—O11	108.2 (3)	H9—C4—H11	108.7
O4—S1—O11	107.9 (3)	H10—C4—H11	109.3
O3—S1—O15	112.4 (3)	N1—C5—H12	109.8
O4—S1—O15	112.2 (3)	N1—C5—H13	110.2
O11—S1—O15	112.5 (4)	H12—C5—H13	109.6
O5—S2—O6^{i}	108.2 (3)	N1—C5—H14	109.3
O5—S2—O7^{ii}	107.4 (3)	H12—C5—H14	108.9
O6^{i}—S2—O7^{ii}	106.3 (3)	H13—C5—H14	109.1
O5—S2—O16	110.8 (3)	N2—C6—H15	109.6
O6^{i}—S2—O16	111.9 (3)	N2—C6—H16	109.9
O7^{ii}—S2—O16	112.0 (3)	H15—C6—H16	109.2
O12^{ii}—S3—O13^{iii}	108.6 (3)	N2—C6—H17	109.6
O12^{ii}—S3—O14	104.6 (3)	H15—C6—H17	109.2
O13^{iii}—S3—O14	108.5 (3)	H16—C6—H17	109.3
O12^{ii}—S3—O17	113.1 (3)	N2—C7—H18	109.8
O13^{iii}—S3—O17	110.3 (3)	N2—C7—H19	109.8
O14—S3—O17	111.5 (3)	H18—C7—H19	109.1
U1—O3—S1	98.8 (2)	N2—C7—H20	109.6
U1—O4—S1	99.7 (2)	H18—C7—H20	109.3
U1—O5—S2	155.4 (3)	H19—C7—H20	109.3

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*+1; (ii) *x*−1, *y*, *z*; (iii) −*x*+1, −*y*+1, −*z*+2; (iv) *x*+1, *y*, *z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O10—H1···O3^{ii}	1.00	2.011	2.877 (7)	143

O10—H2···O4	1.00	1.836	2.788 (7)	158
N1—H21···O16^{v}	1.00	2.121	2.850 (8)	128
N2—H22···O15^{vi}	1.00	2.339	2.935 (8)	117
N2—H22···O17^{vii}	1.00	2.212	2.889 (8)	123

Symmetry codes: (ii) *x*−1, *y*, *z*; (v) −*x*+1, −*y*+1, −*z*+1; (vi) *x*−1, *y*−1, *z*; (vii) *x*, *y*−1, *z*.