(C₃H₁₂N₂)₂[UO₂(H₂O)₂(SO₄)₂]₂·2H₂O: an organically templated uranium sulfate with a novel dimer type

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The title compound, bis(propane-1,2-diaminium) tetra-aquadi-μ_2-sulfato-disulfatotetraoxodiuranate(VI) dihydrate, (C₃H₁₂N₂)₂[U₂O₄(SO₄)₄(H₂O)₄]·2H₂O, contains discrete centrosymmetric anionic {[UO₂(H₂O)₂(SO₄)₂]₂}⁴⁻ dimers with C₃H₁₂N₂²⁺ cations balancing the charge. The dimers form hydrogen-bonded layers. The cations and occluded water molecules participate in an extensive hydrogen-bonding network. Each Uᵛᴵ centre is seven-coordinate with a pentagonal–bipyramidal geometry. Both pendent and bridging sulfate tetrahedra are observed, as well as bound and occluded water molecules.

Comment

Hydrothermal synthesis is a well established method for the formation of inorganic structures templated by organic ions. The majority of these compounds are metal phosphates (Cheetham *et al.*, 1999), with other examples including metal phosphites (Doran *et al.*, 2001; Fernandez *et al.*, 2002), fluorides (Walker *et al.*, 1999), germanates (Reisner *et al.*, 2001; Bu *et al.*, 1998; Conradsson *et al.*, 2000), arsenates (Ekambaram & Sevov, 2000; Bazan *et al.*, 2000), oxalates (Vaidhyanathan *et al.*, 2002) and selenites (Choudhury *et al.*, 2002; Harrison *et al.*, 2000).

(I)
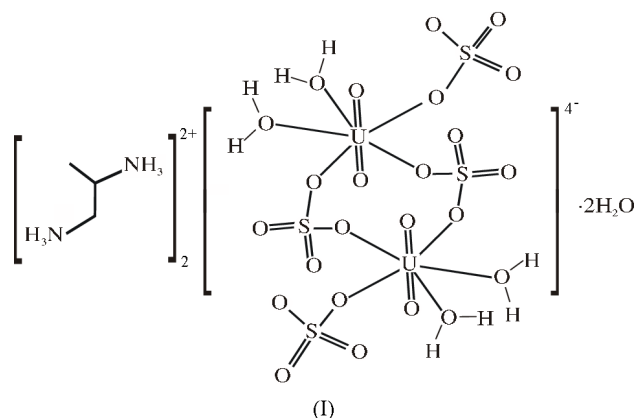
A recently employed strategy for the design of new inorganic architectures involves the use of the sulfate tetrahedron as a primary substituent. Compounds incorporating U (Doran *et al.*, 2002, 2003*a,b,c,d*; Doran, Norquist *et al.*, 2004; Doran, Cockbain *et al.*, 2004; Norquist *et al.*, 2002, 2003*a,b*; Norquist *et al.*, 2003; Thomas *et al.*, 2003; Stuart *et al.*, 2003), Cd (Choudhury *et al.*, 2001; Paul *et al.*, 2002*b*), La (Bataille & Louer, 2002; Xing, Liu *et al.*, 2003; Xing Shi *et al.*, 2003), Ce (Wang *et al.*, 2002), Sc (Bull *et al.*, 2002), Fe (Paul, Choudhury
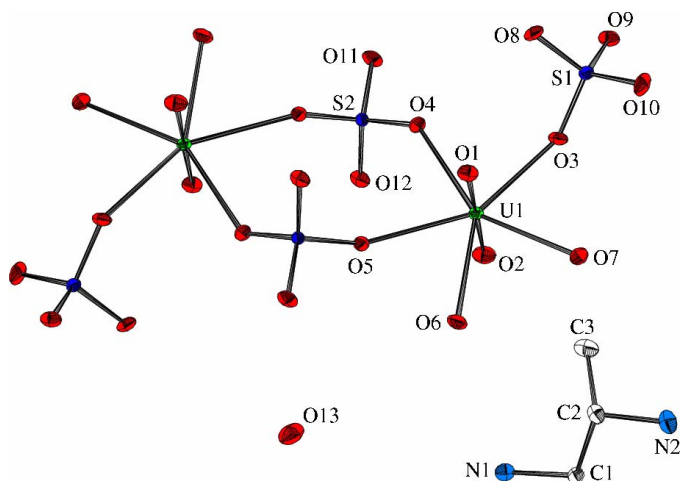
Figure 1
View of the title compound, with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted.

Figure 2
The formation of pseudo-layers by the dimers in USO-31. Green pentagonal bipyramids and blue tetrahedra represent $[UO_7]$ and $[SO_4]$, respectively.

& Rao, 2002a, 2003; Paul et al., 2002), V (Paul, Choudhury, Nagarajan & Rao, 2003; Khan et al., 1999), Zn (Morimoto & Lingafelter, 1970) and Mo (Gutnick et al., 2004) are known. These compounds exhibit great structural diversity, with structures ranging from molecular anions to three-dimensional frameworks. This report contains the synthesis and structure of an organically templated uranium sulfate, $[N_2C_3H_{12}]_2[UO_2(H_2O)_2(SO_4)_2]_2\cdot2H_2O$, (I), designated USO-31 (uranium sulfate from Oxford).

A single independent U atom is present in USO-31. U1 is seven-coordinate (Fig. 1 and Table 1) in a pentagonal–bipyramidal geometry. Two short 'uranyl' bonds to axial O atoms are observed, with U—O distances of 1.765 (3) Å and 1.772 (4) Å, close to the average reported value of 1.758 (3) Å (Burns et al., 1997). The O1—U1—O2 angle is close to 180°, with a value of 178.91 (16)°. Three of the five equatorial coordination sites around U1 are occupied by O atoms of sulfate groups, with U—O distances of 2.335 (3), 2.380 (3) and 2.385 (3) Å. The remaining two equatorial coordination sites are occupied by bound water molecules, with U—O distances of 2.420 (3) and 2.437 (3) Å. The assignment of the bound water molecules was based on the longer U—O bond lengths and hydrogen-bonding interactions. Two distinct sulfur sites are observed in USO-31. S1 and S2 are both at the centre of $[SO_4]$ tetrahedra. S1 tetrahedra link to one U centre and have three terminal O atoms, in contrast with S2 tetrahedra, which bridge between two U centres and have two terminal O atoms. The S—O$_{bridging}$ distances range between 1.490 (3) and 1.500 (3) Å. The S—O$_{terminal}$ distances are shorter, ranging between 1.463 (4) and 1.475 (4) Å.

Centrosymmetric dimers are formed as a result of the connectivities between the $[UO_7]$ and $[SO_4]$ polyhedra. This dimer topology is, to the best of our knowledge, previously unknown in uranium chemistry. It is related to the $[(UO_2)_2(SO_4)_6]^{8-}$ dimers in USO-10 (Norquist et al., 2003a) and USO-12 (Norquist et al., 2003b), which contain edge-shared sulfate groups in place of the bound water molecules of
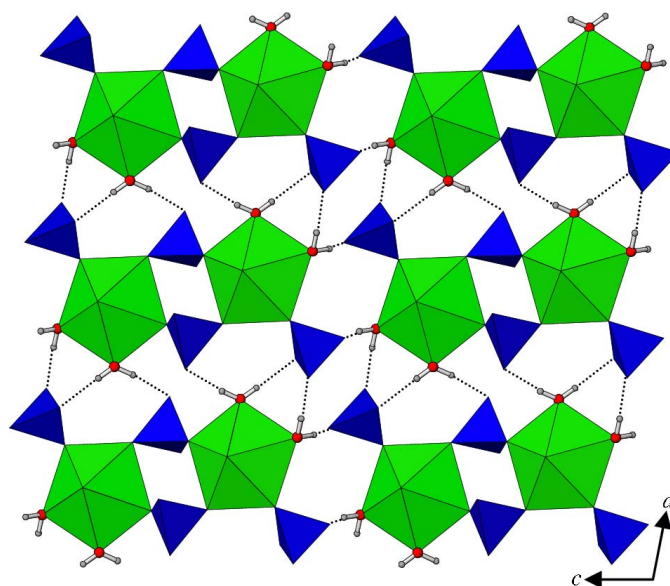
USO-31. Hydrogen-bonded layers are formed (see Fig. 2), because the four bound water molecules of each dimer donate hydrogen bonds to the terminal sulfate O atoms of adjacent dimers. These pseudo-layers propagate in the (010) plane and are separated by template cations and occluded water molecules (see Fig. 3). The interlayer species are involved in hydrogen bonding with the layer (Table 2).

Experimental

Crystal data

$(C_3H_{12}N_2)_2[U_2O_4(SO_4)_4(H_2O)_4]\cdot$	$Z = 1$
$2H_2O$	$D_x = 2.807$ Mg m^{-3}
$M_r = 1184.73$	Mo $K\alpha$ radiation
Triclinic, $P\bar{1}$	Cell parameters from 2953
$a = 7.3983$ (2) Å	reflections
$b = 7.6333$ (2) Å	$\theta = 5–27°$
$c = 12.5946$ (5) Å	$\mu = 11.95$ mm^{-1}
$\alpha = 95.1761$ (12)°	$T = 150$ K
$\beta = 94.6412$ (13)°	Plate, yellow
$\gamma = 96.578$ (2)°	$0.10 \times 0.06 \times 0.01$ mm
$V = 700.70$ (4) Å^3	

Data collection

Nonius KappaCCD diffractometer	2820 reflections with $I > 3\sigma(I)$
ω scans	$R_{int} = 0.02$
Absorption correction: multi-scan	$\theta_{max} = 27.4°$
(Otwinowski & Minor, 1997)	$h = -8 \rightarrow 9$
$T_{min} = 0.46$, $T_{max} = 0.89$	$k = -9 \rightarrow 9$
5870 measured reflections	$l = -16 \rightarrow 16$
3154 independent reflections	

Refinement

Refinement on F^2	(Watkin, 1994), 11.1, 14.6, 7.77,
$R[F^2 > 2\sigma(F^2)] = 0.023$	2.08
$wR(F^2) = 0.056$	$(\Delta/\sigma)_{max} = 0.001$
$S = 0.83$	$\Delta\rho_{max} = 1.22$ e Å^{-3}
2820 reflections	$\Delta\rho_{min} = -1.39$ e Å^{-3}
191 parameters	Extinction correction: Larson
H-atom parameters constrained	(1970)
Modified (Prince, 1982) Chebychev	Extinction coefficient: 12.0 (11)
polynomial with four parameters	

Table 1
Selected geometric parameters (Å, °).

U1—O1	1.765 (3)	S1—O10	1.463 (4)
U1—O2	1.772 (4)	S2—O4	1.493 (4)
U1—O3	2.335 (3)	S2^i—O5	1.490 (3)
U1—O4	2.385 (3)	S2—O11	1.470 (3)
U1—O5	2.380 (3)	S2—O12	1.466 (4)
U1—O6	2.437 (3)	N1—C1	1.498 (6)
U1—O7	2.420 (3)	N2—C2	1.489 (7)
S1—O3	1.500 (3)	C1—C2	1.520 (7)
S1—O8	1.475 (3)	C2—C3	1.530 (7)
S1—O9	1.475 (3)		
O1—U1—O2	178.91 (16)	O6—U1—O7	69.66 (12)
O1—U1—O3	91.88 (14)	O3—S1—O8	109.3 (2)
O1—U1—O4	90.74 (14)	O3—S1—O9	106.8 (2)
O1—U1—O5	92.44 (14)	O3—S1—O10	108.9 (2)
O1—U1—O6	91.42 (14)	O8—S1—O9	109.9 (2)
O1—U1—O7	84.59 (15)	O8—S1—O10	111.2 (2)
O2—U1—O3	88.87 (15)	O9—S1—O10	110.7 (2)
O2—U1—O4	90.22 (15)	O4—S2—O5^i	106.9 (2)
O2—U1—O5	87.36 (15)	O4—S2—O11	107.8 (2)
O2—U1—O6	87.51 (15)	O4—S2—O12	110.8 (2)
O2—U1—O7	94.84 (15)	O5^i—S2—O11	109.6 (2)
O3—U1—O4	74.72 (12)	O5^i—S2—O12	110.0 (2)
O3—U1—O5	147.53 (12)	O11—S2—O12	111.7 (2)
O3—U1—O6	143.92 (12)	U1—O3—S1	138.5 (2)
O3—U1—O7	74.91 (12)	U1—O4—S2	135.8 (2)
O4—U1—O5	73.06 (12)	U1—O5—S2^i	142.6 (2)
O4—U1—O6	141.14 (12)	N1—C1—C2	112.2 (4)
O4—U1—O7	149.08 (12)	N2—C2—C1	106.6 (4)
O5—U1—O6	68.09 (12)	N2—C2—C3	109.1 (4)
O5—U1—O7	137.55 (12)	C1—C2—C3	114.6 (4)

Symmetry code: (i) $-x, -y + 2, -z + 2$.

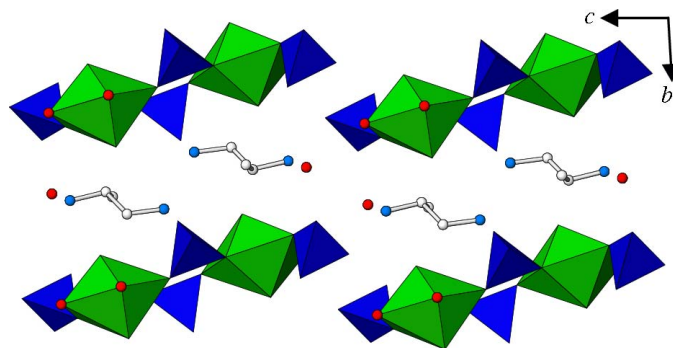**Figure 3**
Three-dimensional packing of USO-31. Green pentagonal bipyramids and blue tetrahedra represent [UO$_7$] and [SO$_4$], respectively. H atoms have been omitted for clarity.

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O6—H1$\cdots$O8^{ii}	1.00	1.71	2.705 (5)	180
O6—H2$\cdots$O11^{ii}	1.00	1.76	2.756 (5)	180
O7—H3$\cdots$O9^{ii}	1.00	1.73	2.732 (5)	180
O7—H4$\cdots$O10^{iii}	1.00	1.67	2.665 (5)	180
N1—H5$\cdots$O12^{iv}	1.00	1.82	2.824 (6)	179
N1—H6$\cdots$O11^i	1.00	1.90	2.846 (5)	156
N1—H7$\cdots$O12^v	1.00	2.18	2.878 (6)	126
N2—H8$\cdots$O13	1.00	1.82	2.812 (6)	170
N2—H9$\cdots$O9^{iv}	1.00	2.01	2.909 (6)	148
N2—H10$\cdots$O8^{ii}	1.00	1.97	2.911 (6)	156
O13—H18$\cdots$O10^{vi}	1.00	1.91	2.909 (5)	180

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

H atoms were placed geometrically after each cycle in idealized locations at 1.00 Å from the carrier atom, such that plausible hydrogen-bonding interactions are made, and refined as riding. The constraint U_{iso}(H) = 1.2U_{eq} (carrier atom) was applied in all cases. The highest peak is 0.96 Å from O1, and the deepest hole is 0.85 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: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ATOMS* (Dowty, 2000); software used to prepare material for publication: *CRYSTALS* (Betteridge *et al.*, 2003).

References

Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.
Bataille, T. & Louer, D. (2002). *J. Mater. Chem.* **12**, 3487–3493.
Bazan, B., Mesa, J. L., Pizarro, J. L., Lezama, L., Arriortua, M. I. & Rojo, T. (2000). *Inorg. Chem.* **39**, 6056–6060.
Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.

Bu, X., Feng, P., Gier, T. E., Zhao, D. & Stucky, G. D. (1998). *J. Am. Chem. Soc.* **120**, 13389–13397.

Bull, I., Wheatley, P. S., Lightfoot, P., Morris, R. E., Sastre, E. & Wright, P. A. (2002). *Chem. Commun.* pp. 1180–1181.

Burns, P. C., Ewing, R. C. & Hawthorne, F. C. (1997). *Can. Mineral.* **35**, 1551–1570.

Cheetham, A. K., Ferey, G. & Loiseau, T. (1999). *Angew. Chem. Int. Ed.* **38**, 3268–3292.

Choudhury, A., Krishnamoorthy, J. & Rao, C. N. R. (2001). *Chem. Commun.* pp. 2610–2611.

Choudhury, A., Kumar, D. U. & Rao, C. N. R. (2002). *Angew. Chem. Int. Ed.* **41**, 158–161.

Conradsson, T., Zou, X. & Dadachov, M. S. (2000). *Inorg. Chem.* **39**, 1716–1720.

Doran, M., Walker, S. M. & O'Hare, D. (2001). *Chem. Commun.* pp. 1988–1989.

Doran, M. B., Cockbain, B. E., Norquist, A. J. & O'Hare, D. (2004). *Dalton Trans.* pp. 3810–3814.

Doran, M. B., Norquist, A. J. & O'Hare, D. (2002). *Chem. Commun.* pp. 2946–2947.

Doran, M. B., Norquist, A. J. & O'Hare, D. (2003*a*). *Inorg. Chem.* **42**, 6989–6995.

Doran, M. B., Norquist, A. J. & O'Hare, D. (2003*b*). *Acta Cryst.* E**59**, m373–m375.

Doran, M. B., Norquist, A. J. & O'Hare, D. (2003*c*). *Acta Cryst.* E**59**, m762–m764.

Doran, M. B., Norquist, A. J. & O'Hare, D. (2003*d*). *Acta Cryst.* E**59**, m765–m767.

Doran, M. B., Norquist, A. J., Stuart, C. S. & O'Hare, D. (2004). *Acta Cryst.* E**60**, m996–m998.

Dowty, E. (2000). *ATOMS*. Version 6.0. Shape Software, 521 Hidden Valley Road, Kingsport, TN 37663, USA.

Ekambaram, S. & Sevov, S. C. (2000). *Inorg. Chem.* **39**, 2405–2410.

Fernandez, S., Mesa, J. L., Pizarro, J. L., Lezama, L., Arriortua, M. I. & Rojo, T. (2002). *Chem. Mater.* **14**, 2300–2307.

Gutnick, J. R., Muller, E. A., Sarjeant, A. N. & Norquist, A. J. (2004). *Inorg. Chem.* **43**, 6528–6530.

Harrison, W. T. A., Phillips, M. L. F., Stanchfield, J. & Nenoff, T. M. (2000). *Angew. Chem. Int. Ed.* **39**, 3808–3810.

Khan, M. I., Cevik, S. & Doedens, R. J. (1999). *Inorg. Chim. Acta*, **292**, 112–116.

Larson, A. C. (1970). *Crystallographic Computing*, edited by F. R. Ahmed, S. R. Hall & C. P. Huber, pp. 291–294. Copenhagen: Munksgaard.

Morimoto, C. N. & Lingafelter, E. C. (1970). *Acta Cryst.* B**26**, 335–341.

Nonius (2001). *COLLECT*. Nonius BV, Delft, The Netherlands.

Norquist, A. J., Doran, M. B. & O'Hare, D. (2003). *Solid State Sci.* **5**, 1149–1158.

Norquist, A. J., Doran, M. B., Thomas, P. M. & O'Hare, D. (2003*a*). *Inorg. Chem.* **42**, 5949–5953.

Norquist, A. J., Doran, M. B., Thomas, P. M. & O'Hare, D. (2003*b*). *J. Chem. Soc. Dalton Trans.* pp. 1168–1175.

Norquist, A. J., Thomas, P. M., Doran, M. B. & O'Hare, D. (2002). *Chem. Mater.* **14**, 5179–5184.

Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.

Paul, G., Choudhury, A., Nagarajan, R. & Rao, C. N. R. (2003). *Inorg. Chem.* **42**, 2004–2013.

Paul, G., Choudhury, A. & Rao, C. N. R. (2002*a*). *Chem. Commun.* pp. 1904–1905.

Paul, G., Choudhury, A. & Rao, C. N. R. (2002*b*). *J. Chem. Soc.* pp. 3859–3867.

Paul, G., Choudhury, A., & Rao, C. N. R. (2003). *Chem. Mater.* **15**, 1174–1180.

Paul, G., Choudhury, A., Sampathkumaran, E. V. & Rao, C. N. R. (2002). *Angew. Chem. Int. Ed.* **41**, 4297–4300.

Prince, E. (1982). *Mathematical Techniques in Crystallography and Materials Science.* New York: Springer-Verlag.

Reisner, B. A., Tripathi, A. & Parise, J. B. (2001). *J. Mater. Chem.* **11**, 887–890.

Stuart, C. L., Doran, M. B., Norquist, A. J. & O'Hare, D. (2003). *Acta Cryst.* E**59**, m446–m448.

Thomas, P. M., Norquist, A. J., Doran, M. B. & O'Hare, D. (2003). *J. Mater. Chem.* **13**, 88–92.

Vaidhyanathan, R., Natarajan, S. & Rao, C. N. R. (2002). *Inorg. Chem.* **41**, 4496–4501.

Walker, S. M., Halasyamani, P. S., Allen, S. & O'Hare, D. (1999). *J. Am. Chem. Soc.* **121**, 10513–10521.

Wang, D., Yu, R., Xu, Y., Feng, S., Xu, R., Kumada, N., Kinomura, N., Matumura, Y. & Takano, M. (2002). *Chem. Lett.* pp. 1120–1121.

Watkin, D. J. (1994). *Acta Cryst.* A**50**, 411–437.

Xing, Y., Liu, Y., Shi, Z., Meng, H. & Pang, W. (2003). *J. Solid State Chem.* **174**, 381–385.

Xing, Y., Shi, Z., Li, G. & Pang, W. (2003). *J. Chem. Soc. Dalton Trans.* pp. 940–943.

supporting information

(C₃H₁₂N₂)₂[UO₂(H₂O)₂(SO₄)₂]₂·2H₂O: an organically templated uranium sulfate with a novel dimer type

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

Bis(propane-1,2-diaminium) tetraaquadi-μ_2-sulfato-disulfatotetraoxodiuranate(VI) dihydrate

Crystal data

(C₃H₁₂N₂)₂[U₂O₄(SO₄)₄(H₂O)₄]·2H₂O	$Z = 1$
$M_r = 1184.73$	$F(000) = 556.000$
Triclinic, $P\bar{1}$	$D_x = 2.807$ Mg m⁻³
Hall symbol: -P 1	Melting point: not measured K
$a = 7.3983\ (2)$ Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 7.6333\ (2)$ Å	Cell parameters from 2953 reflections
$c = 12.5946\ (5)$ Å	$\theta = 5$–27°
$\alpha = 95.1761\ (12)$°	$\mu = 11.95$ mm⁻¹
$\beta = 94.6412\ (13)$°	$T = 150$ K
$\gamma = 96.578\ (2)$°	Plate, yellow
$V = 700.70\ (4)$ Å³	$0.10 \times 0.06 \times 0.01$ mm

Data collection

Nonius KappaCCD diffractometer	3154 independent reflections
Graphite monochromator	2820 reflections with $I > 3u(I)$
ω scans	$R_{int} = 0.02$
Absorption correction: multi-scan (Otwinowski & Minor, 1997)	$\theta_{max} = 27.4$°, $\theta_{min} = 5.4$°
$T_{min} = 0.46$, $T_{max} = 0.89$	$h = -8 \rightarrow 9$
5870 measured reflections	$k = -9 \rightarrow 9$
	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.023$	*P*rince (1982) modified Chebychev polynomial with four parameters (Watkin, 1994), 11.1, 14.6, 7.77, 2.08
$wR(F^2) = 0.056$	
$S = 0.83$	
2820 reflections	$(\Delta/\sigma)_{max} = 0.001$
191 parameters	$\Delta\rho_{max} = 1.22$ e Å⁻³
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{min} = -1.39$ e Å⁻³
	Extinction correction: Larson (1970)
	Extinction coefficient: 12.0 (11)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
H1	0.4926	0.9562	0.7360	0.0200*
H2	0.4688	0.8838	0.8573	0.0200*
H3	0.3216	0.7782	0.5899	0.0169*
H4	0.2260	0.9490	0.5713	0.0169*
H5	0.7946	1.4761	0.9702	0.0158*
H6	0.6236	1.3183	0.9617	0.0158*
H7	0.8328	1.2725	0.9840	0.0158*
H8	0.7950	1.4003	0.6341	0.0211*
H9	0.5882	1.4470	0.6040	0.0211*
H10	0.6208	1.2473	0.6315	0.0211*
H11	0.8955	1.3462	0.8126	0.0138*
H12	0.7246	1.1884	0.8040	0.0138*
H13	0.6912	1.5557	0.7874	0.0156*
H14	0.3772	1.4821	0.7399	0.0245*
H15	0.3931	1.2777	0.7645	0.0245*
H16	0.4326	1.4375	0.8619	0.0245*
H17	1.0886	1.4892	0.6554	0.0311*
H18	1.1066	1.3173	0.5699	0.0311*
N1	0.7531 (6)	1.3485 (6)	0.9457 (3)	0.0129
C3	0.4450 (7)	1.4043 (8)	0.7844 (5)	0.0204
C1	0.7655 (7)	1.3164 (6)	0.8276 (4)	0.0115
C2	0.6471 (7)	1.4282 (6)	0.7642 (4)	0.0133
N2	0.6642 (7)	1.3764 (6)	0.6490 (4)	0.0178
O1	0.0636 (5)	1.0956 (5)	0.7338 (3)	0.0130
O2	0.0987 (5)	0.6631 (5)	0.8066 (3)	0.0165
O3	−0.1697 (5)	0.7613 (5)	0.6487 (3)	0.0131
O4	−0.1830 (5)	0.8925 (5)	0.8680 (3)	0.0118
O5	0.1872 (5)	0.9863 (5)	0.9501 (3)	0.0118
O6	0.4115 (5)	0.9355 (5)	0.7946 (3)	0.0164
O7	0.2060 (5)	0.8312 (5)	0.6002 (3)	0.0138
O8	−0.3691 (5)	0.9913 (4)	0.6361 (3)	0.0129
O9	−0.4780 (5)	0.6865 (5)	0.5722 (3)	0.0140
O10	−0.2593 (6)	0.8546 (5)	0.4764 (3)	0.0183
O11	−0.4306 (5)	0.7929 (5)	0.9675 (3)	0.0143
O12	−0.1290 (5)	0.7093 (5)	1.0132 (3)	0.0135
O13	1.0267 (6)	1.4074 (6)	0.5942 (3)	0.0246
U1	0.07888 (2)	0.87952 (2)	0.770225 (14)	0.0086
S1	−0.32052 (15)	0.82608 (15)	0.58177 (9)	0.0089
S2	−0.23260 (15)	0.84808 (15)	0.97579 (9)	0.0081

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0119 (19)	0.013 (2)	0.014 (2)	0.0007 (15)	0.0033 (15)	0.0043 (16)
C3	0.013 (2)	0.023 (3)	0.025 (3)	0.003 (2)	−0.001 (2)	0.003 (2)

C1	0.014 (2)	0.008 (2)	0.012 (2)	0.0035 (17)	−0.0001 (17)	−0.0002 (17)
C2	0.016 (2)	0.010 (2)	0.013 (2)	0.0004 (18)	−0.0015 (18)	0.0016 (17)
N2	0.023 (2)	0.016 (2)	0.013 (2)	0.0009 (17)	−0.0013 (17)	0.0016 (16)
O1	0.0101 (16)	0.0144 (17)	0.0154 (17)	0.0006 (13)	0.0024 (13)	0.0059 (13)
O2	0.0143 (17)	0.0122 (17)	0.0233 (19)	0.0029 (13)	−0.0001 (14)	0.0039 (14)
O3	0.0085 (16)	0.0141 (17)	0.0168 (18)	0.0037 (13)	−0.0014 (13)	0.0020 (13)
O4	0.0097 (16)	0.0163 (17)	0.0100 (16)	0.0020 (13)	0.0039 (12)	0.0017 (13)
O5	0.0089 (16)	0.0126 (16)	0.0130 (17)	0.0013 (12)	−0.0003 (12)	−0.0021 (13)
O6	0.0082 (16)	0.028 (2)	0.0132 (17)	0.0038 (14)	−0.0002 (13)	0.0038 (15)
O7	0.0109 (16)	0.0180 (17)	0.0127 (17)	0.0016 (13)	0.0041 (13)	0.0007 (14)
O8	0.0110 (16)	0.0101 (16)	0.0184 (18)	0.0042 (13)	0.0043 (13)	−0.0005 (13)
O9	0.0091 (16)	0.0102 (16)	0.0216 (19)	−0.0003 (13)	−0.0011 (13)	0.0005 (14)
O10	0.028 (2)	0.0224 (19)	0.0075 (17)	0.0103 (16)	0.0077 (15)	0.0044 (14)
O11	0.0054 (15)	0.0216 (18)	0.0149 (17)	−0.0030 (13)	0.0001 (13)	0.0039 (14)
O12	0.0122 (16)	0.0115 (16)	0.0164 (18)	0.0012 (13)	−0.0021 (13)	0.0023 (13)
O13	0.027 (2)	0.025 (2)	0.025 (2)	0.0145 (17)	0.0071 (17)	0.0060 (17)
U1	0.0068 (1)	0.0095 (1)	0.0095 (1)	0.00108 (6)	0.00123 (6)	0.00091 (6)
S1	0.0082 (5)	0.0090 (5)	0.0096 (5)	0.0013 (4)	0.0013 (4)	0.0006 (4)
S2	0.0063 (5)	0.0104 (5)	0.0074 (5)	0.0002 (4)	0.0010 (4)	0.0003 (4)

Geometric parameters (Å, º)

U1—O1	1.765 (3)	O6—H1	1.000
U1—O2	1.772 (4)	O6—H2	1.000
U1—O3	2.335 (3)	O7—H3	1.000
U1—O4	2.385 (3)	O7—H4	1.000
U1—O5	2.380 (3)	O13—H17	1.000
U1—O6	2.437 (3)	O13—H18	1.000
U1—O7	2.420 (3)	N1—H5	1.000
S1—O3	1.500 (3)	N1—H6	1.000
S1—O8	1.475 (3)	N1—H7	1.000
S1—O9	1.475 (4)	N2—H8	1.000
S1—O10	1.463 (4)	N2—H9	1.000
S2—O4	1.493 (4)	N2—H10	1.000
S2^i—O5	1.490 (3)	C1—H11	1.000
S2—O11	1.470 (3)	C1—H12	1.000
S2—O12	1.466 (4)	C2—H13	1.000
N1—C1	1.498 (6)	C3—H14	1.000
N2—C2	1.489 (7)	C3—H15	1.000
C1—C2	1.520 (7)	C3—H16	1.000
C2—C3	1.530 (7)		
O1—U1—O2	178.91 (16)	N2—C2—C1	106.6 (4)
O1—U1—O3	91.88 (14)	N2—C2—C3	109.1 (4)
O1—U1—O4	90.74 (14)	C1—C2—C3	114.6 (4)
O1—U1—O5	92.44 (14)	H1—O6—H2	114.895
O1—U1—O6	91.42 (14)	H1—O6—U1	125.07
O1—U1—O7	84.59 (15)	H2—O6—U1	114.75

O2—U1—O3	88.87 (15)	H3—O7—H4	104.304
O2—U1—O4	90.22 (15)	H3—O7—U1	124.91
O2—U1—O5	87.36 (15)	H4—O7—U1	107.01
O2—U1—O6	87.51 (15)	H17—O13—H18	111.243
O2—U1—O7	94.84 (15)	H5—N1—H6	109.476
O3—U1—O4	74.72 (12)	H5—N1—H7	109.476
O3—U1—O5	147.53 (12)	H6—N1—H7	109.476
O3—U1—O6	143.92 (12)	H5—N1—C1	109.5
O3—U1—O7	74.91 (12)	H6—N1—C1	109.4
O4—U1—O5	73.06 (12)	H7—N1—C1	109.5
O4—U1—O6	141.14 (12)	H14—C3—H15	109.476
O4—U1—O7	149.08 (12)	H14—C3—H16	109.476
O5—U1—O6	68.09 (12)	H15—C3—H16	109.476
O5—U1—O7	137.55 (12)	H14—C3—C2	109.5
O6—U1—O7	69.66 (12)	H15—C3—C2	109.4
O3—S1—O8	109.3 (2)	H16—C3—C2	109.5
O3—S1—O9	106.8 (2)	H11—C1—H12	109.467
O3—S1—O10	108.9 (2)	H11—C1—N1	108.8
O8—S1—O9	109.9 (2)	H12—C1—N1	108.8
O8—S1—O10	111.2 (2)	H11—C1—C2	108.8
O9—S1—O10	110.7 (2)	H12—C1—C2	108.8
O4—S2—O5^i	106.9 (2)	H13—C2—C3	105.4
O4—S2—O11	107.8 (2)	H13—C2—C1	107.9
O4—S2—O12	110.8 (2)	H13—C2—N2	113.5
O5^i—S2—O11	109.6 (2)	H8—N2—H9	109.475
O5^i—S2—O12	110.0 (2)	H8—N2—H10	109.476
O11—S2—O12	111.7 (2)	H9—N2—H10	109.476
U1—O3—S1	138.5 (2)	H8—N2—C2	109.5
U1—O4—S2	135.8 (2)	H9—N2—C2	109.4
U1—O5—S2^i	142.6 (2)	H10—N2—C2	109.5
N1—C1—C2	112.2 (4)		

Symmetry code: (i) −x, −y+2, −z+2.

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
O6—H1···O8^{ii}	1.00	1.71	2.705 (5)	180
O6—H2···O11^{ii}	1.00	1.76	2.756 (5)	180
O7—H3···O9^{ii}	1.00	1.73	2.732 (5)	180
O7—H4···O10^{iii}	1.00	1.67	2.665 (5)	180
N1—H5···O12^{iv}	1.00	1.82	2.824 (6)	179
N1—H6···O11^i	1.00	1.90	2.846 (5)	156
N1—H7···O12^v	1.00	2.18	2.878 (6)	126
N2—H8···O13	1.00	1.82	2.812 (6)	170
N2—H9···O9^{iv}	1.00	2.01	2.909 (6)	148

N2—H10···O8[ii]	1.00	1.97	2.911 (6)	156
O13—H18···O10[vi]	1.00	1.91	2.909 (5)	180

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