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Di-μ-acetato-bis[triaquabis(thiocyanato-κN)(4-methylpyridine *N*-oxide-κO)-samarium(III)]

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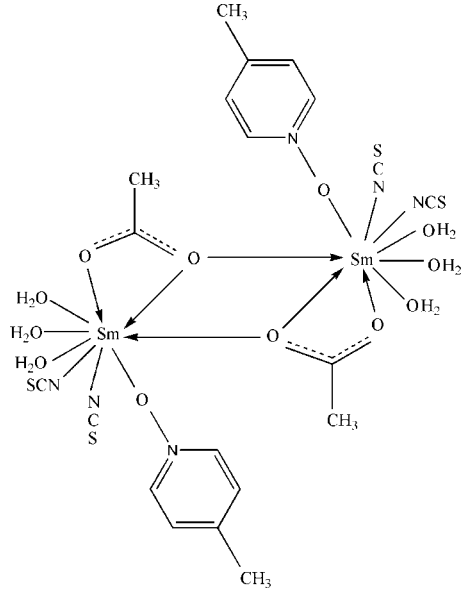
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.028; wR factor = 0.068; data-to-parameter ratio = 18.6.

In the title binuclear complex, $[Sm_2(\mu\text{-}C_2H_3O_2)_2(NCS)_4(C_6H_7NO)_2(H_2O)_6]$, which possesses a crystallographically imposed centre of symmetry, four thiocyanate, two 4-methylpyridine *N*-oxide and six water molecules function as terminal ligands, whereas two acetate anions bridge the two Sm^{III} ions. The crystal packing exhibits an extensive three-dimensional network of intermolecular O—H···S and O—H···O hydrogen bonds.

Related literature

For the crystal structures of related complexes, see: Panagiotopoulos *et al.* (1995); Zhang *et al.* (2006).



Experimental

Crystal data

$[Sm_2(C_2H_3O_2)_2(NCS)_4(C_6H_7NO)_2(H_2O)_6]$
M_r = 977.46
Monoclinic, $P2_1/n$
a = 9.0451 (12) Å
b = 15.997 (2) Å
c = 12.1753 (16) Å
β = 100.910 (2)°
V = 1729.9 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 3.66 mm^{-1}
T = 298 (2) K
0.26 × 0.11 × 0.09 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.449, T_{max} = 0.734
10164 measured reflections
3736 independent reflections
3447 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.068
S = 1.06
3736 reflections
201 parameters
9 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.57 e Å^{-3}
$\Delta\rho_{min}$ = −1.28 e Å^{-3}

Table 1
Selected bond lengths (Å).

Sm1—O2^{i}	2.390 (2)	Sm1—O4	2.467 (2)
Sm1—O2	2.908 (3)	Sm1—N2	2.479 (3)
Sm1—O1	2.398 (2)	Sm1—N3	2.508 (3)
Sm1—O6	2.416 (3)	Sm1—O3	2.530 (3)
Sm1—O5	2.443 (2)		

Symmetry code: (i) $-x+2, -y, -z+1$.

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

D—H···A	D—H	H···A	D···A	D—H···A
O6—H12···S2^{ii}	0.89	2.35	3.240 (3)	172
O3—H8···S1^{iii}	0.88	2.46	3.325 (3)	168
O4—H10···S1^{iv}	0.89	2.39	3.279 (2)	178
O6—H13···O5^{iv}	0.89	1.78	2.674 (3)	177
O4—H11···O1^{i}	0.89	1.79	2.685 (3)	177
O3—H7···S2^{ii}	0.90	2.83	3.643 (3)	152

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2243).

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supplementary materials

Acta Cryst. (2007). E**63**, m1775-m1776 [**doi:10.1107/S1600536807025561**]

Di-μ-acetato-bis[triaquabis(thiocyanato-κN)(4-methylpyridine *N*-oxide-κO)samarium(III)]

S.-G. Zhang and J.-M. Shi

Comment

Acetate, thiocyanate and pyridine N-oxide (or its derivatives) are very useful bridging ligands and many multi-nuclear complexes have been synthesized using these ligands (Panagiotopoulos *et al.*, 1995; Zhang *et al.*, 2006). Some of these complexes display interesting physical properties. We are interested in compounds containing the three types of ligands and hence we have synthesize the title complex, (I), whose crystal structure is reported here.

The asymmetric unit and symmetry-related fragments of (I) are shown in Fig. 1. In the binuclear structure each Sm^{III} ion is coordinated by nine atoms and Table 1 shows the relative coordinated bond lengths and associated angles, and the distance between Sm1 to $Sm1^{i}$ is 4.5032 (5) Å. Atoms Sm1, O2, $Sm1^{i}$, $O2^{i}$ [symmetry code: (i) $-x + 2, -y, -z + 1$] are strictly coplanar and form a parallelogram by virtue of the crystallographic inversion center which is at the middle of the parallelogram. The hydrogen bonds (Table 2) from O atom and S1 atom made the binuclear units connect each other and form the supermolecular two-dimensional sheets on *ac* plane, and the hydrogen bonds dealing with S2 atom made the sheets pile up along *b* axis and led to the formation of the supermolecular three-dimensional structure.

Experimental

$Sm(ClO_4)_3{\cdot}6H_2O$ (0.3128 g, 0.562 mmol), NaSCN (0.0926 g, 1.14 mmol), 4-methylpyridine N-oxide (0.0637 g, 0.584 mmol) and $Na(CH_3COO)$ (0.0480 g, 0.585 mmol) were dissolved in 5 ml of water, respectively, and then the four kinds of solutions were mixed together and stirred for a few minutes. The colourless transparent single crystals were obtained after allowing the solution to stand for two weeks at room temperature.

Refinement

The H atoms from H_2O were found in a difference Fourier map, and placed in idealized positions with O—H = 0.884–0.899 Å. The C-bound H atoms were placed in calculated positions, C—H = 0.93–0.96 Å. All H atoms were refined as riding, with $U_{iso}(H) = 1.2–1.5U_{eq}(C,O)$.

Figures

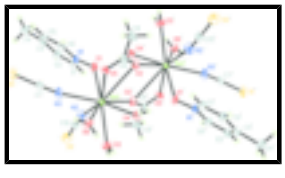


Fig. 1. Molecular structure of (I) showing the atom numbering scheme with displacement ellipsoids drawn at the 30% probability level [symmetry code: (i) $-x + 2, -y, -z + 1$].

Di-μ-acetato-bis[triaquabis(thiocyanato-κ*N*)(4-methylpyridine *N*-oxide-κ*O*)samarium(III)]

Crystal data

$[Sm_2(C_2H_3O_2)_2(NCS)_4(C_6H_7NO)_2(H_2O)_6]$	$F_{000} = 956$
$M_r = 977.46$	$D_x = 1.877$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 5767 reflections
$a = 9.0451$ (12) Å	$\theta = 2.6$–$27.1°$
$b = 15.997$ (2) Å	$\mu = 3.66$ mm^{-1}
$c = 12.1753$ (16) Å	$T = 298$ (2) K
$\beta = 100.910$ (2)°	Prism, colourless
$V = 1729.9$ (4) Å^3	0.26 × 0.11 × 0.09 mm
$Z = 2$	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	3736 independent reflections
Radiation source: fine-focus sealed tube	3447 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.027$
$T = 298$(2) K	$\theta_{max} = 27.1°$
φ and ω scans	$\theta_{min} = 2.1°$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -11 \rightarrow 11$
$T_{min} = 0.449$, $T_{max} = 0.734$	$k = -20 \rightarrow 19$
10164 measured reflections	$l = -14 \rightarrow 15$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.028$	H-atom parameters constrained
$wR(F^2) = 0.068$	$w = 1/[\sigma^2(F_o^2) + (0.035P)^2 + 0.8001P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} = 0.002$
3736 reflections	$\Delta\rho_{max} = 0.57$ e Å^{-3}
201 parameters	$\Delta\rho_{min} = -1.28$ e Å^{-3}
9 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

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

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O2	0.9416 (3)	−0.07880 (15)	0.5244 (2)	0.0362 (6)
Sm1	0.806933 (17)	0.086888 (10)	0.509341 (13)	0.02443 (7)
O4	0.7952 (2)	0.02100 (14)	0.32466 (18)	0.0323 (5)
H10	0.7299	−0.0177	0.2925	0.049*
H11	0.8848	−0.0011	0.3212	0.049*
N1	0.9373 (3)	0.07642 (17)	0.7955 (2)	0.0306 (6)
O5	0.7100 (2)	−0.05216 (15)	0.5392 (2)	0.0331 (5)
O1	0.9396 (3)	0.04823 (17)	0.69183 (19)	0.0391 (6)
N3	0.6276 (3)	0.1216 (2)	0.6366 (3)	0.0446 (8)
C8	0.8150 (4)	−0.1040 (2)	0.5394 (3)	0.0283 (7)
O3	0.7847 (3)	0.19595 (16)	0.3566 (2)	0.0460 (7)
H7	0.7405	0.1789	0.2879	0.069*
H8	0.8383	0.2391	0.3414	0.069*
C7	0.5460 (4)	0.1247 (2)	0.6986 (3)	0.0341 (7)
C1	1.0394 (4)	0.1337 (2)	0.8404 (3)	0.0389 (8)
H1	1.1054	0.1563	0.7984	0.047*
C5	0.8410 (5)	0.0694 (2)	0.9602 (3)	0.0432 (9)
H5	0.7705	0.0480	0.9993	0.052*
C10	0.9366 (4)	0.2917 (2)	0.6101 (3)	0.0382 (8)
C3	0.9481 (5)	0.1260 (2)	1.0121 (3)	0.0434 (9)
C6	0.8381 (4)	0.0448 (2)	0.8523 (3)	0.0375 (8)
H6	0.7670	0.0061	0.8187	0.045*
C2	1.0452 (5)	0.1585 (2)	0.9491 (3)	0.0435 (9)
H2	1.1162	0.1980	0.9806	0.052*
N2	0.8969 (4)	0.2242 (2)	0.5898 (3)	0.0535 (9)
S1	0.43595 (12)	0.12573 (7)	0.79119 (9)	0.0475 (3)
S2	0.99454 (17)	0.38766 (8)	0.63505 (10)	0.0637 (3)
O6	0.5521 (3)	0.09437 (17)	0.4048 (2)	0.0448 (7)
H12	0.5291	0.0953	0.3302	0.067*
H13	0.4642	0.0822	0.4246	0.067*
C9	0.7879 (5)	−0.1937 (2)	0.5573 (5)	0.0588 (12)
H9A	0.6817	−0.2046	0.5412	0.088*
H9B	0.8279	−0.2081	0.6337	0.088*

H9C	0.8367	−0.2266	0.5086	0.088*
C4	0.9579 (7)	0.1507 (3)	1.1326 (3)	0.0727 (15)
H4A	1.0616	0.1521	1.1695	0.109*
H4B	0.9044	0.1106	1.1690	0.109*
H4C	0.9139	0.2050	1.1363	0.109*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0236 (13)	0.0450 (15)	0.0414 (14)	−0.0097 (10)	0.0099 (10)	−0.0056 (11)
Sm1	0.01875 (10)	0.02561 (11)	0.02988 (11)	−0.00155 (6)	0.00701 (7)	−0.00335 (6)
O4	0.0243 (12)	0.0361 (13)	0.0362 (12)	0.0029 (10)	0.0047 (9)	−0.0075 (10)
N1	0.0266 (15)	0.0358 (16)	0.0292 (14)	0.0054 (11)	0.0046 (11)	−0.0044 (11)
O5	0.0252 (12)	0.0286 (13)	0.0468 (14)	0.0006 (10)	0.0106 (10)	0.0021 (11)
O1	0.0342 (13)	0.0556 (17)	0.0266 (12)	0.0100 (12)	0.0035 (10)	−0.0128 (11)
N3	0.0327 (17)	0.058 (2)	0.0454 (18)	0.0062 (15)	0.0121 (14)	−0.0104 (16)
C8	0.0235 (17)	0.0291 (17)	0.0329 (16)	−0.0043 (12)	0.0071 (13)	−0.0037 (13)
O3	0.0601 (18)	0.0334 (14)	0.0464 (15)	−0.0098 (12)	0.0150 (13)	0.0047 (11)
C7	0.0272 (18)	0.0320 (18)	0.0424 (19)	0.0024 (14)	0.0053 (15)	−0.0110 (15)
C1	0.033 (2)	0.047 (2)	0.0371 (19)	−0.0063 (16)	0.0087 (15)	−0.0063 (16)
C5	0.045 (2)	0.044 (2)	0.045 (2)	0.0064 (18)	0.0211 (18)	0.0085 (17)
C10	0.038 (2)	0.042 (2)	0.0319 (18)	−0.0025 (16)	0.0010 (15)	−0.0086 (15)
C3	0.062 (3)	0.035 (2)	0.0339 (19)	0.0112 (18)	0.0124 (18)	−0.0001 (15)
C6	0.0327 (19)	0.036 (2)	0.044 (2)	−0.0011 (15)	0.0068 (15)	0.0009 (16)
C2	0.051 (2)	0.040 (2)	0.038 (2)	−0.0063 (18)	0.0048 (17)	−0.0068 (16)
N2	0.057 (2)	0.041 (2)	0.062 (2)	−0.0124 (17)	0.0105 (18)	−0.0147 (17)
S1	0.0469 (6)	0.0438 (6)	0.0595 (6)	−0.0067 (4)	0.0302 (5)	−0.0165 (5)
S2	0.0994 (10)	0.0416 (6)	0.0495 (6)	−0.0245 (6)	0.0127 (6)	−0.0128 (5)
O6	0.0218 (13)	0.072 (2)	0.0411 (15)	0.0032 (12)	0.0088 (11)	0.0116 (12)
C9	0.041 (2)	0.032 (2)	0.106 (4)	−0.0019 (17)	0.020 (2)	0.009 (2)
C4	0.122 (5)	0.061 (3)	0.038 (2)	0.014 (3)	0.023 (3)	−0.005 (2)

Geometric parameters (Å, °)

O2—C8	1.259 (4)	C7—S1	1.639 (4)
Sm1—O2^{i}	2.390 (2)	C1—C2	1.374 (5)
Sm1—O2	2.908 (3)	C1—H1	0.9300
Sm1—O2^{i}	2.390 (2)	C5—C6	1.367 (5)
Sm1—O1	2.398 (2)	C5—C3	1.388 (6)
Sm1—O6	2.416 (3)	C5—H5	0.9300
Sm1—O5	2.443 (2)	C10—N2	1.150 (5)
Sm1—O4	2.467 (2)	C10—S2	1.632 (4)
Sm1—N2	2.479 (3)	C3—C2	1.372 (5)
Sm1—N3	2.508 (3)	C3—C4	1.506 (5)
Sm1—O3	2.530 (3)	C6—H6	0.9300
O4—H10	0.8934	C2—H2	0.9300
O4—H11	0.8932	O6—H12	0.8937
N1—C6	1.332 (4)	O6—H13	0.8948

N1—C1	1.341 (5)	C9—H9A	0.9600
N1—O1	1.344 (3)	C9—H9B	0.9600
O5—C8	1.260 (4)	C9—H9C	0.9600
N3—C7	1.153 (4)	C4—H4A	0.9600
C8—C9	1.479 (5)	C4—H4B	0.9600
O3—H7	0.8987	C4—H4C	0.9600
O3—H8	0.8839		
C8—O2—Sm1^{i}	158.2 (2)	C1—N1—O1	118.7 (3)
C8—O2—Sm1	85.3 (2)	C8—O5—Sm1	107.9 (2)
Sm1^{i}—O2—Sm1	116.07 (9)	N1—O1—Sm1	133.61 (19)
O2^{i}—Sm1—O1	79.62 (9)	C7—N3—Sm1	169.5 (3)
O2^{i}—Sm1—O6	139.14 (9)	O2—C8—O5	119.7 (3)
O1—Sm1—O6	139.55 (9)	O2—C8—C9	120.8 (3)
O2^{i}—Sm1—O5	110.87 (8)	O5—C8—C9	119.5 (3)
O1—Sm1—O5	76.00 (9)	Sm1—O3—H7	115.8
O6—Sm1—O5	77.88 (8)	Sm1—O3—H8	136.3
O2^{i}—Sm1—O4	72.46 (8)	H7—O3—H8	101.9
O1—Sm1—O4	131.46 (8)	N3—C7—S1	177.0 (4)
O6—Sm1—O4	70.66 (8)	N1—C1—C2	119.2 (3)
O5—Sm1—O4	77.99 (8)	N1—C1—H1	120.4
O2^{i}—Sm1—N2	82.18 (10)	C2—C1—H1	120.4
O1—Sm1—N2	78.06 (11)	C6—C5—C3	120.7 (4)
O6—Sm1—N2	111.92 (11)	C6—C5—H5	119.7
O5—Sm1—N2	148.01 (11)	C3—C5—H5	119.7
O4—Sm1—N2	133.87 (10)	N2—C10—S2	178.2 (4)
O2^{i}—Sm1—N3	149.98 (10)	C2—C3—C5	117.2 (3)
O1—Sm1—N3	75.73 (9)	C2—C3—C4	121.3 (4)
O6—Sm1—N3	69.53 (10)	C5—C3—C4	121.4 (4)
O5—Sm1—N3	79.49 (10)	N1—C6—C5	119.9 (4)
O4—Sm1—N3	137.40 (9)	N1—C6—H6	120.1
N2—Sm1—N3	76.22 (12)	C5—C6—H6	120.1
O2^{i}—Sm1—O3	81.78 (9)	C3—C2—C1	121.2 (4)
O1—Sm1—O3	144.60 (9)	C3—C2—H2	119.4
O6—Sm1—O3	69.11 (9)	C1—C2—H2	119.4
O5—Sm1—O3	139.15 (9)	C10—N2—Sm1	169.4 (3)
O4—Sm1—O3	68.97 (8)	Sm1—O6—H12	123.5
N2—Sm1—O3	69.75 (11)	Sm1—O6—H13	131.0
N3—Sm1—O3	109.28 (10)	H12—O6—H13	103.3
O2^{i}—Sm1—O2	63.93 (9)	C8—C9—H9A	109.5
O1—Sm1—O2	64.94 (8)	C8—C9—H9B	109.5
O6—Sm1—O2	115.14 (8)	H9A—C9—H9B	109.5
O5—Sm1—O2	47.06 (7)	C8—C9—H9C	109.5
O4—Sm1—O2	67.26 (7)	H9A—C9—H9C	109.5
N2—Sm1—O2	132.87 (10)	H9B—C9—H9C	109.5
N3—Sm1—O2	118.38 (10)	C3—C4—H4A	109.5
O3—Sm1—O2	130.46 (8)	C3—C4—H4B	109.5
Sm1—O4—H10	127.3	H4A—C4—H4B	109.5

Sm1—O4—H11	109.2	C3—C4—H4C	109.5
H10—O4—H11	103.6	H4A—C4—H4C	109.5
C6—N1—C1	121.8 (3)	H4B—C4—H4C	109.5
C6—N1—O1	119.5 (3)		

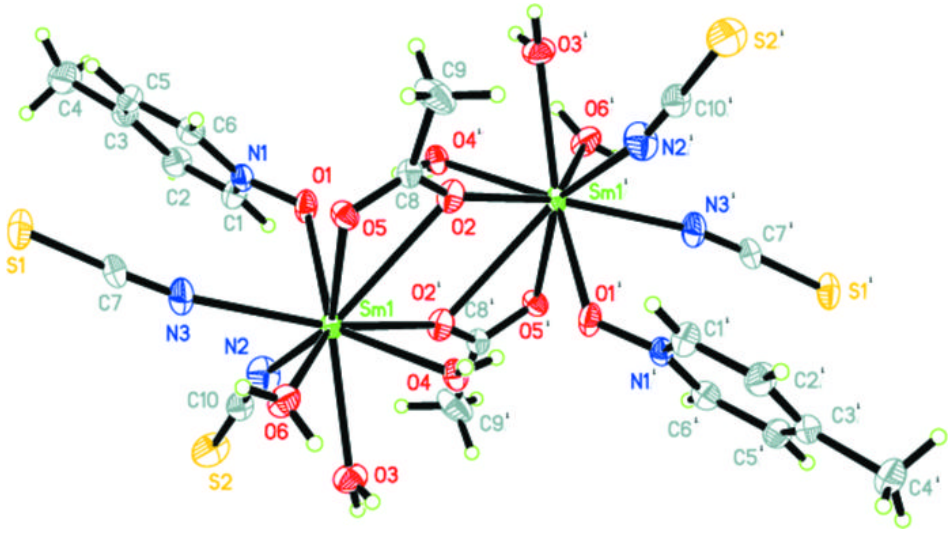
Symmetry codes: (i) −*x*+2, −*y*, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O6—H12···S2^{ii}	0.89	2.35	3.240 (3)	172
O3—H8···S1^{iii}	0.88	2.46	3.325 (3)	168
O4—H10···S1^{iv}	0.89	2.39	3.279 (2)	178
O6—H13···O5^{iv}	0.89	1.78	2.674 (3)	177
O4—H11···O1^{i}	0.89	1.79	2.685 (3)	177
O3—H7···S2^{ii}	0.90	2.83	3.643 (3)	152

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

Fig. 1


O3'
S2'
C9
C5
C4
C6
C3
N1
C2
C1
O1
O5
C8
O4'
O2
O6'
C10'
N2'
Sm1'
N3'
C7'
S1'
S1
C7
N3
Sm1
O2'
C8'
O5'
O1'
C1'
N2
C10
O6
S2
O4
C9'
O3
N1'
C2'
C6'
C3'
C5'
C4'