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Bis(μ-trifluoromethanesulfonato-κ^2O:O')-bis[aqua(1,4,7,10,13,16-hexaoxacyclooctadecane-κ^6O)barium(II)] bis(trifluoromethanesulfonate)

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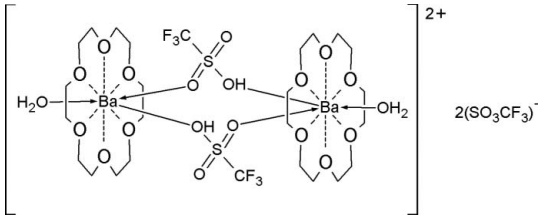
Key indicators: single-crystal X-ray study; T = 175 K; mean σ(C–C) = 0.004 Å; R factor = 0.023; wR factor = 0.022; data-to-parameter ratio = 19.6.

The crystal structure of the title compound, $[Ba_2(CF_3O_3S)_2(C_{12}H_{24}O_6)_2(H_2O)_2](CF_3O_3S)_2$, comprises a ten-coordinated Ba^{2+} cation that is coordinated by 18-crown-6, trifluoromethanesulfonate counter-ions and a water molecule, and an uncoordinated counter-ion. The dinuclear cation lies on a center of inversion. Each coordinated triflate group chelates a Ba atom while being monodentate to the adjacent Ba atom. The uncoordinated counter-ion is hydrogen bonded to the cation. Hydrogen bonds form infinite tubular arrays.

Related literature

For a similar Ba–18-crown-6 compound, see: Wei *et al.* (1988). For examples of macrocycle–cation π interactions, see: Arnal-Hérault *et al.* (2005). For biological and related applications, see: Dougherty (1996); Meyer *et al.* (2003); Ma & Dougherty (1997). For examples of tubular superstructures, see: Barboiu *et al.* (2003); Blondeau *et al.* (2005); Fromm & Bergougnant (2007).



Experimental

Crystal data

$[Ba_2(CF_3O_3S)_2(C_{12}H_{24}O_6)_2(H_2O)_2](CF_3O_3S)_2$
M_r = 1465.34
Triclinic, $P\overline{1}$
a = 8.942 (1) Å
b = 12.3257 (16) Å
c = 12.7431 (14) Å
α = 68.629 (12)°
β = 87.576 (10)°
γ = 83.212 (10)°
V = 1298.8 (3) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 1.79 mm^{-1}
T = 175 K
0.35 × 0.16 × 0.11 mm

Data collection

Oxford Diffraction GEMINI diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2007)
T_{min} = 0.90, T_{max} = 1.00 (expected range = 0.739–0.821)
23806 measured reflections
9409 independent reflections
6364 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.022
S = 1.11
6364 reflections
325 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

Table 1
Selected bond lengths (Å).

Ba1—O3	2.9825 (17)	Ba1—O24	2.8375 (17)
Ba1—O4	2.9669 (17)	Ba1—O27	2.8701 (17)
Ba1—O5^{i}	2.8504 (16)	Ba1—O30	2.8344 (16)
Ba1—O18	2.8336 (15)	Ba1—O33	2.8567 (15)
Ba1—O21	2.8259 (16)	Ba1—O60	2.6916 (18)

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

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

D—H···A	D—H	H···A	D···A	D—H···A
C28—H47···O13^{ii}	0.97	2.51	3.384 (4)	149
C26—H48···O11^{iii}	0.97	2.47	3.316 (4)	146
C20—H56···O12	0.98	2.59	3.310 (4)	130
O60—H61···O13^{ii}	0.86	1.96	2.811 (4)	171
O60—H62···O11^{iv}	0.86	1.95	2.801 (4)	168

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2007); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2007); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2003); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *CAMERON* (Watkin *et al.*, 1996), *Mercury* (Macrae *et al.*, 2006) and *DrawXtl* (Finger *et al.*, 2007); software used to prepare material for publication: *CRYSTALS*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2370).

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supplementary materials

Acta Cryst. (2007). E**63**, m3151-m3152 [**doi:10.1107/S1600536807060564**]

Bis(μ-trifluoromethanesulfonato-κ^2O:O')bis[aqua(1,4,7,10,13,16-hexaoxacyclooctadecane-κ^6O)barium(II)] bis(trifluoromethanesulfonate)

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Comment

Macrocycle-cation-π-interactions are of particular biological interest (Dougherty, 1996). In an attempt to co-crystallize an aromatic derivative with a macrocyclic (18-crown-6)·($BaTf_2$) complex (Arnal-Hérault *et al.*, 2005), the latter complex crystallized without the organic compound. Crown-ethers are well known complexants to metal cations, with selective recognition properties relative to the size of the ring. The structure of $Ba(C_{12}O_8H_{24})\cdot(H_2O),(CF_3SO_3)_2$ is forming tubular arrays (Barboiu *et al.*, 2003) made of crown ether rings. The oxygen atoms of the crown ether are coordinating the cation in equatorial position while the trifluoromethanesulfonate (CF_3SO_3 or Tf) anions are coordinating the Ba^{2+} in apical position. The second apical position is coordinated by a bridging water molecule which is simultaneously H-bonded to both trifluoromethanesulfonate anions (O(Tf)–O(H_2O) ≃ 2.805 Å) and coordinated to the Ba^{2+} cation (Ba–O(H_2O) = 2.689 (2) Å). Fig. 1 shows the full coordination pattern of barium ions. Trifluoromethanesulfonate anions are either coordinated to two barium cations, or to two water molecules, them-self coordinated to Ba^{2+} cations. Each barium cation has the same environment *i.e.* it is coordinating to ten oxygen atoms of which six from the 18 C6, three from two trifluoromethanesulfonate on one face of the 18 C6 and one from the water molecule on the other face of the 18 C6. The decahedral geometry of barium can be characterized as a sliced distorted tetrahedral environment as depicted in Fig. 2. Molecular columns of barium are formed with an average Ba–Ba distance of 8.5 Å between the anions inside one row (See Fig. 3). Each 18 C6 tube is closely packed to two others through hydrophobic contacts, forming planar sheets. About 7.5 Å separate two rows of barium of one sheet, while the distance between the rows of barium in two distinct sheets is about 9.9 Å. Trifluoromethanesulfonate fluoride atoms close contacts are observed between two sheets (Fig. 4) with F–F = 2.932 (2) Å. The overall packing of the title compound is unusual in the way the trifluoromethanesulfonate anions are inserted between the parallel crown ethers and form supramolecular polymers containing metal cations, counter anions and water molecules. In all crystal structures including trifluoromethanesulfonate and 18 C6, the ion is pushed away in the hydrophobic gaps between the crown ether tubular arrays and does not form an ionic bridge between two metal cations (Wei *et al.*, 1988).

Experimental

The title compound was prepared by dissolving in acetone (10 ml), in equimolar proportions, 1-(1*H*-indol-5-yl)-3-phenylurea (0.100 g), 18 C6 (0.105 g) and barium trifluoromethanesulfonate (0.173 g). The metallo-organic compound was crystallized by layering this solution with isopropyl ether. The crystals were formed over two days, by slow diffusion of the non-solvent in the solvent phase. The initial goal of the work was to grow co-crystals of inclusion complexes of Ba-18 C6 bound to the ureido-derivative ligand through cation-π interaction. Indeed, Arnal-Hérault *et al.* (2005) previously showed that the cation was favourably coordinated by the crown ether while the counter-ion was complexing with the ureido moiety of the co-crystallized ligand.

Refinement

The H atoms, including those of the water molecule, were all located in a difference map, and repositioned geometrically. The H atoms were initially refined with soft restraints on the bond lengths and angles to regularize their geometry (C—H in the range 0.93–0.98, O—H = 0.87 Å) and U_{iso}(H) (in the range 1.2–1.5 times U_{eq} of the parent atom), after which the positions were refined with riding constraints.

Figures

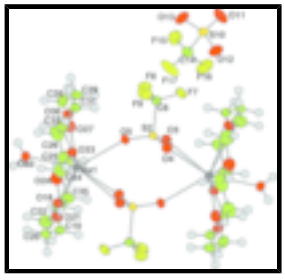

Fig. 1. Representation of (1) with the numbering scheme adopted. The Ba atom is in dark-grey, the F atom in light-green, the S atom in yellow, the C atom in green, the O atoms in red and the H atom in grey. Displacement ellipsoids are drawn at the 50% level. Non-labelled atoms are related by inversion symmetry (−1 halfway the two sulfur and barium atoms) to labeled atoms.

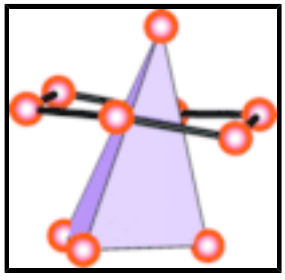

Fig. 2. Sliced distorted tetrahedron environment for the barium cation. The red balls represent oxygen atoms. The oxygen atoms forming the base of the tetrahedron belong to the trifluoromethanesulfonate ions and the oxygen atom at the summit is part of the water molecule.

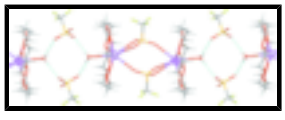

Fig. 3. Representation of the repeating pattern with sandwiched trifluoromethanesulfonate anions between crown ethers-barium motifs.

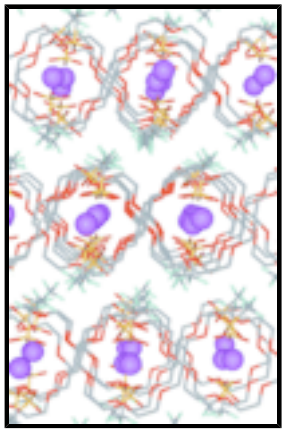

Fig. 4. The three-dimensional packing showing discrete sheets of crown ether tubes.

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Crystal data

$[Ba_2(CF_3O_3S)_2(C_{12}H_{24}O_6)_2(H_2O)_2](CF_3O_3S)_2$	$Z = 1$
$M_r = 1465.34$	$F_{000} = 712$
Triclinic, $P\overline{1}$	$D_x = 1.835$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 8.9420$ (10) Å	Cell parameters from 23806 reflections
$b = 12.3257$ (16) Å	$\theta = 4$–33º
$c = 12.7431$ (14) Å	$\mu = 1.79$ mm^{-1}
$\alpha = 68.629$ (12)º	$T = 175$ K
$\beta = 87.576$ (10)º	Needle, colourless
$\gamma = 83.212$ (10)º	0.35 × 0.16 × 0.11 mm
$V = 1298.8$ (3) Å^3	

Data collection

Oxford Diffraction GEMINI diffractometer	9409 independent reflections
Radiation source: Enhance (Mo) X-ray Source	6364 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.022$
Detector resolution: 16.0143 pixels mm^{-1}	$\theta_{max} = 33.5$º
$T = 175$ K	$\theta_{min} = 3.8$º
φ & ω scans	$h = -13\rightarrow13$
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2007); Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.	$k = -18\rightarrow19$
$T_{min} = 0.90$, $T_{max} = 1.00$	$l = -19\rightarrow16$
23806 measured reflections	

Refinement

Refinement on F	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.023$	Method, part 1, Chebychev polynomial, (Watkin, 1994, *P*rince, 1982) [*w*eight] = 1.0/[A_0*T_0(x) + A_1*T_1(x) ··· + A_{n-1}]*T_{n-1}(x)] where A_i are the Chebychev coefficients listed below*w* and x = *F* /*F*max Method = Robust Weighting (*P*rince, 1982) W = [*w*eight] * [1-(delta*F*/6*sigma*F*)^2]^2 A_i are: 19.2 -26.3 13.3 -3.95
$wR(F^2) = 0.022$	$(\Delta/\sigma)_{max} = 0.001$

S = 1.11	$\Delta\rho_{max}$ = 0.45 e Å^{-3}
6364 reflections	$\Delta\rho_{min}$ = −0.48 e Å^{-3}
325 parameters	Extinction correction: None
Primary atom site location: structure-invariant direct methods	

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Ba1	0.823886 (15)	0.307417 (11)	0.715055 (11)	0.0197
S2	0.98425 (6)	0.57362 (4)	0.60939 (4)	0.0214
O3	0.82673 (17)	0.56535 (14)	0.59766 (13)	0.0280
O4	1.06495 (18)	0.46352 (14)	0.67793 (14)	0.0304
O5	1.0606 (2)	0.63842 (15)	0.50802 (14)	0.0329
C6	0.9852 (3)	0.6661 (2)	0.6942 (2)	0.0316
F7	0.92051 (19)	0.61984 (15)	0.79318 (13)	0.0447
F8	0.9110 (2)	0.77100 (14)	0.64130 (16)	0.0568
F9	1.1246 (2)	0.68206 (19)	0.71126 (17)	0.0593
S10	0.55301 (6)	0.83623 (5)	0.86965 (5)	0.0256
O11	0.4194 (2)	0.8629 (2)	0.92588 (16)	0.0484
O12	0.6165 (2)	0.71647 (15)	0.91215 (17)	0.0410
O13	0.6602 (2)	0.92020 (17)	0.84453 (19)	0.0479
C14	0.4807 (3)	0.8563 (2)	0.7315 (2)	0.0361
F15	0.4128 (2)	0.96364 (15)	0.67946 (14)	0.0525
F16	0.3796 (2)	0.78288 (17)	0.73984 (17)	0.0622
F17	0.5887 (2)	0.8363 (2)	0.66466 (17)	0.0740
O18	0.55879 (17)	0.42809 (13)	0.76730 (13)	0.0266
C19	0.5834 (3)	0.5078 (2)	0.8213 (2)	0.0308
C20	0.6720 (3)	0.4413 (2)	0.9258 (2)	0.0348
O21	0.81117 (19)	0.38901 (14)	0.89606 (13)	0.0302
C22	0.9059 (4)	0.3309 (3)	0.9923 (2)	0.0452
C23	1.0526 (3)	0.2859 (3)	0.9547 (3)	0.0488
O24	1.0229 (2)	0.20633 (16)	0.90171 (15)	0.0382
C25	1.1552 (3)	0.1405 (3)	0.8804 (3)	0.0544
C26	1.1083 (4)	0.0470 (3)	0.8449 (3)	0.0536
O27	1.0265 (2)	0.10039 (15)	0.74158 (15)	0.0378
C28	0.9786 (3)	0.0131 (2)	0.7045 (3)	0.0456
C29	0.8885 (3)	0.0720 (2)	0.5994 (2)	0.0430
O30	0.7606 (2)	0.13963 (14)	0.62381 (14)	0.0313
C31	0.6505 (3)	0.1803 (2)	0.5361 (2)	0.0362
C32	0.5118 (3)	0.2330 (2)	0.5771 (2)	0.0359
O33	0.54743 (17)	0.33276 (14)	0.59967 (13)	0.0269
C34	0.4159 (3)	0.3861 (3)	0.6381 (2)	0.0388
C35	0.4590 (3)	0.4808 (2)	0.6740 (2)	0.0340
H36	0.3703	0.5207	0.6964	0.0416*
H37	0.5078	0.5375	0.6122	0.0413*
H38	0.3740	0.3246	0.7009	0.0500*
H39	0.3438	0.4169	0.5780	0.0503*

H40	0.4765	0.1765	0.6458	0.0466*
H41	0.4322	0.2567	0.5207	0.0471*
H42	0.6903	0.2365	0.4685	0.0455*
H43	0.6257	0.1139	0.5185	0.0464*
H44	0.9487	0.1209	0.5398	0.0525*
H45	0.8550	0.0117	0.5767	0.0528*
H46	1.0667	−0.0352	0.6916	0.0524*
H47	0.9167	−0.0359	0.7628	0.0518*
H48	1.1964	−0.0017	0.8344	0.0574*
H49	1.0434	−0.0014	0.9024	0.0574*
H50	1.2142	0.1046	0.9496	0.0613*
H51	1.2145	0.1921	0.8223	0.0613*
H52	1.1166	0.2439	1.0196	0.0582*
H53	1.1021	0.3498	0.9018	0.0580*
H54	0.9236	0.3861	1.0276	0.0541*
H55	0.8569	0.2662	1.0459	0.0543*
H56	0.6949	0.4938	0.9634	0.0436*
H57	0.6147	0.3809	0.9773	0.0441*
H58	0.6369	0.5710	0.7702	0.0387*
H59	0.4879	0.5406	0.8420	0.0391*
O60	0.6654 (3)	0.14219 (16)	0.85835 (16)	0.0479
H61	0.6682	0.0712	0.8616	0.0500*
H62	0.6264	0.1452	0.9203	0.0500*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ba1	0.02160 (5)	0.01736 (5)	0.01886 (5)	−0.00117 (3)	0.00021 (4)	−0.00535 (3)
S2	0.0234 (2)	0.0229 (2)	0.0205 (2)	−0.00578 (18)	0.00364 (17)	−0.01018 (18)
O3	0.0247 (7)	0.0310 (8)	0.0309 (8)	−0.0048 (6)	−0.0008 (6)	−0.0137 (6)
O4	0.0271 (8)	0.0287 (8)	0.0329 (8)	−0.0013 (6)	0.0007 (6)	−0.0086 (7)
O5	0.0442 (10)	0.0322 (8)	0.0262 (8)	−0.0151 (7)	0.0139 (7)	−0.0133 (7)
C6	0.0353 (12)	0.0341 (11)	0.0342 (12)	−0.0162 (9)	0.0110 (9)	−0.0203 (10)
F7	0.0561 (10)	0.0593 (10)	0.0333 (8)	−0.0285 (8)	0.0189 (7)	−0.0292 (7)
F8	0.0837 (14)	0.0319 (8)	0.0588 (11)	−0.0036 (8)	0.0180 (10)	−0.0240 (8)
F9	0.0455 (10)	0.0929 (15)	0.0712 (12)	−0.0395 (10)	0.0172 (9)	−0.0593 (12)
S10	0.0262 (2)	0.0247 (2)	0.0286 (3)	−0.00262 (19)	−0.0003 (2)	−0.0126 (2)
O11	0.0415 (11)	0.0666 (14)	0.0318 (9)	0.0138 (10)	0.0028 (8)	−0.0178 (9)
O12	0.0410 (10)	0.0279 (8)	0.0532 (12)	0.0017 (7)	−0.0108 (9)	−0.0143 (8)
O13	0.0494 (12)	0.0365 (10)	0.0630 (13)	−0.0178 (9)	−0.0062 (10)	−0.0195 (9)
C14	0.0395 (13)	0.0369 (12)	0.0349 (12)	−0.0054 (10)	0.0018 (10)	−0.0164 (10)
F15	0.0683 (12)	0.0403 (9)	0.0386 (9)	0.0014 (8)	−0.0133 (8)	−0.0030 (7)
F16	0.0720 (13)	0.0532 (11)	0.0662 (12)	−0.0212 (10)	−0.0296 (10)	−0.0198 (9)
F17	0.0673 (13)	0.1174 (19)	0.0463 (11)	0.0111 (13)	0.0071 (9)	−0.0475 (12)
O18	0.0282 (8)	0.0236 (7)	0.0288 (8)	0.0001 (6)	−0.0001 (6)	−0.0115 (6)
C19	0.0336 (12)	0.0257 (10)	0.0358 (12)	−0.0024 (9)	0.0083 (9)	−0.0156 (9)
C20	0.0477 (14)	0.0323 (11)	0.0307 (11)	−0.0103 (10)	0.0082 (10)	−0.0181 (10)
O21	0.0402 (9)	0.0306 (8)	0.0201 (7)	−0.0028 (7)	−0.0035 (6)	−0.0094 (6)

C22	0.0611 (18)	0.0471 (15)	0.0265 (12)	−0.0050 (13)	−0.0162 (12)	−0.0108 (11)
C23	0.0487 (16)	0.0527 (17)	0.0401 (15)	−0.0070 (13)	−0.0205 (13)	−0.0083 (13)
O24	0.0304 (9)	0.0394 (9)	0.0376 (9)	0.0031 (7)	−0.0103 (7)	−0.0063 (8)
C25	0.0251 (12)	0.079 (2)	0.0434 (16)	0.0120 (13)	−0.0069 (11)	−0.0086 (15)
C26	0.0485 (17)	0.0469 (16)	0.0449 (16)	0.0266 (13)	0.0000 (13)	−0.0022 (13)
O27	0.0385 (9)	0.0288 (8)	0.0356 (9)	0.0087 (7)	0.0044 (7)	−0.0034 (7)
C28	0.0454 (15)	0.0235 (11)	0.0596 (18)	0.0040 (10)	0.0164 (13)	−0.0094 (11)
C29	0.0556 (17)	0.0301 (12)	0.0480 (15)	−0.0016 (11)	0.0137 (13)	−0.0222 (11)
O30	0.0411 (9)	0.0247 (7)	0.0294 (8)	−0.0011 (7)	0.0044 (7)	−0.0127 (6)
C31	0.0523 (15)	0.0336 (12)	0.0288 (11)	−0.0124 (11)	−0.0006 (10)	−0.0160 (10)
C32	0.0392 (13)	0.0377 (12)	0.0345 (12)	−0.0141 (10)	−0.0030 (10)	−0.0142 (10)
O33	0.0242 (7)	0.0295 (8)	0.0288 (8)	−0.0040 (6)	−0.0024 (6)	−0.0120 (6)
C34	0.0229 (11)	0.0525 (16)	0.0444 (14)	−0.0021 (10)	−0.0009 (10)	−0.0220 (12)
C35	0.0269 (11)	0.0345 (12)	0.0387 (13)	0.0077 (9)	−0.0038 (9)	−0.0140 (10)
O60	0.0825 (15)	0.0312 (9)	0.0358 (10)	−0.0251 (10)	0.0259 (10)	−0.0161 (8)

Geometric parameters (Å, °)

Ba1—O3	2.9825 (17)	C22—C23	1.492 (4)
Ba1—O4	2.9669 (17)	C22—H54	0.972
Ba1—O5^{i}	2.8504 (16)	C22—H55	0.977
Ba1—O18	2.8336 (15)	C23—O24	1.431 (4)
Ba1—O21	2.8259 (16)	C23—H52	0.968
Ba1—O24	2.8375 (17)	C23—H53	0.970
Ba1—O27	2.8701 (17)	O24—C25	1.427 (3)
Ba1—O30	2.8344 (16)	C25—C26	1.490 (5)
Ba1—O33	2.8567 (15)	C25—H50	0.975
Ba1—O60	2.6916 (18)	C25—H51	0.969
O60—Ba1	2.6916 (18)	C26—O27	1.427 (3)
O60—O11^{ii}	2.801 (3)	C26—H48	0.967
O60—O13^{iii}	2.811 (3)	C26—H49	0.978
S2—O3	1.4420 (16)	O27—C28	1.436 (3)
S2—O4	1.4433 (17)	C28—C29	1.488 (4)
S2—O5	1.4406 (16)	C28—H46	0.975
S2—C6	1.835 (2)	C28—H47	0.969
C6—F7	1.321 (3)	C29—O30	1.425 (3)
C6—F8	1.329 (3)	C29—H44	0.968
C6—F9	1.323 (3)	C29—H45	0.971
S10—O11	1.4335 (19)	O30—C31	1.427 (3)
S10—O12	1.4282 (18)	C31—C32	1.494 (4)
S10—O13	1.4348 (19)	C31—H42	0.973
S10—C14	1.822 (3)	C31—H43	0.976
C14—F15	1.328 (3)	C32—O33	1.433 (3)
C14—F16	1.328 (3)	C32—H40	0.967
C14—F17	1.322 (3)	C32—H41	0.975
O18—C19	1.430 (3)	O33—C34	1.435 (3)
O18—C35	1.422 (3)	C34—C35	1.494 (4)
C19—C20	1.488 (4)	C34—H38	0.978

C19—H58	0.975	C34—H39	0.958
C19—H59	0.969	C35—H36	0.971
C20—O21	1.434 (3)	C35—H37	0.970
C20—H56	0.977	O60—H61	0.858
C20—H57	0.977	O60—H62	0.861
O21—C22	1.428 (3)		
O3—Ba1—O4	47.65 (4)	O18—C19—H59	110.0
O3—Ba1—O5^{i}	68.86 (5)	C20—C19—H59	108.5
O4—Ba1—O5^{i}	70.15 (5)	H58—C19—H59	109.4
O3—Ba1—O18	69.95 (5)	C19—C20—O21	108.74 (19)
O4—Ba1—O18	106.62 (5)	C19—C20—H56	110.5
O5^{i}—Ba1—O18	123.16 (5)	O21—C20—H56	108.5
O3—Ba1—O21	77.40 (5)	C19—C20—H57	109.9
O4—Ba1—O21	72.76 (5)	O21—C20—H57	110.3
O5^{i}—Ba1—O21	140.95 (5)	H56—C20—H57	108.9
O18—Ba1—O21	57.87 (5)	C20—O21—C22	111.26 (19)
O3—Ba1—O24	114.31 (5)	O21—C22—C23	108.7 (2)
O4—Ba1—O24	72.98 (5)	O21—C22—H54	109.5
O5^{i}—Ba1—O24	119.71 (6)	C23—C22—H54	109.2
O18—Ba1—O24	112.12 (5)	O21—C22—H55	109.4
O21—Ba1—O24	58.03 (5)	C23—C22—H55	110.3
O3—Ba1—O27	136.71 (5)	H54—C22—H55	109.7
O4—Ba1—O27	94.93 (5)	C22—C23—O24	108.0 (2)
O5^{i}—Ba1—O27	79.00 (5)	C22—C23—H52	109.5
O18—Ba1—O27	153.05 (5)	O24—C23—H52	108.7
O21—Ba1—O27	116.26 (5)	C22—C23—H53	110.8
O3—Ba1—O30	128.43 (5)	O24—C23—H53	109.7
O4—Ba1—O30	134.15 (5)	H52—C23—H53	110.1
O5^{i}—Ba1—O30	68.81 (5)	C23—O24—C25	113.7 (2)
O18—Ba1—O30	112.40 (5)	O24—C25—C26	108.4 (2)
O21—Ba1—O30	150.20 (5)	O24—C25—H50	109.0
O3—Ba1—O33	86.75 (5)	C26—C25—H50	109.4
O4—Ba1—O33	132.11 (5)	O24—C25—H51	109.6
O5^{i}—Ba1—O33	81.31 (5)	C26—C25—H51	111.0
O18—Ba1—O33	58.91 (5)	H50—C25—H51	109.5
O21—Ba1—O33	116.52 (5)	C25—C26—O27	109.0 (2)
O3—Ba1—O60	143.63 (5)	C25—C26—H48	109.7
O4—Ba1—O60	149.11 (6)	O27—C26—H48	109.7
O5^{i}—Ba1—O60	136.56 (5)	C25—C26—H49	109.8
O18—Ba1—O60	73.70 (6)	O27—C26—H49	109.1
O21—Ba1—O60	82.49 (5)	H48—C26—H49	109.5
O24—Ba1—O27	58.58 (6)	C26—O27—C28	110.8 (2)
O24—Ba1—O30	111.57 (5)	O27—C28—C29	109.2 (2)
O27—Ba1—O30	57.95 (5)	O27—C28—H46	109.4
O24—Ba1—O33	153.87 (5)	C29—C28—H46	110.7
O27—Ba1—O33	116.84 (5)	O27—C28—H47	108.7
O30—Ba1—O33	58.94 (5)	C29—C28—H47	109.5

O24—Ba1—O60	78.38 (6)	H46—C28—H47	109.3
O27—Ba1—O60	79.49 (6)	C28—C29—O30	108.2 (2)
O30—Ba1—O60	67.77 (5)	C28—C29—H44	110.7
O3—S2—O4	112.80 (10)	O30—C29—H44	111.1
O3—S2—O5	116.11 (10)	C28—C29—H45	107.9
O4—S2—O5	115.10 (10)	O30—C29—H45	109.4
O3—S2—C6	104.15 (10)	H44—C29—H45	109.4
O4—S2—C6	103.97 (11)	C29—O30—C31	112.9 (2)
O5—S2—C6	102.62 (10)	O30—C31—C32	108.66 (19)
S2—C6—F7	111.90 (15)	O30—C31—H42	110.2
S2—C6—F8	110.42 (18)	C32—C31—H42	111.6
F7—C6—F8	107.6 (2)	O30—C31—H43	109.0
S2—C6—F9	110.85 (16)	C32—C31—H43	108.9
F7—C6—F9	108.3 (2)	H42—C31—H43	108.4
F8—C6—F9	107.6 (2)	C31—C32—O33	108.8 (2)
O11—S10—O12	115.32 (13)	C31—C32—H40	110.1
O11—S10—O13	114.89 (14)	O33—C32—H40	109.0
O12—S10—O13	114.95 (12)	C31—C32—H41	110.4
O11—S10—C14	102.02 (12)	O33—C32—H41	109.9
O12—S10—C14	103.74 (12)	H40—C32—H41	108.6
O13—S10—C14	103.41 (13)	C32—O33—C34	110.17 (18)
S10—C14—F15	112.46 (18)	O33—C34—C35	109.32 (19)
S10—C14—F16	111.11 (18)	O33—C34—H38	107.4
F15—C14—F16	106.6 (2)	C35—C34—H38	111.2
S10—C14—F17	111.77 (19)	O33—C34—H39	108.7
F15—C14—F17	107.8 (2)	C35—C34—H39	111.1
F16—C14—F17	106.8 (2)	H38—C34—H39	109.1
C19—O18—C35	112.02 (17)	C34—C35—O18	108.0 (2)
O18—C19—C20	108.15 (18)	C34—C35—H36	110.3
Ba1—O60—O11^{ii}	123.03 (8)	O18—C35—H36	109.5
Ba1—O60—O13^{iii}	122.84 (8)	C34—C35—H37	109.9
O11^{ii}—O60—O13^{iii}	110.59 (9)	O18—C35—H37	110.3
O18—C19—H58	109.6	H36—C35—H37	108.8
C20—C19—H58	111.1	H61—O60—H62	107.6
O(4)—Ba(1)—O(3)—S(2)	3.37 (7)	O(4)—Ba(1)—O(18)—C(19)	−29.00 (15)
O(24)—Ba(1)—O(3)—S(2)	35.53 (9)	Ba(1)—O(27)—C(26)—C(25)	−38.9 (3)
O(33)—Ba(1)—O(3)—S(2)	−160.50 (8)	C(26)—O(27)—C(28)—C(29)	177.7 (2)
O(3)—Ba(1)—O(4)—S(2)	−3.37 (7)	Ba(1)—O(30)—C(31)—C(32)	−52.9 (2)
O(24)—Ba(1)—O(4)—S(2)	−152.88 (10)	C(34)—O(33)—C(32)—C(31)	179.39 (19)
O(33)—Ba(1)—O(4)—S(2)	18.58 (12)	O(18)—C(19)—C(20)—O(21)	58.5 (3)
O(3)—Ba(1)—O(18)—C(19)	−59.98 (14)	O(27)—C(28)—C(29)—O(30)	−60.3 (3)
O(18)—Ba(1)—O(3)—S(2)	141.49 (9)	C(28)—O(27)—C(26)—C(25)	−179.8 (2)
O(27)—Ba(1)—O(3)—S(2)	−33.69 (11)	Ba(1)—O(30)—C(29)—C(28)	56.3 (2)
O(60)—Ba(1)—O(3)—S(2)	139.44 (9)	C(29)—O(30)—C(31)—C(32)	171.0 (2)
O(18)—Ba(1)—O(4)—S(2)	−44.24 (9)	Ba(1)—O(33)—C(34)—C(35)	36.3 (2)
O(27)—Ba(1)—O(4)—S(2)	152.13 (8)	O(21)—C(22)—C(23)—O(24)	−59.7 (3)
O(60)—Ba(1)—O(4)—S(2)	−130.14 (10)	O(30)—C(31)—C(32)—O(33)	63.1 (2)
O(3)—Ba(1)—O(18)—C(35)	70.12 (15)	Ba(1)—O(27)—C(28)—C(29)	37.2 (3)

O(21)—Ba(1)—O(3)—S(2)	81.40 (8)	C(31)—O(30)—C(29)—C(28)	−167.8 (2)
O(30)—Ba(1)—O(3)—S(2)	−115.32 (8)	Ba(1)—O(33)—C(32)—C(31)	−43.4 (2)
O(5)^{i}—Ba(1)—O(3)—S(2)	−78.56 (8)	C(32)—O(33)—C(34)—C(35)	173.37 (19)
O(21)—Ba(1)—O(4)—S(2)	−91.88 (9)	O(24)—C(25)—C(26)—O(27)	63.3 (3)
O(30)—Ba(1)—O(4)—S(2)	103.31 (9)	O(33)—C(34)—C(35)—O(18)	−63.6 (2)
O(5)^{i}—Ba(1)—O(4)—S(2)	75.70 (9)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C28—H47···O13^{iii}	0.97	2.51	3.384 (4)	149
C26—H48···O11^{iv}	0.97	2.47	3.316 (4)	146
C20—H56···O12	0.98	2.59	3.310 (4)	130
O60—H61···O13^{iii}	0.86	1.96	2.811 (4)	171
O60—H62···O11^{ii}	0.86	1.95	2.801 (4)	168

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

Fig. 1

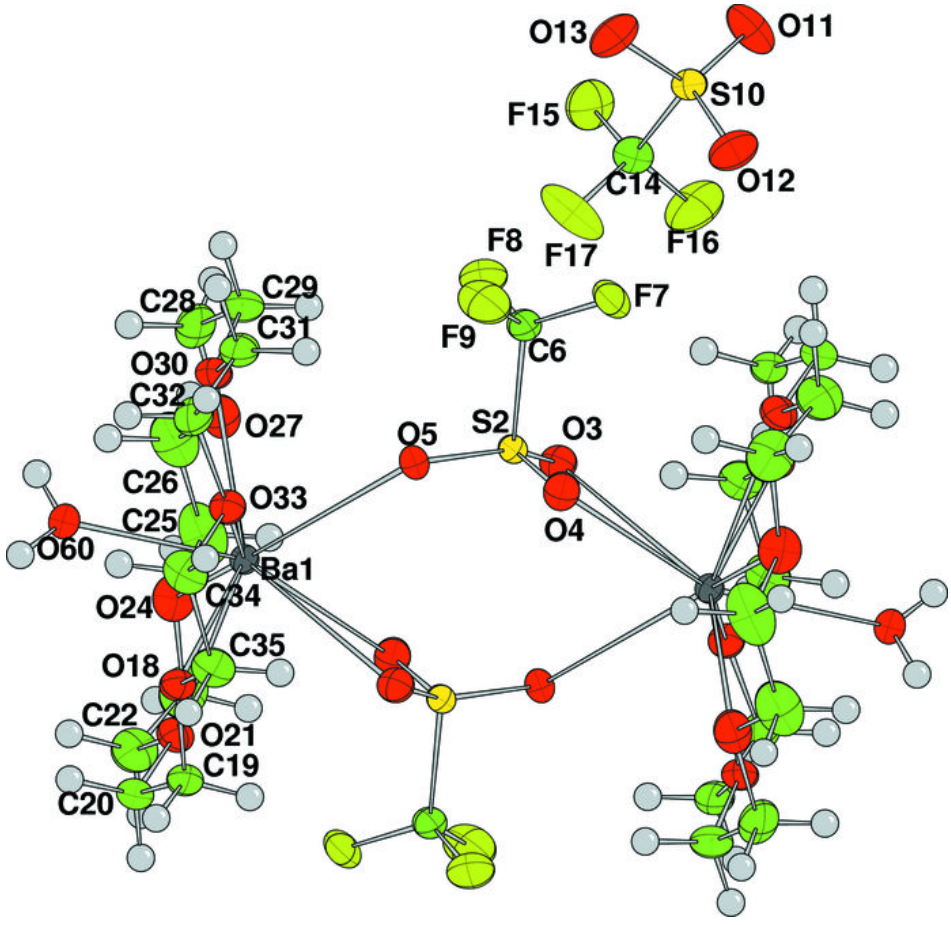
O13
O11
S10
F15
O12
C14
F8
F17
F16
F7
F9
C6
C28
C29
C31
O30
C32
O27
S2
O5
O3
C26
O33
C25
O4
O60
Ba1
C34
O24
C35
O18
C22
O21
C19
C20

Fig. 2

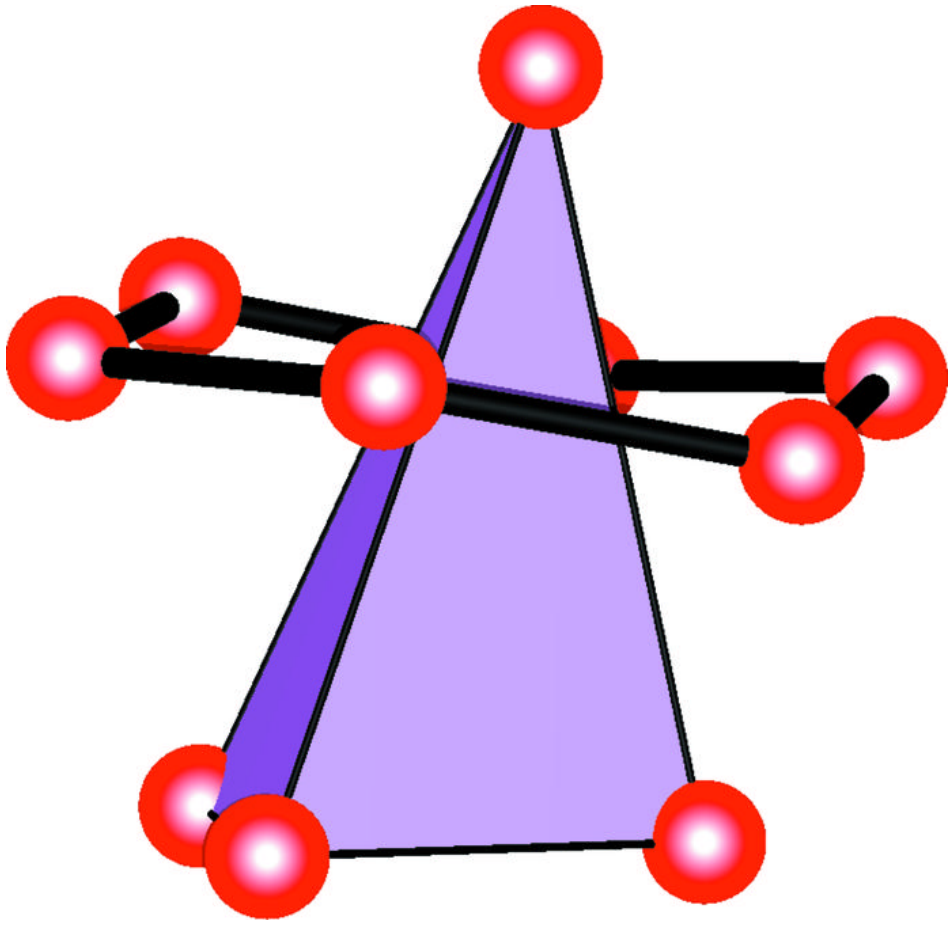

Fig. 3

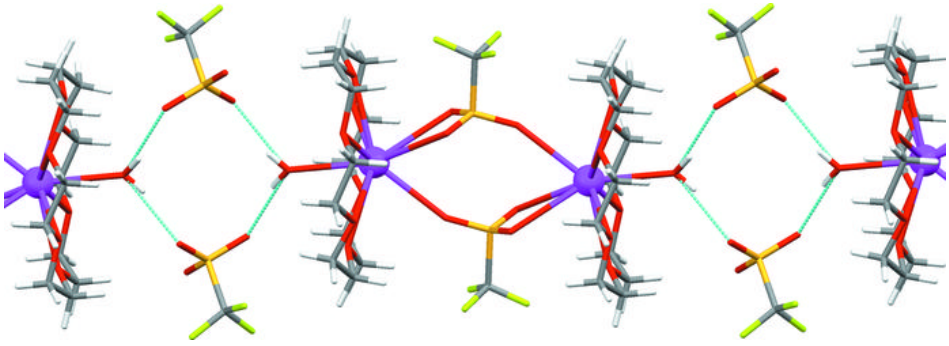

Fig. 4