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Bis{4-[(2-hydroxybenzylidine)hydrazino]-8-(trifluoromethyl)quinolinium} sulfate tetrahydrate

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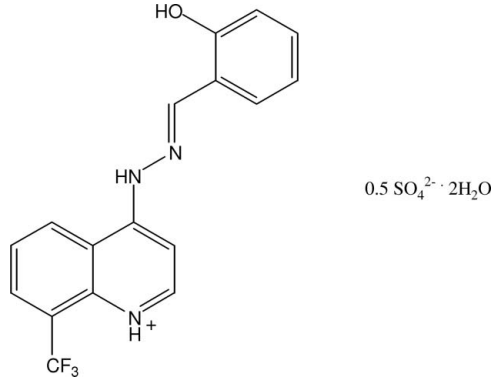
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.041; wR factor = 0.103; data-to-parameter ratio = 11.6.

The geometric parameters of the title compound, $2C_{17}H_{13}F_3N_3O^+\cdot SO_4^{2-}\cdot 4H_2O$, are in the usual ranges. There are two organic cations in the asymmetric unit with intramolecular dihedral angles of 10.94 (9) and 7.14 (8)° between their aromatic ring mean planes. The crystal packing is stabilized by numerous N—H···O, O—H···O and O—H···N hydrogen bonds.

Related literature

For related structures, see: Shi & Yuan (2006); Qian *et al.* (2006); Jing & Yu (2007). For background literature, see: Roma *et al.* (2000); Maguire *et al.* (1994); Zhang & Jenekhe (2000); El-Masry *et al.* (2000); Pandey *et al.* (1999); Singh *et al.* (1988); Hodnett *et al.* (1970); Desai *et al.* (2001); Aydogan *et al.* (2001); Taggi *et al.* (2002).



Experimental

Crystal data

$2C_{17}H_{13}F_3N_3O^+\cdot SO_4^{2-}\cdot 4H_2O$
M_r = 832.73
Triclinic, $P\overline{1}$
a = 11.210 (2) Å
b = 12.084 (2) Å
c = 14.919 (3) Å
α = 84.64 (4)°
β = 68.16 (4)°
γ = 74.38 (3)°
V = 1806.6 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.19 mm^{-1}
T = 173 (2) K
0.24 × 0.23 × 0.19 mm

Data collection

Stoe IPDSII two-circle diffractometer
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.958, T_{max} = 0.968
25452 measured reflections
6759 independent reflections
5130 reflections with $I > 2\sigma(I)$
R_{int} = 0.059

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.103
S = 0.99
6759 reflections
581 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1O···O4S^{i}	0.90 (5)	1.87 (5)	2.715 (3)	156 (4)
O1′—H1′···N3	0.84	1.84	2.602 (7)	149
N1—H1···O4W	0.88 (3)	1.90 (3)	2.713 (3)	153 (2)
N2—H2···O1S^{i}	0.83 (2)	2.06 (2)	2.873 (2)	166 (2)
O1A—H1OA···O3S^{ii}	0.86 (3)	1.81 (3)	2.671 (2)	175 (3)
N1A—H1A···O2S^{iii}	0.85 (3)	2.00 (3)	2.740 (2)	146 (2)
N2A—H2A···O1W^{ii}	0.88 (3)	1.99 (3)	2.855 (3)	169 (2)
O1W—H1WA···O4S^{iv}	0.85 (3)	1.92 (4)	2.752 (3)	170 (3)
O1W—H1WB···O3W^{v}	0.88 (4)	1.99 (4)	2.831 (3)	158 (3)
O2W—H2WA···O1A^{vi}	1.04 (9)	2.48 (9)	3.262 (3)	132 (6)
O2W—H2WB···O4S^{vii}	0.96 (4)	1.85 (4)	2.806 (3)	173 (3)
O3W—H3WA···O2S^{viii}	0.83 (4)	2.03 (4)	2.825 (2)	158 (3)
O3W—H3WB···O1S^{ix}	0.88 (4)	1.91 (4)	2.763 (2)	165 (3)
O4W—H4WA···O2W	0.81 (4)	1.99 (4)	2.793 (3)	171 (4)
O4W—H4WB···O3W	0.91 (4)	1.90 (4)	2.805 (3)	170 (3)

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

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2387).

References

Aydogan, F., Ocal, N., Turgut, Z. & Yolacan, C. (2001). *Bull. Korean Chem. Soc.* **22**, 476–480.

Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.

Desai, S. B., Desai, P. B. & Desai, K. R. (2001). *Heterocycl. Commun.* **7**, 83–90.

El-Masry, A. H., Fahmy, H. H. & Abdelwahed, S. H. A. (2000). *Molecules*, **5**, 1429–1438.

Hodnett, E. M. & Dunn, W. J. (1970). *J. Med. Chem.* **13**, 768–770.

Jing, Z.-L. & Yu, M. (2007). *Acta Cryst.* E**63**, o509–o510.

Maguire, M. P., Sheets, K. R., McVety, K., Spada, A. P. & Zilberstein, A. (1994). *J. Med. Chem.* **37**, 2129–2137.

Pandey, S. N., Sriram, D., Nath, G. & De Clercq, E. (1999). *Il Farma*co, **54**, 624–628.

Qian, H.-Y., Yin, Z.-G., Jia, J., Liu, S.-M. & Feng, L.-Q. (2006). *Acta Cryst.* E**62**, o3623–o3624.

Roma, G., Braccio, M. D., Grossi, G., Mattioli, F. & Ghia, M. (2000). *Eur. J. Med. Chem.* **35**, 1021–1035.

Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Shi, X.-F. & Yuan, C.-C. (2006). *Acta Cryst.* E**62**, o3290–o3291.

Singh, W. M. & Dash, B. C. (1988). *Pesticides*, **22**, 33–37.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Stoe & Cie (2001). *X-AREA*. Stoe & Cie, Darmstadt, Germany.

Taggi, A. E., Hafez, A. M., Wack, H., Young, B., Ferraris, D. & Lectka, T. (2002). *J. Am. Chem. Soc.* **124**, 6626–6635.

Zhang, X. & Jenekhe, S. A. (2000). *Macromolecules*, **33**, 2069–2082.

supplementary materials

Acta Cryst. (2007). E**63**, o2720-o2721 [**doi:10.1107/S1600536807020430**]

Bis{4-[(2-hydroxybenzylidine)hydrazino]-8-(trifluoromethyl)quinolinium} sulfate tetrahydrate

H. S. Yathirajan, B. K. Sarojini, B. Narayana, K. Sunil and M. Bolte

Comment

It is well known that the quinoline ring system is an important structural unit widely existing in alkaloids, therapeutics and synthetic analogues with interesting biological activities. A large variety of quinoline derivatives have been used as antimalarial, anti-inflammatory agents, antiasthmatic, antibacterial, antihypertensive and tyrokinase PDGF-RTK inhibiting agents. Furthermore, poly-substituted quinolines have been found to undergo hierarchical self-assembly into a variety of nano- and mesostructures with enhanced electronic and photonic functions.

Schiff bases are used as substrates in the preparation of number of industrial and biologically active compounds via ring closure, cycloaddition and replacement reactions. Moreover, Schiff bases are also known to have biological activities such as antimicrobial, antifungal, antitumor and as herbicides. Schiff bases have also been employed as ligands for complexation of metal ions. On the industrial scale, they have wide range of applications such as dyes and pigments.

The title compound, (I), a new Schiff base was synthesized and its crystal structure has been determined. The geometric parameters for (I) are in the usual ranges. There are two organic molecules in the asymmetric unit. The dihedral angles between the mean planes of their two aromatic ring systems are 10.94 (9)° and 7.14 (8)°, respectively. The crystal packing is stabilized by many N—H···O, O—H···O and O—H···N hydrogen bonds (Table 1).

Experimental

A mixture of 4-hydrazino-8-(trifluoromethyl)quinoline (1.13 g, 0.005 mol) and salicylaldehyde (0.61 g, 0.005 mol) in 15 ml of absolute ethanol containing 2 drops of 4 M sulfuric acid was refluxed for three hours. On cooling, the solid that separated was filtered and recrystallized from a (1:1 v/v) DMF & acetone mixture to yield crystals of (I) (m.p.: Above 523 K). Analysis for $C_{34}H_{34}F_6N_6O_{10}S$: Found (Calculated): C: 48.87 (48.99); H: 4.02 (4.08); N: 10.01 (10.09): S: 3.78% (3.84%)

Refinement

The hydroxyphenyl ring of one molecule is disordered over two sites by a twofold rotation. Thus, the hydroxyl group is found in both ortho positions with site occupation factors of 0.778 (5) and 0.222 (5). H atoms were found in a difference map, but those bonded to C were refined using a riding model with C—H = 0.95 Å, and U_{iso}(H) = 1.2U_{eq}(C). H atoms bonded to O were freely refined, except for the minor component of the disordered hydroxyl H atoms which was refined using a riding model with O—H = 0.84 Å, and U_{iso}(H) = 1.2U_{eq}(O). The torsion angle about the C—O bond was allowed to refine.

Figures

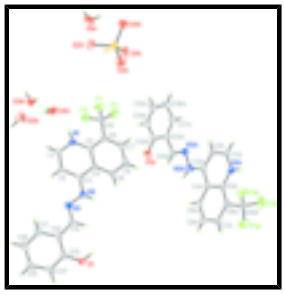


Fig. 1. Perspective view of (I); displacement ellipsoids are at the 50% probability level (arbitrary spheres for the H atoms).

Bis{4-[(2-hydroxybenzylidine)hydrazino]-8-(trifluoromethyl)quinolinium} sulfate tetrahydrate

Crystal data

$2C_{17}H_{13}F_3N_3O^+\cdot SO_4^{2-}\cdot 4H_2O$	$Z = 2$
$M_r = 832.73$	$F_{000} = 860$
Triclinic, $P\overline{1}$	$D_x = 1.531$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 11.210$ (2) Å	Cell parameters from 13707 reflections
$b = 12.084$ (2) Å	$\theta = 3.5$–25.1º
$c = 14.919$ (3) Å	$\mu = 0.19$ mm^{-1}
$\alpha = 84.64$ (4)º	$T = 173$ (2) K
$\beta = 68.16$ (4)º	Block, colourless
$\gamma = 74.38$ (3)º	0.24 × 0.23 × 0.19 mm
$V = 1806.6$ (8) Å^3	

Data collection

Stoe IPDSII two-circle diffractometer	6759 independent reflections
Radiation source: fine-focus sealed tube	5130 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.059$
$T = 173$(2) K	$\theta_{max} = 25.7$º
ω scans	$\theta_{min} = 3.4$º
Absorption correction: multi-scan (MULABS; Spek, 2003; Blessing, 1995)	$h = -13\rightarrow13$
$T_{min} = 0.958$, $T_{max} = 0.968$	$k = -14\rightarrow14$
25452 measured reflections	$l = -18\rightarrow18$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$

$wR(F^2) = 0.103$

$S = 0.99$

6759 reflections

581 parameters

1 restraint

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

$w = 1/[\sigma^2(F_o^2) + (0.0641P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.22$ e Å^{-3}

$\Delta\rho_{min} = -0.43$ e Å^{-3}

Extinction correction: none

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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}	Occ. (<1)
O1	1.3775 (2)	−0.22947 (19)	0.22630 (14)	0.0412 (7)	0.778 (5)
H1O	1.318 (5)	−0.172 (4)	0.211 (3)	0.081 (14)*	0.778 (5)
O1'	1.1034 (6)	−0.2603 (7)	0.5458 (5)	0.052 (3)	0.222 (5)
H1'	1.0558	−0.2007	0.5307	0.062*	0.222 (5)
N1	0.52896 (17)	0.09054 (14)	0.60698 (12)	0.0243 (3)	
H1	0.454 (3)	0.116 (2)	0.6551 (18)	0.039 (7)*	
N2	0.90860 (16)	−0.03268 (14)	0.40931 (13)	0.0241 (3)	
H2	0.932 (2)	−0.0153 (19)	0.3513 (17)	0.027 (6)*	
N3	1.00050 (15)	−0.11454 (14)	0.43810 (12)	0.0256 (4)	
F1	0.31984 (13)	0.33610 (11)	0.61261 (10)	0.0474 (4)	
F2	0.23127 (13)	0.30820 (15)	0.51411 (10)	0.0577 (4)	
F3	0.26362 (11)	0.17934 (11)	0.61711 (9)	0.0396 (3)	
C1	0.62302 (19)	0.01488 (17)	0.63286 (14)	0.0265 (4)	
H1B	0.6005	−0.0089	0.6983	0.032*	
C2	0.74900 (19)	−0.02940 (16)	0.56998 (13)	0.0252 (4)	
H2B	0.8118	−0.0832	0.5914	0.030*	
C3	0.78468 (18)	0.00583 (15)	0.47277 (13)	0.0213 (4)	
C4	0.68345 (18)	0.08474 (15)	0.44273 (13)	0.0222 (4)	
C5	0.55447 (18)	0.12711 (16)	0.51282 (13)	0.0218 (4)	
C6	0.45417 (19)	0.20490 (16)	0.48440 (14)	0.0258 (4)	
C7	0.4832 (2)	0.23695 (19)	0.38910 (15)	0.0335 (5)	
H7	0.4167	0.2892	0.3703	0.040*	

C8	0.6095 (2)	0.1934 (2)	0.31951 (15)	0.0383 (5)	
H8	0.6270	0.2148	0.2538	0.046*	
C9	0.7080 (2)	0.12030 (19)	0.34560 (14)	0.0308 (4)	
H9	0.7937	0.0931	0.2980	0.037*	
C10	0.3180 (2)	0.25691 (18)	0.55618 (15)	0.0301 (4)	
C11	1.11525 (19)	−0.14833 (17)	0.37231 (14)	0.0272 (4)	
H11	1.1313	−0.1141	0.3103	0.033*	
C12	1.22281 (19)	−0.23742 (16)	0.38853 (14)	0.0250 (4)	
C13	1.3457 (2)	−0.27358 (18)	0.31309 (15)	0.0308 (4)	
H13	1.3620	−0.2358	0.2526	0.037*	0.222 (5)
C14	1.4443 (2)	−0.3651 (2)	0.32688 (18)	0.0395 (5)	
H14	1.5272	−0.3896	0.2755	0.047*	
C15	1.4219 (2)	−0.4200 (2)	0.41452 (18)	0.0428 (6)	
H15	1.4887	−0.4834	0.4226	0.051*	
C16	1.3023 (2)	−0.3834 (2)	0.49132 (18)	0.0432 (6)	
H16	1.2880	−0.4202	0.5522	0.052*	
C17	1.2044 (2)	−0.29276 (18)	0.47793 (16)	0.0339 (5)	
H17	1.1229	−0.2675	0.5304	0.041*	0.778 (5)
O1A	0.32607 (15)	0.20844 (12)	0.20878 (11)	0.0347 (3)	
H1OA	0.269 (3)	0.169 (2)	0.219 (2)	0.052 (8)*	
N1A	0.85453 (16)	0.67791 (15)	−0.06464 (11)	0.0249 (3)	
H1A	0.892 (2)	0.732 (2)	−0.0841 (17)	0.037 (7)*	
N2A	0.64348 (17)	0.43207 (15)	0.04510 (12)	0.0271 (4)	
H2A	0.676 (2)	0.358 (2)	0.0356 (17)	0.036 (6)*	
N3A	0.51353 (16)	0.46623 (14)	0.11072 (11)	0.0272 (4)	
F1A	1.26581 (13)	0.57259 (14)	−0.23741 (11)	0.0570 (4)	
F2A	1.09414 (14)	0.68921 (14)	−0.25668 (10)	0.0513 (4)	
F3A	1.12818 (13)	0.69213 (13)	−0.12537 (9)	0.0452 (3)	
C1A	0.72595 (18)	0.70800 (17)	−0.00552 (13)	0.0253 (4)	
H1A1	0.6845	0.7869	0.0101	0.030*	
C2A	0.65299 (19)	0.62951 (17)	0.03271 (13)	0.0255 (4)	
H2A1	0.5631	0.6539	0.0750	0.031*	
C3A	0.71164 (19)	0.51209 (16)	0.00923 (13)	0.0243 (4)	
C4A	0.85016 (19)	0.47867 (17)	−0.05538 (13)	0.0254 (4)	
C5A	0.92011 (19)	0.56478 (17)	−0.09054 (13)	0.0249 (4)	
C6A	1.0574 (2)	0.53261 (19)	−0.15102 (13)	0.0309 (5)	
C7A	1.1190 (2)	0.4183 (2)	−0.17435 (15)	0.0387 (5)	
H7A	1.2107	0.3967	−0.2134	0.046*	
C8A	1.0493 (2)	0.3342 (2)	−0.14170 (16)	0.0411 (6)	
H8A	1.0935	0.2562	−0.1595	0.049*	
C9A	0.9176 (2)	0.36283 (18)	−0.08410 (15)	0.0344 (5)	
H9A	0.8710	0.3046	−0.0631	0.041*	
C10A	1.1356 (2)	0.6208 (2)	−0.19247 (14)	0.0339 (5)	
C11A	0.4553 (2)	0.38410 (17)	0.14187 (14)	0.0277 (4)	
H11A	0.5008	0.3080	0.1185	0.033*	
C12A	0.3197 (2)	0.40731 (17)	0.21290 (14)	0.0269 (4)	
C13A	0.2583 (2)	0.31612 (17)	0.24648 (15)	0.0288 (4)	
C14A	0.1308 (2)	0.33696 (19)	0.31723 (16)	0.0346 (5)	
H14A	0.0902	0.2752	0.3409	0.042*	

C15A	0.0632 (2)	0.4472 (2)	0.35304 (18)	0.0400 (5)
H15A	−0.0232	0.4605	0.4014	0.048*
C16A	0.1210 (2)	0.5386 (2)	0.31869 (18)	0.0407 (5)
H16A	0.0739	0.6143	0.3425	0.049*
C17A	0.2477 (2)	0.51808 (19)	0.24951 (16)	0.0340 (5)
H17A	0.2871	0.5806	0.2262	0.041*
S1	0.86919 (5)	0.97050 (4)	0.84026 (3)	0.02299 (12)
O1S	0.98907 (14)	1.01484 (12)	0.79747 (10)	0.0305 (3)
O2S	0.87614 (14)	0.89968 (12)	0.92511 (10)	0.0322 (3)
O3S	0.85853 (15)	0.90418 (13)	0.76706 (10)	0.0355 (3)
O4S	0.75019 (14)	1.07005 (13)	0.87035 (11)	0.0379 (4)
O1W	0.2831 (2)	0.81279 (15)	0.96894 (15)	0.0537 (5)
H1WA	0.280 (3)	0.843 (3)	1.019 (3)	0.069 (10)*
H1WB	0.239 (4)	0.863 (3)	0.938 (3)	0.084 (11)*
O2W	0.50009 (19)	0.0371 (2)	0.89926 (14)	0.0581 (5)
H2WA	0.521 (8)	−0.051 (8)	0.906 (6)	0.26 (4)*
H2WB	0.582 (4)	0.054 (3)	0.893 (3)	0.095 (12)*
O3W	0.15544 (16)	0.01500 (14)	0.89528 (12)	0.0356 (4)
H3WA	0.125 (3)	0.047 (3)	0.949 (3)	0.069 (10)*
H3WB	0.091 (3)	0.022 (3)	0.873 (2)	0.069 (9)*
O4W	0.34561 (17)	0.13431 (16)	0.78822 (11)	0.0397 (4)
H4WA	0.395 (4)	0.111 (3)	0.818 (3)	0.089 (13)*
H4WB	0.283 (4)	0.093 (3)	0.817 (2)	0.074 (10)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0259 (11)	0.0488 (13)	0.0276 (11)	0.0117 (9)	−0.0022 (8)	0.0072 (9)
O1'	0.019 (4)	0.076 (6)	0.032 (4)	0.010 (3)	0.000 (3)	0.031 (4)
N1	0.0198 (8)	0.0262 (8)	0.0223 (8)	−0.0021 (7)	−0.0045 (7)	−0.0034 (7)
N2	0.0205 (8)	0.0259 (9)	0.0237 (9)	−0.0008 (7)	−0.0095 (7)	0.0012 (7)
N3	0.0200 (8)	0.0256 (8)	0.0309 (9)	0.0001 (7)	−0.0127 (7)	−0.0009 (7)
F1	0.0396 (8)	0.0431 (8)	0.0521 (8)	−0.0084 (6)	−0.0038 (6)	−0.0251 (6)
F2	0.0277 (7)	0.0840 (11)	0.0456 (8)	0.0193 (7)	−0.0176 (6)	−0.0047 (7)
F3	0.0223 (6)	0.0421 (7)	0.0479 (7)	−0.0074 (5)	−0.0047 (6)	−0.0049 (6)
C1	0.0263 (10)	0.0266 (10)	0.0238 (9)	−0.0029 (8)	−0.0089 (8)	0.0013 (8)
C2	0.0240 (10)	0.0240 (10)	0.0257 (10)	−0.0005 (8)	−0.0108 (8)	0.0004 (8)
C3	0.0195 (9)	0.0190 (9)	0.0262 (9)	−0.0032 (7)	−0.0096 (8)	−0.0031 (7)
C4	0.0218 (9)	0.0218 (9)	0.0236 (9)	−0.0039 (7)	−0.0098 (8)	−0.0014 (7)
C5	0.0207 (9)	0.0215 (9)	0.0247 (9)	−0.0035 (7)	−0.0101 (8)	−0.0042 (7)
C6	0.0225 (10)	0.0261 (10)	0.0297 (10)	−0.0015 (8)	−0.0125 (8)	−0.0050 (8)
C7	0.0270 (11)	0.0377 (12)	0.0351 (11)	0.0020 (9)	−0.0177 (9)	0.0014 (9)
C8	0.0310 (12)	0.0522 (14)	0.0257 (11)	0.0006 (10)	−0.0123 (9)	0.0044 (10)
C9	0.0219 (10)	0.0393 (12)	0.0249 (10)	−0.0009 (9)	−0.0065 (8)	0.0014 (9)
C10	0.0253 (10)	0.0312 (11)	0.0346 (11)	−0.0002 (9)	−0.0155 (9)	−0.0043 (9)
C11	0.0245 (10)	0.0267 (10)	0.0283 (10)	−0.0016 (8)	−0.0104 (8)	0.0002 (8)
C12	0.0215 (10)	0.0213 (9)	0.0331 (10)	0.0000 (8)	−0.0140 (8)	−0.0032 (8)
C13	0.0257 (10)	0.0313 (11)	0.0346 (11)	0.0006 (8)	−0.0143 (9)	−0.0047 (9)

C14	0.0270 (11)	0.0409 (12)	0.0478 (13)	0.0072 (9)	−0.0181 (10)	−0.0140 (11)
C15	0.0383 (13)	0.0344 (12)	0.0598 (15)	0.0068 (10)	−0.0331 (12)	−0.0010 (11)
C16	0.0420 (14)	0.0413 (13)	0.0506 (14)	−0.0050 (11)	−0.0278 (12)	0.0109 (11)
C17	0.0287 (12)	0.0332 (11)	0.0401 (12)	−0.0039 (9)	−0.0162 (10)	0.0028 (9)
O1A	0.0327 (8)	0.0241 (7)	0.0488 (9)	−0.0058 (6)	−0.0182 (7)	0.0030 (6)
N1A	0.0222 (8)	0.0281 (9)	0.0211 (8)	−0.0020 (7)	−0.0075 (7)	0.0021 (7)
N2A	0.0290 (9)	0.0226 (9)	0.0272 (9)	−0.0015 (7)	−0.0104 (7)	−0.0013 (7)
N3A	0.0249 (9)	0.0294 (9)	0.0261 (8)	−0.0041 (7)	−0.0097 (7)	−0.0008 (7)
F1A	0.0204 (7)	0.0686 (10)	0.0597 (9)	−0.0010 (6)	0.0036 (6)	−0.0009 (8)
F2A	0.0483 (8)	0.0716 (10)	0.0438 (8)	−0.0236 (7)	−0.0285 (7)	0.0298 (7)
F3A	0.0381 (7)	0.0619 (9)	0.0347 (7)	−0.0152 (6)	−0.0101 (6)	−0.0003 (6)
C1A	0.0216 (9)	0.0273 (10)	0.0219 (9)	0.0025 (8)	−0.0072 (8)	−0.0039 (8)
C2A	0.0195 (9)	0.0280 (10)	0.0220 (9)	0.0016 (8)	−0.0047 (8)	−0.0020 (8)
C3A	0.0255 (10)	0.0271 (10)	0.0186 (9)	0.0008 (8)	−0.0111 (8)	−0.0002 (7)
C4A	0.0256 (10)	0.0279 (10)	0.0165 (9)	0.0040 (8)	−0.0080 (8)	−0.0005 (7)
C5A	0.0236 (10)	0.0298 (10)	0.0153 (8)	0.0042 (8)	−0.0085 (8)	0.0018 (7)
C6A	0.0237 (10)	0.0427 (12)	0.0183 (9)	0.0035 (9)	−0.0074 (8)	0.0012 (8)
C7A	0.0270 (11)	0.0473 (13)	0.0244 (10)	0.0120 (10)	−0.0044 (9)	−0.0017 (9)
C8A	0.0408 (13)	0.0332 (12)	0.0311 (11)	0.0118 (10)	−0.0063 (10)	−0.0039 (9)
C9A	0.0369 (12)	0.0288 (11)	0.0268 (10)	0.0049 (9)	−0.0083 (9)	−0.0014 (8)
C10A	0.0209 (10)	0.0493 (13)	0.0221 (10)	0.0024 (9)	−0.0059 (8)	0.0034 (9)
C11A	0.0319 (11)	0.0258 (10)	0.0311 (10)	−0.0060 (8)	−0.0193 (9)	0.0027 (8)
C12A	0.0292 (10)	0.0281 (10)	0.0298 (10)	−0.0076 (8)	−0.0178 (9)	0.0015 (8)
C13A	0.0312 (11)	0.0266 (10)	0.0364 (11)	−0.0063 (8)	−0.0227 (9)	0.0040 (8)
C14A	0.0302 (11)	0.0342 (11)	0.0446 (12)	−0.0119 (9)	−0.0174 (10)	0.0034 (9)
C15A	0.0270 (11)	0.0438 (13)	0.0490 (14)	−0.0090 (10)	−0.0117 (10)	−0.0068 (11)
C16A	0.0344 (12)	0.0354 (12)	0.0531 (14)	−0.0068 (10)	−0.0147 (11)	−0.0140 (10)
C17A	0.0348 (12)	0.0307 (11)	0.0418 (12)	−0.0116 (9)	−0.0168 (10)	−0.0027 (9)
S1	0.0192 (2)	0.0248 (2)	0.0228 (2)	−0.00320 (18)	−0.00665 (18)	−0.00071 (18)
O1S	0.0268 (7)	0.0397 (8)	0.0277 (7)	−0.0130 (6)	−0.0113 (6)	0.0064 (6)
O2S	0.0354 (8)	0.0335 (8)	0.0252 (7)	−0.0094 (6)	−0.0085 (6)	0.0039 (6)
O3S	0.0429 (9)	0.0366 (8)	0.0314 (8)	−0.0144 (7)	−0.0143 (7)	−0.0036 (6)
O4S	0.0277 (8)	0.0406 (9)	0.0373 (8)	0.0068 (7)	−0.0120 (7)	−0.0061 (7)
O1W	0.0787 (14)	0.0294 (9)	0.0528 (11)	0.0094 (9)	−0.0367 (11)	−0.0120 (8)
O2W	0.0390 (10)	0.0758 (14)	0.0551 (11)	−0.0068 (10)	−0.0198 (9)	0.0093 (10)
O3W	0.0351 (9)	0.0434 (9)	0.0304 (8)	−0.0074 (7)	−0.0153 (7)	−0.0025 (7)
O4W	0.0249 (8)	0.0587 (11)	0.0285 (8)	−0.0048 (8)	−0.0051 (7)	−0.0025 (7)

Geometric parameters (Å, °)

O1—C13	1.311 (3)	N2A—C3A	1.343 (3)
O1—H1O	0.90 (5)	N2A—N3A	1.393 (2)
O1'—C17	1.201 (6)	N2A—H2A	0.88 (3)
O1'—H1'	0.8400	N3A—C11A	1.290 (3)
N1—C1	1.347 (3)	F1A—C10A	1.344 (2)
N1—C5	1.380 (3)	F2A—C10A	1.339 (2)
N1—H1	0.88 (3)	F3A—C10A	1.346 (3)
N2—C3	1.341 (3)	C1A—C2A	1.365 (3)
N2—N3	1.388 (2)	C1A—H1A1	0.9500

N2—H2	0.83 (2)	C2A—C3A	1.410 (3)
N3—C11	1.280 (3)	C2A—H2A1	0.9500
F1—C10	1.341 (2)	C3A—C4A	1.458 (3)
F2—C10	1.335 (2)	C4A—C5A	1.416 (3)
F3—C10	1.347 (3)	C4A—C9A	1.420 (3)
C1—C2	1.365 (3)	C5A—C6A	1.430 (3)
C1—H1B	0.9500	C6A—C7A	1.380 (3)
C2—C3	1.410 (3)	C6A—C10A	1.505 (3)
C2—H2B	0.9500	C7A—C8A	1.389 (4)
C3—C4	1.454 (3)	C7A—H7A	0.9500
C4—C9	1.418 (3)	C8A—C9A	1.369 (3)
C4—C5	1.422 (3)	C8A—H8A	0.9500
C5—C6	1.428 (3)	C9A—H9A	0.9500
C6—C7	1.378 (3)	C11A—C12A	1.463 (3)
C6—C10	1.505 (3)	C11A—H11A	0.9500
C7—C8	1.402 (3)	C12A—C17A	1.403 (3)
C7—H7	0.9500	C12A—C13A	1.410 (3)
C8—C9	1.371 (3)	C13A—C14A	1.398 (3)
C8—H8	0.9500	C14A—C15A	1.387 (3)
C9—H9	0.9500	C14A—H14A	0.9500
C11—C12	1.463 (3)	C15A—C16A	1.391 (3)
C11—H11	0.9500	C15A—H15A	0.9500
C12—C13	1.404 (3)	C16A—C17A	1.383 (3)
C12—C17	1.404 (3)	C16A—H16A	0.9500
C13—C14	1.399 (3)	C17A—H17A	0.9500
C13—H13	0.9500	S1—O3S	1.4701 (15)
C14—C15	1.377 (4)	S1—O2S	1.4752 (15)
C14—H14	0.9500	S1—O1S	1.4786 (15)
C15—C16	1.392 (4)	S1—O4S	1.4897 (16)
C15—H15	0.9500	O1W—H1WA	0.85 (3)
C16—C17	1.386 (3)	O1W—H1WB	0.88 (4)
C16—H16	0.9500	O2W—H2WA	1.04 (9)
C17—H17	0.9500	O2W—H2WB	0.96 (4)
O1A—C13A	1.369 (3)	O3W—H3WA	0.83 (4)
O1A—H1OA	0.86 (3)	O3W—H3WB	0.88 (4)
N1A—C1A	1.349 (3)	O4W—H4WA	0.81 (4)
N1A—C5A	1.380 (3)	O4W—H4WB	0.91 (4)
N1A—H1A	0.85 (3)		
C13—O1—H1O	120 (3)	C5A—N1A—H1A	122.5 (17)
C17—O1'—H1'	109.5	C3A—N2A—N3A	118.62 (17)
C1—N1—C5	121.24 (17)	C3A—N2A—H2A	125.6 (16)
C1—N1—H1	114.2 (16)	N3A—N2A—H2A	115.5 (16)
C5—N1—H1	124.5 (16)	C11A—N3A—N2A	114.67 (17)
C3—N2—N3	119.40 (17)	N1A—C1A—C2A	122.61 (18)
C3—N2—H2	123.2 (16)	N1A—C1A—H1A1	118.7
N3—N2—H2	117.1 (16)	C2A—C1A—H1A1	118.7
C11—N3—N2	115.17 (17)	C1A—C2A—C3A	119.73 (18)
N1—C1—C2	123.41 (18)	C1A—C2A—H2A1	120.1
N1—C1—H1B	118.3	C3A—C2A—H2A1	120.1

C2—C1—H1B	118.3	N2A—C3A—C2A	121.69 (18)
C1—C2—C3	119.09 (18)	N2A—C3A—C4A	120.16 (17)
C1—C2—H2B	120.5	C2A—C3A—C4A	118.16 (18)
C3—C2—H2B	120.5	C5A—C4A—C9A	118.79 (18)
N2—C3—C2	121.02 (17)	C5A—C4A—C3A	118.82 (17)
N2—C3—C4	120.64 (17)	C9A—C4A—C3A	122.40 (19)
C2—C3—C4	118.34 (17)	N1A—C5A—C4A	119.19 (17)
C9—C4—C5	118.53 (17)	N1A—C5A—C6A	121.44 (19)
C9—C4—C3	122.43 (17)	C4A—C5A—C6A	119.36 (18)
C5—C4—C3	119.04 (17)	C7A—C6A—C5A	119.4 (2)
N1—C5—C4	118.83 (17)	C7A—C6A—C10A	119.03 (19)
N1—C5—C6	121.51 (17)	C5A—C6A—C10A	121.59 (19)
C4—C5—C6	119.66 (17)	C6A—C7A—C8A	121.2 (2)
C7—C6—C5	119.53 (18)	C6A—C7A—H7A	119.4
C7—C6—C10	118.45 (18)	C8A—C7A—H7A	119.4
C5—C6—C10	121.99 (17)	C9A—C8A—C7A	120.6 (2)
C6—C7—C8	120.90 (19)	C9A—C8A—H8A	119.7
C6—C7—H7	119.5	C7A—C8A—H8A	119.7
C8—C7—H7	119.5	C8A—C9A—C4A	120.7 (2)
C9—C8—C7	120.48 (19)	C8A—C9A—H9A	119.7
C9—C8—H8	119.8	C4A—C9A—H9A	119.7
C7—C8—H8	119.8	F2A—C10A—F1A	106.92 (17)
C8—C9—C4	120.88 (19)	F2A—C10A—F3A	105.39 (18)
C8—C9—H9	119.6	F1A—C10A—F3A	105.61 (17)
C4—C9—H9	119.6	F2A—C10A—C6A	112.75 (17)
F2—C10—F1	106.85 (17)	F1A—C10A—C6A	112.27 (19)
F2—C10—F3	105.94 (17)	F3A—C10A—C6A	113.31 (16)
F1—C10—F3	105.51 (16)	N3A—C11A—C12A	120.48 (18)
F2—C10—C6	112.75 (17)	N3A—C11A—H11A	119.8
F1—C10—C6	112.05 (16)	C12A—C11A—H11A	119.8
F3—C10—C6	113.18 (17)	C17A—C12A—C13A	118.32 (19)
N3—C11—C12	122.74 (18)	C17A—C12A—C11A	122.14 (19)
N3—C11—H11	118.6	C13A—C12A—C11A	119.54 (18)
C12—C11—H11	118.6	O1A—C13A—C14A	121.48 (19)
C13—C12—C17	118.20 (18)	O1A—C13A—C12A	118.72 (19)
C13—C12—C11	120.45 (18)	C14A—C13A—C12A	119.80 (19)
C17—C12—C11	121.28 (18)	C15A—C14A—C13A	120.4 (2)
O1—C13—C14	114.9 (2)	C15A—C14A—H14A	119.8
O1—C13—C12	125.01 (19)	C13A—C14A—H14A	119.8
C14—C13—C12	120.1 (2)	C14A—C15A—C16A	120.5 (2)
C14—C13—H13	119.9	C14A—C15A—H15A	119.8
C12—C13—H13	119.9	C16A—C15A—H15A	119.8
C15—C14—C13	120.4 (2)	C17A—C16A—C15A	119.3 (2)
C15—C14—H14	119.8	C17A—C16A—H16A	120.4
C13—C14—H14	119.8	C15A—C16A—H16A	120.4
C14—C15—C16	120.6 (2)	C16A—C17A—C12A	121.7 (2)
C14—C15—H15	119.7	C16A—C17A—H17A	119.2
C16—C15—H15	119.7	C12A—C17A—H17A	119.2
C17—C16—C15	119.2 (2)	O3S—S1—O2S	111.38 (9)

C17—C16—H16	120.4	O3S—S1—O1S	109.47 (9)
C15—C16—H16	120.4	O2S—S1—O1S	109.60 (9)
O1'—C17—C16	118.2 (4)	O3S—S1—O4S	108.09 (9)
O1'—C17—C12	120.3 (4)	O2S—S1—O4S	109.72 (9)
C16—C17—C12	121.5 (2)	O1S—S1—O4S	108.52 (9)
C16—C17—H17	119.3	H1WA—O1W—H1WB	111 (3)
C12—C17—H17	119.3	H2WA—O2W—H2WB	103 (5)
C13A—O1A—H1OA	107.9 (19)	H3WA—O3W—H3WB	110 (3)
C1A—N1A—C5A	121.48 (18)	H4WA—O4W—H4WB	101 (3)
C1A—N1A—H1A	116.0 (17)		
C3—N2—N3—C11	178.41 (17)	C11—C12—C17—C16	174.8 (2)
C5—N1—C1—C2	1.1 (3)	C3A—N2A—N3A—C11A	−179.78 (16)
N1—C1—C2—C3	0.7 (3)	C5A—N1A—C1A—C2A	0.4 (3)
N3—N2—C3—C2	4.5 (3)	N1A—C1A—C2A—C3A	−1.1 (3)
N3—N2—C3—C4	−175.15 (15)	N3A—N2A—C3A—C2A	−3.1 (3)
C1—C2—C3—N2	177.82 (17)	N3A—N2A—C3A—C4A	176.57 (15)
C1—C2—C3—C4	−2.5 (3)	C1A—C2A—C3A—N2A	−179.91 (17)
N2—C3—C4—C9	3.0 (3)	C1A—C2A—C3A—C4A	0.4 (3)
C2—C3—C4—C9	−176.66 (17)	N2A—C3A—C4A—C5A	−178.81 (16)
N2—C3—C4—C5	−177.67 (16)	C2A—C3A—C4A—C5A	0.9 (2)
C2—C3—C4—C5	2.6 (2)	N2A—C3A—C4A—C9A	1.0 (3)
C1—N1—C5—C4	−0.9 (3)	C2A—C3A—C4A—C9A	−179.33 (18)
C1—N1—C5—C6	178.29 (17)	C1A—N1A—C5A—C4A	1.0 (3)
C9—C4—C5—N1	178.35 (16)	C1A—N1A—C5A—C6A	−178.22 (17)
C3—C4—C5—N1	−1.0 (2)	C9A—C4A—C5A—N1A	178.65 (17)
C9—C4—C5—C6	−0.9 (3)	C3A—C4A—C5A—N1A	−1.6 (3)
C3—C4—C5—C6	179.83 (16)	C9A—C4A—C5A—C6A	−2.1 (3)
N1—C5—C6—C7	−178.44 (18)	C3A—C4A—C5A—C6A	177.65 (16)
C4—C5—C6—C7	0.7 (3)	N1A—C5A—C6A—C7A	179.49 (18)
N1—C5—C6—C10	3.8 (3)	C4A—C5A—C6A—C7A	0.3 (3)
C4—C5—C6—C10	−177.00 (17)	N1A—C5A—C6A—C10A	−2.4 (3)
C5—C6—C7—C8	0.6 (3)	C4A—C5A—C6A—C10A	178.40 (17)
C10—C6—C7—C8	178.4 (2)	C5A—C6A—C7A—C8A	1.3 (3)
C6—C7—C8—C9	−1.8 (4)	C10A—C6A—C7A—C8A	−176.86 (19)
C7—C8—C9—C4	1.7 (3)	C6A—C7A—C8A—C9A	−1.0 (3)
C5—C4—C9—C8	−0.3 (3)	C7A—C8A—C9A—C4A	−1.0 (3)
C3—C4—C9—C8	178.94 (19)	C5A—C4A—C9A—C8A	2.5 (3)
C7—C6—C10—F2	14.8 (3)	C3A—C4A—C9A—C8A	−177.29 (19)
C5—C6—C10—F2	−167.38 (17)	C7A—C6A—C10A—F2A	111.4 (2)
C7—C6—C10—F1	−105.8 (2)	C5A—C6A—C10A—F2A	−66.7 (2)
C5—C6—C10—F1	72.0 (2)	C7A—C6A—C10A—F1A	−9.4 (3)
C7—C6—C10—F3	135.07 (19)	C5A—C6A—C10A—F1A	172.50 (17)
C5—C6—C10—F3	−47.2 (2)	C7A—C6A—C10A—F3A	−128.9 (2)
N2—N3—C11—C12	−177.54 (16)	C5A—C6A—C10A—F3A	53.0 (2)
N3—C11—C12—C13	178.24 (18)	N2A—N3A—C11A—C12A	178.52 (15)
N3—C11—C12—C17	1.3 (3)	N3A—C11A—C12A—C17A	0.3 (3)
C17—C12—C13—O1	−179.4 (2)	N3A—C11A—C12A—C13A	−179.57 (17)
C11—C12—C13—O1	3.6 (3)	C17A—C12A—C13A—O1A	177.83 (17)
C17—C12—C13—C14	2.2 (3)	C11A—C12A—C13A—O1A	−2.3 (3)

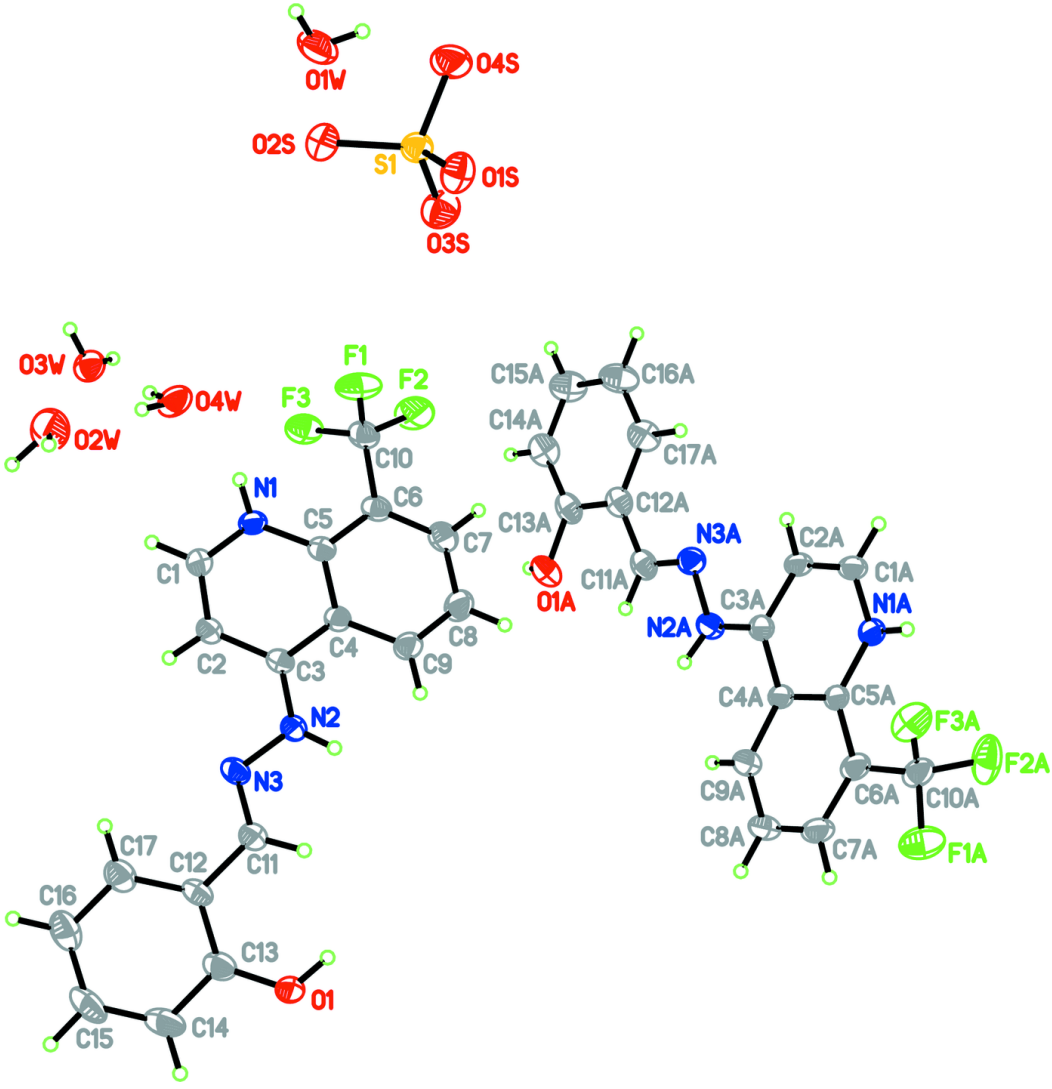
C11—C12—C13—C14	−174.81 (18)	C17A—C12A—C13A—C14A	−2.4 (3)
O1—C13—C14—C15	−178.9 (2)	C11A—C12A—C13A—C14A	177.42 (17)
C12—C13—C14—C15	−0.3 (3)	O1A—C13A—C14A—C15A	−178.85 (19)
C13—C14—C15—C16	−1.7 (3)	C12A—C13A—C14A—C15A	1.4 (3)
C14—C15—C16—C17	1.7 (4)	C13A—C14A—C15A—C16A	0.4 (3)
C15—C16—C17—O1'	−177.9 (6)	C14A—C15A—C16A—C17A	−1.1 (4)
C15—C16—C17—C12	0.3 (3)	C15A—C16A—C17A—C12A	0.0 (3)
C13—C12—C17—O1'	175.9 (6)	C13A—C12A—C17A—C16A	1.7 (3)
C11—C12—C17—O1'	−7.1 (6)	C11A—C12A—C17A—C16A	−178.12 (19)
C13—C12—C17—C16	−2.2 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1O···O4S^{i}	0.90 (5)	1.87 (5)	2.715 (3)	156 (4)
O1'—H1'···N3	0.84	1.84	2.602 (7)	149
N1—H1···O4W	0.88 (3)	1.90 (3)	2.713 (3)	153 (2)
N2—H2···O1S^{i}	0.83 (2)	2.06 (2)	2.873 (2)	166 (2)
O1A—H1OA···O3S^{ii}	0.86 (3)	1.81 (3)	2.671 (2)	175 (3)
N1A—H1A···O2S^{iii}	0.85 (3)	2.00 (3)	2.740 (2)	146 (2)
N2A—H2A···O1W^{ii}	0.88 (3)	1.99 (3)	2.855 (3)	169 (2)
O1W—H1WA···O4S^{iv}	0.85 (3)	1.92 (4)	2.752 (3)	170 (3)
O1W—H1WB···O3W^{v}	0.88 (4)	1.99 (4)	2.831 (3)	158 (3)
O2W—H2WA···O1A^{vi}	1.04 (9)	2.48 (9)	3.262 (3)	132 (6)
O2W—H2WB···O4S^{vii}	0.96 (4)	1.85 (4)	2.806 (3)	173 (3)
O3W—H3WA···O2S^{viii}	0.83 (4)	2.03 (4)	2.825 (2)	158 (3)
O3W—H3WB···O1S^{ix}	0.88 (4)	1.91 (4)	2.763 (2)	165 (3)
O4W—H4WA···O2W	0.81 (4)	1.99 (4)	2.793 (3)	171 (4)
O4W—H4WB···O3W	0.91 (4)	1.90 (4)	2.805 (3)	170 (3)

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

Fig. 1


O1W
O4S
O2S
S1
O1S
O3S
O3W
O4W
O2W
F1
F2
F3
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N1
C5
C6
C7
C1
C2
C3
C4
C8
C9
N2
N3
C11
C12
C17
C16
C13
O1
C15
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C15A
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C2A
C1A
C3A
N1A
C4A
C5A
F3A
F2A
C9A
C6A
C10A
C8A
C7A
F1A