Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Crystal structure and Hirshfeld surface analysis of dimethyl 3-methyl-8-{[4-(trifluoromethyl)phenyl]-sulfonyl}-7,8-dihydro-4*H*-4,6a-epoxybenzo[*b*]-naphtho[1,8-*de*]azepine-5,6-dicarboxylate

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Received 9 May 2025
Accepted 16 May 2025

Edited by X. Hao, Institute of Chemistry, Chinese Academy of Sciences

Keywords: crystal structure; disorder; acylation; furan; sulfonamide; 4+2 cycloaddition; weak interactions; Hirshfeld surface analysis.

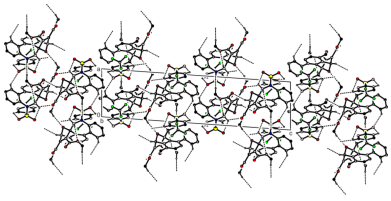
CCDC reference: 2451675

Supporting information: this article has supporting information at journals.iucr.org/e

The molecular conformation of the title compound, $C_{29}H_{22}F_3NO_7S$, is stable due to the intramolecular C—H···O hydrogen bonds. The central seven-membered ring adopts a distorted chair form. In the 7-oxabicyclo[2.2.1]hepta-2,5-diene unit, the five-membered rings adopt envelope conformations. In the crystal, the molecules are linked by C—H···O and C—H···F interactions, forming sheets parallel to the (002) plane. Additionally, S—O···π and π–π interactions [centroid-to-centroid distance = 3.6159 (7) Å] connect the molecules along the *a*-axis direction. van der Waals interactions between the molecular sheets reinforce the molecular packing. A Hirshfeld surface analysis was conducted to visualize the various intermolecular interactions, indicating that the largest contribution to the surface contacts is from H···H interactions (37.3%), followed by O···H/H···O (24.1%), F···H/H···F (19.0%), and C···H/H···C (10.3%) interactions.

1. Chemical context

7-Oxabicyclo[2.2.1]heptenes, products of the thermic reaction between furans and alkenes or alkynes, have great synthetic potential as a useful tool for the design of a broad diversity of substances with various practical properties. For example, these scaffolds can be used in the synthesis of polycyclic arenes – fragments of graphene – and serve as models for new carbon-based electronic materials (Eda *et al.*, 2015; Criado *et al.*, 2013; Furrer *et al.*, 2013). The 7-oxabicyclo[2.2.1]heptane moiety annelated with other rings serves as a scaffold for the preparation of molecular tweezers (Murphy *et al.*, 2016; Warrener *et al.*, 1999), supramolecular systems (Chou *et al.*, 2015; Oh *et al.*, 2010; Eckert-Maksić *et al.*, 2005), bridging donor–acceptor molecules (Chakrabarti *et al.*, 2007), various bioactive and natural products (Roscalesa *et al.*, 2017; Enev *et al.*, 2012; Gromov *et al.*, 2009; Schindler *et al.*, 2009; Vogel *et al.*, 1999), high-molecular-weight materials (Margetić *et al.*, 2010; Warrener *et al.*, 2001; Vogel *et al.*, 1999), *etc*. Under acid catalysis and temperature, cycloaddition intermediates can be converted into phenols, cyclohexenoles, or substituted aromatic hydrocarbons (Zaytsev *et al.*, 2019; Zubkov *et al.*, 2012*a*,*b*; Guliyeva *et al.*, 2024). Continuing our research into the chemistry of furyl-substituted sulfonamides (Burkin *et al.*, 2024; Mammadova *et al.*, 2023*a*,*b*), a new approach toward the



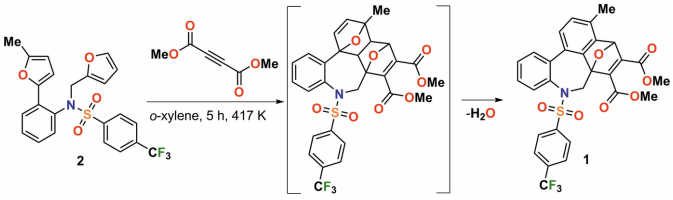
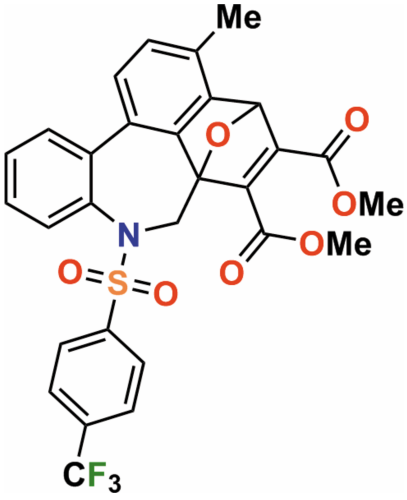


Figure 1
Synthesis of dimethyl 3-methyl-8-{[4-(trifluoromethyl)phenyl]sulfonyl}-7,8-dihydro-4*H*-4,6a-epoxybenzo[*b*]naphtho[1,8-*de*]azepine-5,6-dicarboxylate.

cycloaddition of dimethyl but-2-ynedioate (DMAD) with substituted furans (Zubkov *et al.*, 2009; Borisova *et al.*, 2018*a*, *b*) has been developed. In particular, in the course of the thermic [4 + 2] cycloaddition between DMAD and sulfamide **2**, an interesting sequence of reaction steps was observed; [4 + 2] cycloaddition, cleavage of the epoxy bridge, and a subsequent aromatization of the cyclohexene ring (Fig. 1).



2. Structural commentary

Fig. 2 shows the molecular structure of the title compound, intramolecular C—H···O hydrogen bonds, and naming of the rings in the molecule. The molecular conformation is stable due to the intramolecular hydrogen bonds C7—H7*B*···O17, C7—H7*A*···O1 and C19—H19···O2, which form *S*(6), *S*(5) and *S*(5) ring motifs, respectively (Fig. 2; Table 1; Bernstein *et al.*, 1995). Fig. 3 shows a detailed view of the central rings of the molecule. The central ring *A* (C6*A*/C7/N8/C8*A*/C12*A*/C12*B*/C12*C*) exhibits a distorted chair form [puckering parameters: $q2 = 0.708$ (1), $q3 = 207$ (1) Å, $\varphi(2) = -29.76$ (9), $\varphi(3) = -138.1$ (4) °, $Q_T = 0.738$ (1) Å, and spherical polar angle $\theta(2) = 73.70$ (9)°]. Ring *A* (r.m.s. deviation of fitted atoms = 0.2783 Å) subtends dihedral angles of 20.58 (5), 50.46 (5), 30.64 (5) and 28.18 (5)°, respectively, with rings *D* (C1–C3/C3*A*/C12*C*/C12*B*), *E* (C3*A*/C4–C6/C6*A*/C12*C*), *F* (C8*A*/C9–C12/C12*A*) and *G* (C18–C23). In the 7-oxabicyclo[2.2.1]hepta-2,5-diene unit, the five-membered rings *B* (O13/C4/C3*A*/C12*C*/C6*A*) and *C* (O13/C4–C6/C6*A*) show envelope conformations on atom O13 [*B*: $q(2) = 0.5436$ (12) Å, $\varphi(2) = 0.35$ (14)° and *C*: $q(2) = 0.5395$ (12) Å, $\varphi(2) = 179.95$ (14)°]. The bond lengths and angles in the title compound are in good agreement with those reported for related compounds (see *Database survey* section).

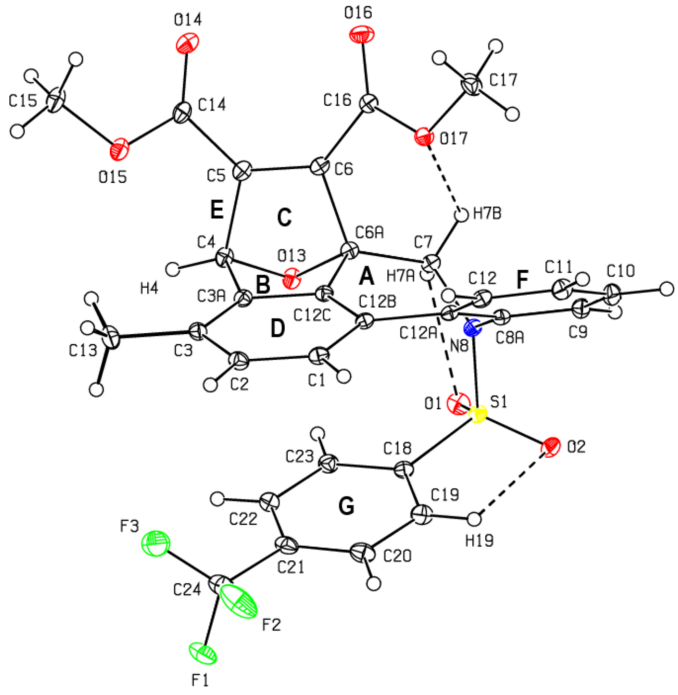


Figure 2
Molecular structure of the title compound showing atom labelling and ellipsoids at the 30% probability level. The minor disorder component has been omitted for clarity.

3. Supramolecular features and Hirshfeld surface analysis

In the crystal, molecules form $R_2^2(17)$ ring motifs by C—H···O interactions and are linked by C—H···F interactions to form sheets parallel to the (002) plane (Figs. 4 and 5; Table 1).

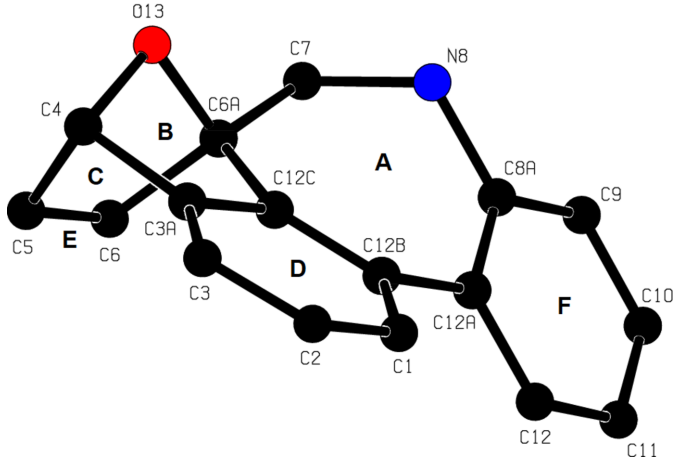


Figure 3
A detailed view of the central rings of the title molecule.

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O16^{i}	1.00	2.49	3.3893 (16)	149
C7—H7A···O1	0.99	2.35	2.8481 (15)	111
C7—H7B···O17	0.99	2.46	3.0071 (14)	115
C12—H12···O2^{ii}	0.95	2.55	3.1357 (15)	120
C15—H15A···O13^{ii}	0.98	2.47	3.3741 (17)	153
C17—H17B···F1^{iii}	0.98	2.54	3.3033 (17)	135
C19—H19···O2	0.95	2.52	2.9003 (17)	104
C22—H22···O14^{i}	0.95	2.37	3.2698 (19)	158

Symmetry codes: (i) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$; (ii) $x+1, y, z$; (iii) $x+1, y+1, z$.

Table 2
Summary of short interatomic contacts (Å) in the title compound.

Contact	distance	Symmetry operation
F3···H17C	2.72	$x, -1+y, z$
F1···H17B	2.54	$-1+x, -1+y, z$
H12···H1	2.54	$1-x, 1-y, 1-z$
O13···H15A	2.47	$-1+x, y, z$
O14···H22	2.37	$1-x, \frac{1}{2}+y, \frac{3}{2}-z$
O14···H15B	2.64	$2-x, \frac{1}{2}+y, \frac{3}{2}-z$
H22···O14	2.37	$1-x, -\frac{1}{2}+y, \frac{3}{2}-z$
H19···H19	2.27	$-x, 1-y, 1-z$
H17A···H10	2.51	$1-x, 2-y, 1-z$
H10···H9	2.58	$1-x, 2-y, 1-z$

Additionally, S—O···π (Fig. 5; Table 1) and π–π interactions [Fig. 6; $Cg3$···$Cg6$ = 3.6159 (7) Å, slippage = 0.804 Å; where $Cg3$ and $Cg6$ are the centroids of rings D (C1–C3/C3A/C12C/C12B) and G (C18–C23), respectively] link the molecules along the a-axis direction. van der Waals interactions between the molecular sheets reinforce the molecular packing.

Hirshfeld surfaces and the corresponding two-dimensional fingerprint plots were created using *CrystalExplorer17.5* (Spackman *et al.*, 2021) in order to visualize the intermolecular interactions (Tables 1 and 2). Fig. 7 shows the full two-dimensional fingerprint plot and those delineated into the major contacts: H···H (37.3%), O···H/H···O (24.1%), F···H/H···F (19.0%) and C···H/H···C (10.3%). Smaller

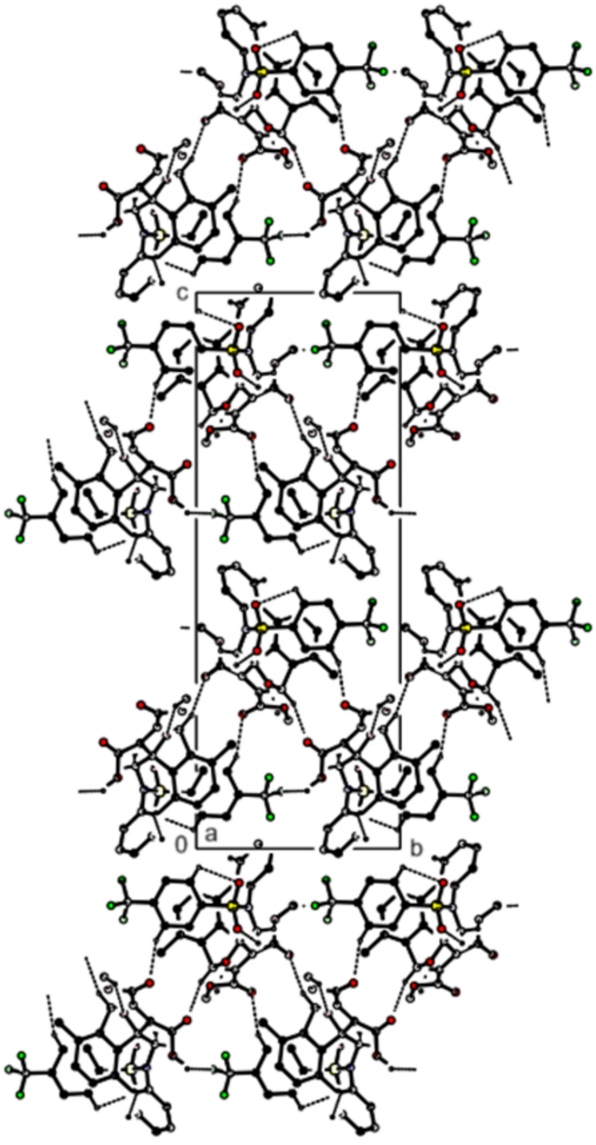


Figure 4
A view along the a axis of the title compound, showing the crystal packing. C—H···O and C—H···F hydrogen bonds are shown as dashed lines; H atoms not involved in hydrogen bonding have been omitted.

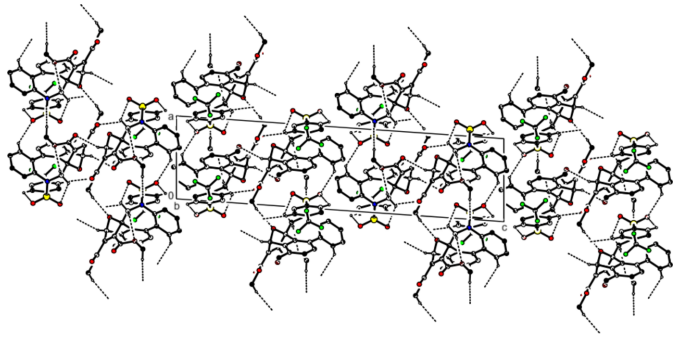


Figure 5
A view along the b axis of the title compound, showing the crystal packing. C—H···O and C—H···F hydrogen bonds are shown as dashed lines; H atoms not involved in hydrogen bonding have been omitted.

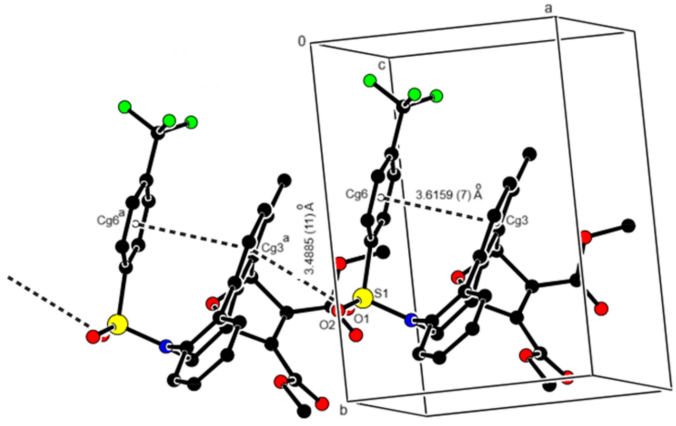


Figure 6
A partial packing diagram showing S—O···π and π–π interactions as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

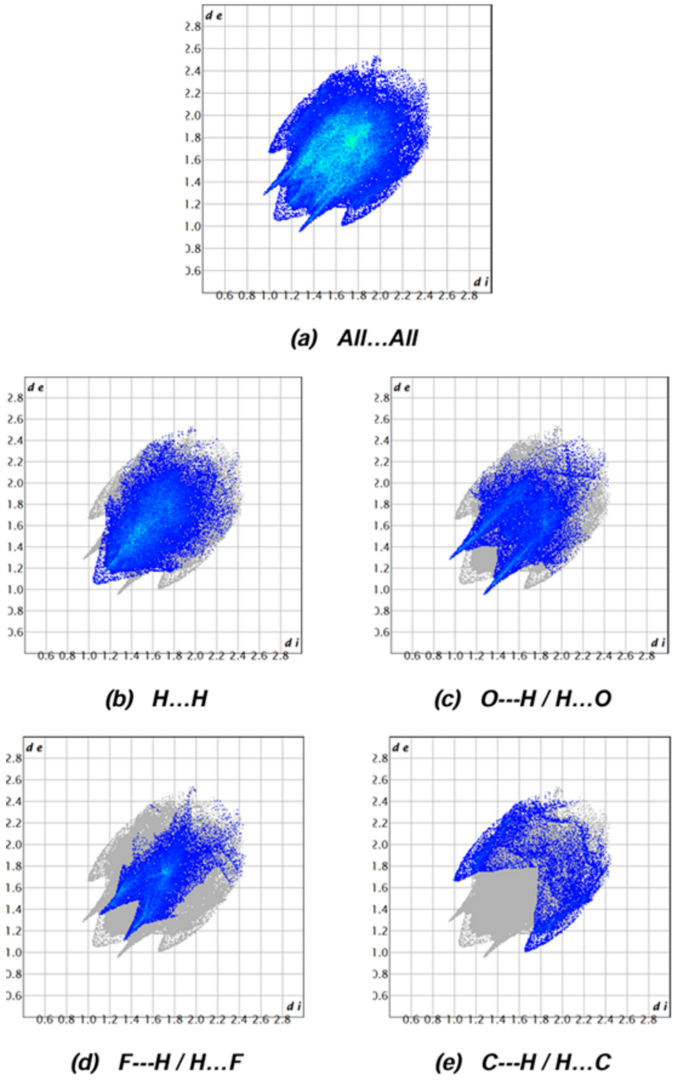


Figure 7
(*a*) The full two-dimensional fingerprint plot for the title compound and those delineated into (*b*) H···H, (*c*) O···H/H···O, (*c*) F···H/H···F and (*c*) C···H/H···C contacts.

contributions are made by O···C/C···O(4.9%), O···O(1.6%), C···C (1.5%), F···C/C···F (0.7%), F···O/O···F (0.4%), N···H/H···N (0.2%), S···C/C···S (0.1%) and S···H/H···S (0.1%) interactions.

4. Database survey

A search of the Cambridge Structural Database (Version 5.41, last update November 2019; Groom *et al.*, 2016) for 11-oxatricyclo[6.2.1.0^{2,7}]undecanes gave 739 hits, while a search for 3-methyl-11-oxatricyclo[6.2.1.0^{2,7}]undecanes gave zero hits. In these searches, the most related compounds are CSD refcode COKHAP (Sadikhova *et al.*, 2024) and POYBEL (Zubkov *et al.*, 2009). In COKHAP, two hexane rings and one oxane ring are fused together. The two hexane rings tend toward a distorted boat conformation, while the tetrahydrofuran and dihydrofuran rings adopt envelope conformations. The oxane ring is puckered. In the crystal, C—H···O hydrogen bonds connect the molecules into a three-dimensional network. POYBEL comprises a fused pentacyclic system containing two five-membered (cyclopentane and tetrahydrofuran) and three six-membered (tetrahydropyridinone, tetrahydropyridine and benzene) rings. Both five-membered rings of the bicyclic fragment have the usual envelope conformations, and the two central six-membered rings adopt sofa and non-symmetrical half-chair conformations.

In addition, three related compounds containing the O═S═O group are YIKROD (Mammadova *et al.*, 2023*a*), KETGID (Schinke *et al.*, 2022) and LUJKUA (Yakuth *et al.*, 2024). In YIKROD, intramolecular interactions are observed between the furan and benzene rings of the 4-cyanophenyl group. In the crystal, molecules are connected *via* C—H···O and C—H···N hydrogen bonds, forming layers parallel to the (100) plane. These layers are interconnected by C···H interactions and weak van der Waals interactions. In KETGID, the 1,2-oxazole and methanone fragments form an almost coplanar unit. The crystal structure features three short intermolecular C—H···O contacts involving the methanesulfonyl-O atoms. In LUJKUA, the asymmetric unit contains two distinct molecules, which exhibit differences in conformation resulting from a variation in key torsion angles. These distinctions influence the molecular orientation and intermolecular interactions, with strong N—H···N and N—H···O hydrogen bonds forming a centrosymmetric tetramer stabilized by π–π stacking.

5. Synthesis and crystallization

Dimethyl but-2-ynedioate (133.2 µL, 1.1 mmol) was added to a solution of *N*-(furan-2-ylmethyl)-*N*-[2-(5-methylfuran-2-yl) phenyl]-4-(trifluoromethyl)benzenesulfonamide **2** (100 mg, 0.22 mmol) in *o*-xylene (5 mL). The mixture was refluxed for 5 h. After cooling of the reaction to r.t, the solvent was evaporated under reduced pressure and the crude product was purified by column chromatography (eluent: from hexane to ethyl acetate). The title compound was obtained as colourless powder, yield 27%, 35 mg (0.059 mmol); m.p. 486–487 K. A single crystal of the title compound was grown from ethanol. IR (KBr), ν (cm^{-1}): 1753 (CO_2), 1325 (ν_{as} SO_2), 1169 (ν_s SO_2). ^{1}H NMR (700.2 MHz, $CDCl_3$) (*J*, Hz): δ 7.71 (*dd*, *J* = 7.6, 1.7, 1H, H Ar), 7.50–7.44 (*m*, 5H, H Ar), 7.20 (*d*, *J* = 8.1, 2H, H Ar), 6.69 (*d*, *J* = 7.9, 1H, H Ar), 6.61 (*d*, *J* = 8.1, 1H, H Ar), 5.91 (*s*, 1H, H Ar), 5.15 (*d*, *J* = 16.7, 1H, NC*H*), 4.47 (*d*, *J* = 16.7, 1H, NC*H*), 3.76 (*s*, 3H, OCH_3), 3.47 (*s*, 3H, OCH_3), 2.29 (*s*, 3H, CH_3). ^{13}C{^{1}H} NMR (176.1 MHz, $CDCl_3$): δ there are no signal of CF_3 163.1, 162.4, 151.2, 150.6, 145.3, 144.1, 142.7, 137.4, 137.0, 133.3 (*q*, *J* = 32.4, 1 C), 132.4, 130.9, 130.0, 129.7, 129.2 (2C), 128.3, 127.9 (2C), 126.1, 124.5 (*q*, *J* = 4.1, 2 C), 96.9, 81.3, 54.8, 52.5, 52.2, 17.4. ^{19}F{^{1}H} NMR (658.8 MHz, $CDCl_3$): −63.27. MS (ESI) *m/z*: $[M + H]^+$ 586. Elemental analysis calculated (%) for $C_{29}H_{22}F_3NO_7S$: C 59.49, H 3.79, N 2.39, S 5.48; found: C 59.81, H 3.48, N 2.19, S 5.33.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All C-bound H atoms were positioned geometrically (C—H = 0.95 and 1.00 Å) and refined using a riding model with U_{iso}(H) = 1.2 or 1.5U_{eq}(C). The methyl group (C13) attached to the benzene ring was found to be disordered over two positions with a refined occupancy ratio of 0.53 (2): 0.47 (2). A SADI instruction was used to restrain the C3—C13 and C3—C13′ bonds. The anisotropic displacement parameters of both parts of the carbon atom of the disordered methyl group were restrained to be similar with EADP instruction.

Table 3
Experimental details.

Crystal data	
Chemical formula	$C_{29}H_{22}F_3NO_7S$
M_r	585.53
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
a, b, c (Å)	7.6375 (5), 11.0324 (6), 30.2019 (8)
β (°)	93.983 (1)
V (Å^3)	2538.7 (2)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	1.79
Crystal size (mm)	0.35 × 0.18 × 0.17
Data collection	
Diffractometer	Rigaku XtaLAB Synergy-S, HyPix-6000HE area-detector
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2021)
T_{min}, T_{max}	0.713, 0.737
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	31570, 5526, 5187
R_{int}	0.049
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.639
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.037, 0.103, 1.05
No. of reflections	5526
No. of parameters	379
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.45, −0.43

Computer programs: *CrysAlis PRO* (Rigaku OD, 2021), *SHELXT2016/6* (Sheldrick, 2015*a*), *SHELXL2016/6* (Sheldrick, 2015*b*, *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2020).

Acknowledgements

GMB and SA thank to Common Use Center 'Physical and Chemical Research of New Materials, Substances and Catalytic Systems'. This publication has been supported by the RUDN University Scientific Projects Grant System, project No. 021408–2-000, as well as by the Baku Engineering University (Azerbaijan) and Azerbaijan Medical University. The author's contributions are as follows. Conceptualization, MA and GMM; synthesis, GMB and SA; AGK NMR analysis; X-ray analysis, VNK, NAG; writing (review and editing of the manuscript) MA and GMM; funding acquisition KIH; supervision, MA and GMM.

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supporting information

Acta Cryst. (2025). E**81**, 543-548 [https://doi.org/10.1107/S2056989025004426]

Crystal structure and Hirshfeld surface analysis of dimethyl 3-methyl-8-{[4-(trifluoromethyl)phenyl]sulfonyl}-7,8-dihydro-4*H*-4,6a-epoxybenzo[*b*]naphtho[1,8-*de*]azepine-5,6-dicarboxylate

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Computing details

Dimethyl 3-methyl-8-{[4-(trifluoromethyl)phenyl]sulfonyl}-7,8-dihydro-4*H*-4,6a-epoxybenzo[*b*]naphtho[1,8-*de*]azepine-5,6-dicarboxylate

Crystal data

$C_{29}H_{22}F_3NO_7S$
$M_r = 585.53$
Monoclinic, $P2_1/c$
$a = 7.6375$ (5) Å
$b = 11.0324$ (6) Å
$c = 30.2019$ (8) Å
$\beta = 93.983$ (1)°
$V = 2538.7$ (2) Å^3
$Z = 4$
$F(000) = 1208$
$D_x = 1.532$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 20964 reflections
$\theta = 2.9$–$79.9°$
$\mu = 1.79$ mm^{-1}
$T = 100$ K
Prism, colourless
0.35 × 0.18 × 0.17 mm

Data collection

Rigaku XtaLAB Synergy-S, HyPix-6000HE area-detector diffractometer
Radiation source: micro-focus sealed X-ray tube
φ and ω scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2021)
$T_{min} = 0.713$, $T_{max} = 0.737$
31570 measured reflections
5526 independent reflections
5187 reflections with $I > 2\sigma(I)$
$R_{int} = 0.049$
$\theta_{max} = 80.1°$, $\theta_{min} = 2.9°$
$h = -9 \rightarrow 8$
$k = -14 \rightarrow 13$
$l = -38 \rightarrow 38$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.103$
$S = 1.05$
5526 reflections
379 parameters
1 restraint
Primary atom site location: difference Fourier map
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0633P)^2 + 0.8141P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.45$ e Å^{-3}
$\Delta\rho_{min} = -0.43$ e Å^{-3}

Extinction correction: SHELXL-2019/2 (Sheldrick, 2015b), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00089 (14)

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
S1	−0.08268 (3)	0.68046 (3)	0.60331 (2)	0.01536 (10)	
F1	0.03672 (12)	0.08285 (8)	0.60261 (3)	0.0324 (2)	
F2	0.22478 (16)	0.13678 (9)	0.55695 (5)	0.0501 (3)	
F3	0.29014 (14)	0.14333 (10)	0.62756 (5)	0.0539 (3)	
O1	−0.16062 (12)	0.70213 (9)	0.64434 (3)	0.0216 (2)	
O2	−0.17402 (12)	0.70996 (9)	0.56162 (3)	0.0225 (2)	
C1	0.48862 (15)	0.49339 (11)	0.57852 (4)	0.0171 (2)	
H1	0.496038	0.479440	0.547665	0.021*	
C2	0.55556 (16)	0.40746 (11)	0.60859 (4)	0.0187 (2)	
H2	0.607100	0.336071	0.597644	0.022*	
C3	0.54990 (15)	0.42217 (11)	0.65483 (4)	0.0172 (2)	
C3A	0.47063 (15)	0.52723 (11)	0.66816 (4)	0.0156 (2)	
C4	0.44151 (15)	0.58212 (12)	0.71373 (4)	0.0171 (2)	
H4	0.444763	0.524274	0.739264	0.021*	
C5	0.56803 (16)	0.69088 (11)	0.71852 (4)	0.0165 (2)	
C6	0.50225 (15)	0.77414 (11)	0.68956 (4)	0.0149 (2)	
C6A	0.33411 (15)	0.71572 (11)	0.66698 (4)	0.0141 (2)	
C7	0.18668 (15)	0.79745 (11)	0.64834 (4)	0.0158 (2)	
H7A	0.096592	0.803245	0.670287	0.019*	
H7B	0.234316	0.879823	0.644221	0.019*	
N8	0.10246 (13)	0.75554 (9)	0.60592 (3)	0.0148 (2)	
C8A	0.19749 (15)	0.76703 (11)	0.56657 (4)	0.0144 (2)	
C9	0.14221 (16)	0.85518 (12)	0.53567 (4)	0.0186 (2)	
H9	0.041411	0.902688	0.540357	0.022*	
C10	0.23335 (17)	0.87407 (12)	0.49810 (4)	0.0217 (3)	
H10	0.195141	0.934034	0.477015	0.026*	
C11	0.38118 (17)	0.80441 (12)	0.49158 (4)	0.0202 (3)	
H11	0.445235	0.817581	0.466155	0.024*	
C12	0.43538 (16)	0.71573 (12)	0.52212 (4)	0.0171 (2)	
H12	0.535760	0.668175	0.517074	0.020*	
C12A	0.34499 (15)	0.69485 (11)	0.56029 (4)	0.0143 (2)	
C12B	0.40963 (14)	0.60123 (11)	0.59260 (4)	0.0144 (2)	
C12C	0.40171 (14)	0.61400 (11)	0.63793 (4)	0.0137 (2)	
O13	0.27786 (11)	0.64524 (8)	0.70381 (3)	0.01706 (19)	
C13	0.6235 (19)	0.3275 (12)	0.6870 (4)	0.0238 (3)	0.53 (2)

H13A	0.732679	0.294903	0.676576	0.036*	0.53 (2)
H13B	0.538176	0.261703	0.689067	0.036*	0.53 (2)
H13C	0.647483	0.364271	0.716356	0.036*	0.53 (2)
C13′	0.625 (2)	0.3275 (13)	0.6867 (5)	0.0238 (3)	0.47 (2)
H13D	0.747394	0.348027	0.695799	0.036*	0.47 (2)
H13E	0.621140	0.248114	0.672104	0.036*	0.47 (2)
H13F	0.556513	0.324858	0.712874	0.036*	0.47 (2)
C14	0.74252 (17)	0.68539 (12)	0.74318 (4)	0.0187 (3)	
O14	0.82551 (14)	0.77048 (10)	0.75805 (4)	0.0298 (2)	
O15	0.79316 (12)	0.56938 (9)	0.74709 (3)	0.0221 (2)	
C15	0.96586 (17)	0.54877 (14)	0.76935 (5)	0.0252 (3)	
H15A	1.051895	0.601521	0.756353	0.038*	
H15B	0.999568	0.463854	0.765660	0.038*	
H15C	0.962354	0.567077	0.801032	0.038*	
C16	0.58807 (15)	0.88141 (11)	0.67119 (4)	0.0160 (2)	
O16	0.68066 (13)	0.95432 (9)	0.69113 (3)	0.0247 (2)	
O17	0.54422 (11)	0.88538 (8)	0.62730 (3)	0.01773 (19)	
C17	0.63324 (19)	0.97498 (13)	0.60257 (5)	0.0244 (3)	
H17A	0.585829	0.974193	0.571579	0.037*	
H17B	0.759006	0.956514	0.603902	0.037*	
H17C	0.615553	1.055289	0.615390	0.037*	
C18	−0.02522 (15)	0.52580 (11)	0.60113 (4)	0.0161 (2)	
C19	−0.01841 (17)	0.47033 (12)	0.56005 (4)	0.0213 (3)	
H19	−0.055255	0.512804	0.533666	0.026*	
C20	0.04285 (18)	0.35202 (13)	0.55788 (5)	0.0241 (3)	
H20	0.049166	0.312688	0.530058	0.029*	
C21	0.09484 (17)	0.29210 (12)	0.59721 (5)	0.0220 (3)	
C22	0.08252 (17)	0.34703 (12)	0.63835 (5)	0.0224 (3)	
H22	0.115329	0.303757	0.664827	0.027*	
C23	0.02216 (17)	0.46516 (12)	0.64049 (4)	0.0195 (3)	
H23	0.013327	0.503979	0.668321	0.023*	
C24	0.16202 (19)	0.16488 (13)	0.59579 (6)	0.0296 (3)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01088 (15)	0.01765 (16)	0.01767 (16)	0.00027 (9)	0.00183 (11)	0.00049 (10)
F1	0.0335 (5)	0.0180 (4)	0.0464 (5)	−0.0065 (3)	0.0085 (4)	−0.0001 (4)
F2	0.0591 (7)	0.0245 (5)	0.0717 (8)	0.0035 (4)	0.0407 (6)	−0.0050 (5)
F3	0.0374 (5)	0.0263 (5)	0.0941 (10)	0.0095 (4)	−0.0236 (6)	−0.0078 (5)
O1	0.0165 (4)	0.0240 (5)	0.0252 (5)	0.0012 (3)	0.0086 (4)	−0.0018 (4)
O2	0.0159 (4)	0.0257 (5)	0.0250 (5)	−0.0014 (3)	−0.0049 (3)	0.0034 (4)
C1	0.0160 (5)	0.0185 (6)	0.0170 (5)	−0.0007 (4)	0.0020 (4)	−0.0035 (4)
C2	0.0174 (5)	0.0157 (5)	0.0231 (6)	0.0007 (4)	0.0027 (4)	−0.0026 (4)
C3	0.0137 (5)	0.0166 (6)	0.0213 (6)	−0.0015 (4)	0.0013 (4)	0.0031 (4)
C3A	0.0129 (5)	0.0186 (6)	0.0154 (5)	−0.0025 (4)	0.0017 (4)	0.0016 (4)
C4	0.0153 (5)	0.0215 (6)	0.0148 (5)	0.0005 (4)	0.0025 (4)	0.0028 (4)
C5	0.0169 (6)	0.0218 (6)	0.0110 (5)	0.0010 (4)	0.0028 (4)	−0.0018 (4)

C6	0.0143 (5)	0.0193 (6)	0.0114 (5)	0.0005 (4)	0.0023 (4)	−0.0036 (4)
C6A	0.0137 (5)	0.0174 (5)	0.0116 (5)	−0.0014 (4)	0.0035 (4)	0.0002 (4)
C7	0.0142 (5)	0.0178 (5)	0.0155 (5)	0.0011 (4)	0.0013 (4)	−0.0029 (4)
N8	0.0123 (4)	0.0175 (5)	0.0144 (5)	−0.0002 (4)	0.0012 (4)	−0.0006 (4)
C8A	0.0133 (5)	0.0157 (5)	0.0141 (5)	−0.0026 (4)	0.0011 (4)	−0.0011 (4)
C9	0.0182 (5)	0.0183 (6)	0.0192 (6)	0.0011 (4)	0.0003 (4)	0.0015 (5)
C10	0.0244 (6)	0.0218 (6)	0.0188 (6)	−0.0007 (5)	0.0002 (5)	0.0054 (5)
C11	0.0210 (6)	0.0258 (6)	0.0141 (5)	−0.0032 (5)	0.0033 (4)	0.0017 (5)
C12	0.0150 (5)	0.0217 (6)	0.0146 (5)	−0.0011 (4)	0.0011 (4)	−0.0027 (4)
C12A	0.0139 (5)	0.0161 (5)	0.0128 (5)	−0.0021 (4)	−0.0010 (4)	−0.0022 (4)
C12B	0.0117 (5)	0.0165 (5)	0.0151 (5)	−0.0014 (4)	0.0012 (4)	−0.0010 (4)
C12C	0.0108 (5)	0.0148 (5)	0.0155 (5)	−0.0007 (4)	0.0018 (4)	−0.0009 (4)
O13	0.0149 (4)	0.0225 (4)	0.0142 (4)	0.0008 (3)	0.0043 (3)	0.0036 (3)
C13	0.0248 (7)	0.0199 (6)	0.0267 (7)	0.0026 (5)	0.0027 (6)	0.0074 (5)
C13′	0.0248 (7)	0.0199 (6)	0.0267 (7)	0.0026 (5)	0.0027 (6)	0.0074 (5)
C14	0.0192 (6)	0.0258 (6)	0.0112 (5)	0.0013 (5)	0.0007 (4)	0.0012 (4)
O14	0.0306 (5)	0.0287 (5)	0.0282 (5)	−0.0025 (4)	−0.0117 (4)	−0.0016 (4)
O15	0.0185 (4)	0.0259 (5)	0.0216 (4)	0.0025 (4)	−0.0020 (3)	0.0031 (4)
C15	0.0177 (6)	0.0360 (7)	0.0217 (6)	0.0042 (5)	−0.0010 (5)	0.0066 (5)
C16	0.0151 (5)	0.0168 (5)	0.0164 (5)	0.0021 (4)	0.0024 (4)	−0.0020 (4)
O16	0.0266 (5)	0.0233 (5)	0.0236 (5)	−0.0069 (4)	−0.0016 (4)	−0.0053 (4)
O17	0.0190 (4)	0.0192 (4)	0.0150 (4)	−0.0035 (3)	0.0011 (3)	0.0015 (3)
C17	0.0274 (6)	0.0215 (6)	0.0246 (6)	−0.0037 (5)	0.0051 (5)	0.0066 (5)
C18	0.0127 (5)	0.0180 (6)	0.0179 (6)	−0.0022 (4)	0.0028 (4)	−0.0001 (4)
C19	0.0243 (6)	0.0231 (6)	0.0167 (6)	−0.0038 (5)	0.0035 (5)	−0.0010 (5)
C20	0.0276 (7)	0.0220 (6)	0.0235 (6)	−0.0045 (5)	0.0069 (5)	−0.0050 (5)
C21	0.0169 (6)	0.0171 (6)	0.0325 (7)	−0.0035 (5)	0.0045 (5)	−0.0018 (5)
C22	0.0218 (6)	0.0198 (6)	0.0250 (6)	−0.0031 (5)	−0.0016 (5)	0.0030 (5)
C23	0.0210 (6)	0.0199 (6)	0.0175 (6)	−0.0025 (5)	0.0010 (4)	−0.0003 (4)
C24	0.0227 (7)	0.0199 (6)	0.0469 (9)	−0.0026 (5)	0.0067 (6)	−0.0021 (6)

Geometric parameters (Å, º)

S1—O1	1.4314 (9)	C10—H10	0.9500
S1—O2	1.4338 (10)	C11—C12	1.3877 (18)
S1—N8	1.6359 (10)	C11—H11	0.9500
S1—C18	1.7642 (13)	C12—C12A	1.4034 (16)
F1—C24	1.3434 (17)	C12—H12	0.9500
F2—C24	1.334 (2)	C12A—C12B	1.4819 (17)
F3—C24	1.343 (2)	C12B—C12C	1.3816 (16)
C1—C2	1.3857 (18)	C13—H13A	0.9800
C1—C12B	1.4129 (17)	C13—H13B	0.9800
C1—H1	0.9500	C13—H13C	0.9800
C2—C3	1.4096 (18)	C13′—H13D	0.9800
C2—H2	0.9500	C13′—H13E	0.9800
C3—C3A	1.3805 (17)	C13′—H13F	0.9800
C3—C13′	1.507 (10)	C14—O14	1.2022 (18)
C3—C13	1.509 (9)	C14—O15	1.3400 (17)

C3A—C12C	1.4001 (17)	O15—C15	1.4562 (15)
C3A—C4	1.5340 (17)	C15—H15A	0.9800
C4—O13	1.4439 (15)	C15—H15B	0.9800
C4—C5	1.5411 (17)	C15—H15C	0.9800
C4—H4	1.0000	C16—O16	1.2045 (16)
C5—C6	1.3416 (18)	C16—O17	1.3452 (15)
C5—C14	1.4820 (17)	O17—C17	1.4378 (15)
C6—C16	1.4795 (17)	C17—H17A	0.9800
C6—C6A	1.5517 (16)	C17—H17B	0.9800
C6A—O13	1.4466 (13)	C17—H17C	0.9800
C6A—C7	1.5198 (16)	C18—C19	1.3876 (17)
C6A—C12C	1.5358 (16)	C18—C23	1.3899 (18)
C7—N8	1.4680 (15)	C19—C20	1.390 (2)
C7—H7A	0.9900	C19—H19	0.9500
C7—H7B	0.9900	C20—C21	1.393 (2)
N8—C8A	1.4405 (15)	C20—H20	0.9500
C8A—C9	1.3928 (17)	C21—C22	1.391 (2)
C8A—C12A	1.4031 (17)	C21—C24	1.4961 (19)
C9—C10	1.3877 (18)	C22—C23	1.3854 (19)
C9—H9	0.9500	C22—H22	0.9500
C10—C11	1.3910 (19)	C23—H23	0.9500

O1—S1—O2	121.06 (6)	C12—C12A—C12B	119.67 (11)
O1—S1—N8	106.49 (6)	C12C—C12B—C1	115.67 (11)
O2—S1—N8	107.06 (5)	C12C—C12B—C12A	123.09 (11)
O1—S1—C18	108.27 (6)	C1—C12B—C12A	121.22 (11)
O2—S1—C18	107.08 (6)	C12B—C12C—C3A	122.41 (11)
N8—S1—C18	105.97 (5)	C12B—C12C—C6A	132.85 (11)
C2—C1—C12B	121.64 (11)	C3A—C12C—C6A	104.66 (10)
C2—C1—H1	119.2	C4—O13—C6A	96.88 (8)
C12B—C1—H1	119.2	C3—C13—H13A	109.5
C1—C2—C3	122.36 (11)	C3—C13—H13B	109.5
C1—C2—H2	118.8	H13A—C13—H13B	109.5
C3—C2—H2	118.8	C3—C13—H13C	109.5
C3A—C3—C2	115.46 (11)	H13A—C13—H13C	109.5
C3A—C3—C13′	123.5 (7)	H13B—C13—H13C	109.5
C2—C3—C13′	121.1 (7)	C3—C13′—H13D	109.5
C3A—C3—C13	123.0 (6)	C3—C13′—H13E	109.5
C2—C3—C13	121.5 (6)	H13D—C13′—H13E	109.5
C3—C3A—C12C	122.45 (11)	C3—C13′—H13F	109.5
C3—C3A—C4	133.39 (11)	H13D—C13′—H13F	109.5
C12C—C3A—C4	104.11 (10)	H13E—C13′—H13F	109.5
O13—C4—C3A	100.46 (9)	O14—C14—O15	124.85 (12)
O13—C4—C5	99.91 (10)	O14—C14—C5	126.04 (12)
C3A—C4—C5	105.22 (9)	O15—C14—C5	109.10 (11)
O13—C4—H4	116.3	C14—O15—C15	115.88 (11)
C3A—C4—H4	116.3	O15—C15—H15A	109.5
C5—C4—H4	116.3	O15—C15—H15B	109.5

C6—C5—C14	129.68 (12)	H15A—C15—H15B	109.5
C6—C5—C4	105.54 (11)	O15—C15—H15C	109.5
C14—C5—C4	123.47 (11)	H15A—C15—H15C	109.5
C5—C6—C16	129.54 (11)	H15B—C15—H15C	109.5
C5—C6—C6A	105.26 (10)	O16—C16—O17	124.64 (12)
C16—C6—C6A	122.82 (10)	O16—C16—C6	127.35 (12)
O13—C6A—C7	110.59 (9)	O17—C16—C6	108.01 (10)
O13—C6A—C12C	100.14 (9)	C16—O17—C17	116.09 (10)
C7—C6A—C12C	119.46 (10)	O17—C17—H17A	109.5
O13—C6A—C6	99.57 (9)	O17—C17—H17B	109.5
C7—C6A—C6	119.05 (10)	H17A—C17—H17B	109.5
C12C—C6A—C6	104.71 (9)	O17—C17—H17C	109.5
N8—C7—C6A	113.89 (10)	H17A—C17—H17C	109.5
N8—C7—H7A	108.8	H17B—C17—H17C	109.5
C6A—C7—H7A	108.8	C19—C18—C23	121.92 (12)
N8—C7—H7B	108.8	C19—C18—S1	119.01 (10)
C6A—C7—H7B	108.8	C23—C18—S1	118.95 (10)
H7A—C7—H7B	107.7	C18—C19—C20	119.34 (12)
C8A—N8—C7	118.49 (9)	C18—C19—H19	120.3
C8A—N8—S1	119.23 (8)	C20—C19—H19	120.3
C7—N8—S1	121.75 (8)	C19—C20—C21	118.86 (12)
C9—C8A—C12A	120.98 (11)	C19—C20—H20	120.6
C9—C8A—N8	117.87 (11)	C21—C20—H20	120.6
C12A—C8A—N8	121.12 (10)	C22—C21—C20	121.45 (13)
C10—C9—C8A	120.45 (12)	C22—C21—C24	118.61 (13)
C10—C9—H9	119.8	C20—C21—C24	119.93 (13)
C8A—C9—H9	119.8	C23—C22—C21	119.65 (12)
C9—C10—C11	119.40 (12)	C23—C22—H22	120.2
C9—C10—H10	120.3	C21—C22—H22	120.2
C11—C10—H10	120.3	C22—C23—C18	118.72 (12)
C12—C11—C10	120.20 (12)	C22—C23—H23	120.6
C12—C11—H11	119.9	C18—C23—H23	120.6
C10—C11—H11	119.9	F2—C24—F3	107.37 (13)
C11—C12—C12A	121.37 (11)	F2—C24—F1	106.36 (13)
C11—C12—H12	119.3	F3—C24—F1	105.22 (13)
C12A—C12—H12	119.3	F2—C24—C21	112.84 (13)
C8A—C12A—C12	117.59 (11)	F3—C24—C21	112.32 (13)
C8A—C12A—C12B	122.72 (10)	F1—C24—C21	112.23 (11)

C12B—C1—C2—C3	−0.27 (19)	C12—C12A—C12B—C12C	143.46 (12)
C1—C2—C3—C3A	0.97 (18)	C8A—C12A—C12B—C1	146.80 (12)
C1—C2—C3—C13′	−179.4 (8)	C12—C12A—C12B—C1	−34.76 (17)
C1—C2—C3—C13	180.0 (7)	C1—C12B—C12C—C3A	1.32 (17)
C2—C3—C3A—C12C	−0.53 (17)	C12A—C12B—C12C—C3A	−176.99 (11)
C13′—C3—C3A—C12C	179.9 (8)	C1—C12B—C12C—C6A	177.79 (11)
C13—C3—C3A—C12C	−179.5 (7)	C12A—C12B—C12C—C6A	−0.5 (2)
C2—C3—C3A—C4	−177.38 (12)	C3—C3A—C12C—C12B	−0.65 (18)
C13′—C3—C3A—C4	3.0 (8)	C4—C3A—C12C—C12B	177.00 (11)

C13—C3—C3A—C4	3.6 (7)
C3—C3A—C4—O13	−148.68 (13)
C12C—C3A—C4—O13	34.06 (11)
C3—C3A—C4—C5	107.93 (15)
C12C—C3A—C4—C5	−69.34 (11)
O13—C4—C5—C6	−33.53 (11)
C3A—C4—C5—C6	70.27 (12)
O13—C4—C5—C14	158.45 (10)
C3A—C4—C5—C14	−97.75 (13)
C14—C5—C6—C16	4.6 (2)
C4—C5—C6—C16	−162.42 (11)
C14—C5—C6—C6A	167.07 (12)
C4—C5—C6—C6A	0.07 (12)
C5—C6—C6A—O13	33.30 (11)
C16—C6—C6A—O13	−162.73 (10)
C5—C6—C6A—C7	153.40 (10)
C16—C6—C6A—C7	−42.62 (15)
C5—C6—C6A—C12C	−69.93 (11)
C16—C6—C6A—C12C	94.05 (12)
O13—C6A—C7—N8	−105.16 (11)
C12C—C6A—C7—N8	10.18 (15)
C6—C6A—C7—N8	140.52 (10)
C6A—C7—N8—C8A	−72.75 (13)
C6A—C7—N8—S1	98.88 (11)
O1—S1—N8—C8A	−170.14 (9)
O2—S1—N8—C8A	−39.32 (10)
C18—S1—N8—C8A	74.73 (10)
O1—S1—N8—C7	18.29 (11)
O2—S1—N8—C7	149.11 (9)
C18—S1—N8—C7	−96.85 (10)
C7—N8—C8A—C9	−107.68 (13)
S1—N8—C8A—C9	80.47 (13)
C7—N8—C8A—C12A	70.01 (15)
S1—N8—C8A—C12A	−101.83 (12)
C12A—C8A—C9—C10	−0.56 (19)
N8—C8A—C9—C10	177.14 (11)
C8A—C9—C10—C11	−0.2 (2)
C9—C10—C11—C12	0.9 (2)
C10—C11—C12—C12A	−0.7 (2)
C9—C8A—C12A—C12	0.70 (17)
N8—C8A—C12A—C12	−176.93 (10)
C9—C8A—C12A—C12B	179.17 (11)
N8—C8A—C12A—C12B	1.54 (17)
C11—C12—C12A—C8A	−0.05 (18)
C11—C12—C12A—C12B	−178.57 (11)
C2—C1—C12B—C12C	−0.87 (17)
C2—C1—C12B—C12A	177.47 (11)
C8A—C12A—C12B—C12C	−34.98 (17)
C3—C3A—C12C—C6A	−177.97 (11)
C4—C3A—C12C—C6A	−0.32 (11)
O13—C6A—C12C—C12B	149.70 (12)
C7—C6A—C12C—C12B	28.95 (18)
C6—C6A—C12C—C12B	−107.51 (14)
O13—C6A—C12C—C3A	−33.39 (11)
C7—C6A—C12C—C3A	−154.14 (10)
C6—C6A—C12C—C3A	69.41 (11)
C3A—C4—O13—C6A	−54.37 (10)
C5—C4—O13—C6A	53.28 (10)
C7—C6A—O13—C4	−179.14 (10)
C12C—C6A—O13—C4	53.93 (10)
C6—C6A—O13—C4	−53.03 (10)
C6—C5—C14—O14	36.1 (2)
C4—C5—C14—O14	−158.94 (13)
C6—C5—C14—O15	−144.93 (13)
C4—C5—C14—O15	20.01 (16)
O14—C14—O15—C15	−3.06 (18)
C5—C14—O15—C15	177.98 (10)
C5—C6—C16—O16	−44.8 (2)
C6A—C6—C16—O16	155.42 (12)
C5—C6—C16—O17	135.61 (13)
C6A—C6—C16—O17	−24.19 (14)
O16—C16—O17—C17	8.00 (17)
C6—C16—O17—C17	−172.38 (10)
O1—S1—C18—C19	152.89 (10)
O2—S1—C18—C19	20.83 (11)
N8—S1—C18—C19	−93.20 (11)
O1—S1—C18—C23	−30.97 (11)
O2—S1—C18—C23	−163.03 (10)
N8—S1—C18—C23	82.94 (11)
C23—C18—C19—C20	−2.14 (19)
S1—C18—C19—C20	173.88 (10)
C18—C19—C20—C21	0.4 (2)
C19—C20—C21—C22	1.6 (2)
C19—C20—C21—C24	−179.74 (12)
C20—C21—C22—C23	−1.9 (2)
C24—C21—C22—C23	179.43 (12)
C21—C22—C23—C18	0.18 (19)
C19—C18—C23—C22	1.85 (19)
S1—C18—C23—C22	−174.17 (9)
C22—C21—C24—F2	−158.19 (13)
C20—C21—C24—F2	23.13 (19)
C22—C21—C24—F3	−36.65 (18)
C20—C21—C24—F3	144.67 (14)
C22—C21—C24—F1	81.66 (17)
C20—C21—C24—F1	−97.02 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O16^{i}	1.00	2.49	3.3893 (16)	149
C7—H7*A*···O1	0.99	2.35	2.8481 (15)	111
C7—H7*B*···O17	0.99	2.46	3.0071 (14)	115
C12—H12···O2^{ii}	0.95	2.55	3.1357 (15)	120
C15—H15*A*···O13^{ii}	0.98	2.47	3.3741 (17)	153
C17—H17*B*···F1^{iii}	0.98	2.54	3.3033 (17)	135
C19—H19···O2	0.95	2.52	2.9003 (17)	104
C22—H22···O14^{i}	0.95	2.37	3.2698 (19)	158

Symmetry codes: (i) −*x*+1, *y*−1/2, −*z*+3/2; (ii) *x*+1, *y*, *z*; (iii) *x*+1, *y*+1, *z*.