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# 2-[(*E*)-2-(4-Ethoxyphenyl)ethenyl]-1-methylquinolinium 4-fluorobenzenesulfonate

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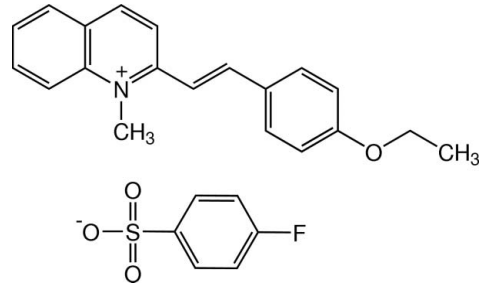
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.004 Å; disorder in main residue; $R$ factor = 0.065; $wR$ factor = 0.154; data-to-parameter ratio = 12.7.

In the structure of the title salt, $C_{20}H_{20}NO^{+}\cdot C_6H_4FO_3S^{-}$, the 4-(ethoxyphenyl)ethenyl unit is disordered over two positions with a refined site-occupancy ratio of 0.610 (6):0.390 (6). The cation is nearly planar, the dihedral angle between the quinolinium and benzene rings being 6.7 (4) and 1.7 (7)° for the major and minor components, respectively. The ethoxy group is essentially coplanar with the benzene ring [C—O—C—$C_{methy}$ = 177.1 (8) and 177.8 (12)° for the major and minor components, respectively]. In the crystal, cations and anions are linked into chains along the *b*-axis direction by C—H···$O_{sulfonyl}$ weak interactions. These chains are further connected into sheets parallel to (001) by C—H···$O_{sulfonyl}$ weak interactions. The chains are also stacked along the *a* axis through $\pi$–$\pi$ interactions involving the quinolinium and benzene rings [centroid–centroid distances = 3.636 (5) Å for the major component and 3.800 (9) Å for the minor component]. C—H···$\pi$ interactions are also present.

## Related literature

For background to the bioactivity and non-linear optical properties of quinolinium derivatives, see: Chanawanno *et al.* (2010); Hopkins *et al.* (2005); Musiol *et al.* (2006); O'Donnell *et al.* (2010); Ruanwas *et al.* (2010). For related structures, see: Chantrapromma *et al.* (2011); Fun *et al.* (2010); Ruanwas *et al.* (2010). For bond-length data, see: Allen *et al.* (1987). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986).



## Experimental

### Crystal data

$C_{20}H_{20}NO^{+}\cdot C_6H_4FO_3S^{-}$
$M_r$ = 465.52
Monoclinic, $P2_1/n$
$a$ = 6.4366 (3) Å
$b$ = 9.8909 (5) Å
$c$ = 34.3628 (15) Å
$\beta$ = 95.102 (2)°
$V$ = 2179.00 (18) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.19 mm$^{-1}$
$T$ = 100 K
0.37 × 0.12 × 0.05 mm

### Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min}$ = 0.932, $T_{max}$ = 0.991
19050 measured reflections
4993 independent reflections
3609 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.060

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.154
$S$ = 1.09
4993 reflections
392 parameters
418 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.49 e Å$^{-3}$

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

*Cg*4 and *Cg*5 are the centroids of the C12*B*–C17*B* and C21–C26 rings, respectively.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C2—H2*A*···O2$^{i}$ | 0.93 | 2.55 | 3.456 (4) | 166 |
| C8—H8*A*···O4$^{ii}$ | 0.93 | 2.41 | 3.306 (3) | 161 |
| C10—H10*A*···O3 | 0.96 | 2.55 | 3.483 (4) | 164 |
| C11*A*—H11*A*···O4$^{ii}$ | 0.93 | 2.52 | 3.408 (19) | 159 |
| C17*A*—H17*A*···O3 | 0.93 | 2.58 | 3.510 (10) | 177 |
| C20—H20*B*···O2$^{i}$ | 0.96 | 2.53 | 3.441 (4) | 158 |
| C20—H20*C*···O3 | 0.96 | 2.44 | 3.085 (4) | 124 |
| C25—H25*A*···O4$^{iii}$ | 0.93 | 2.55 | 3.264 (4) | 134 |
| C13*A*—H13*A*···*Cg*5$^{ii}$ | 0.93 | 2.82 | 3.575 (10) | 139 |
| C16*A*—H16*A*···*Cg*5 | 0.93 | 2.98 | 3.826 (9) | 151 |
| C19*A*—H19*B*···*Cg*4$^{iii}$ | 0.96 | 2.99 | 3.862 (11) | 152 |
| C13*B*—H13*B*···*Cg*5$^{ii}$ | 0.93 | 2.95 | 3.765 (16) | 147 |
| C16*B*—H16*B*···*Cg*5 | 0.93 | 2.70 | 3.562 (13) | 155 |

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PLATON* (Spek, 2009), *Mercury* (Macrae *et al.*, 2006) and *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ5097).

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# supporting information

*Acta Cryst.* (2014). E**70**, o11–o12 [https://doi.org/10.1107/S1600536813032509]

## 2-[(*E*)-2-(4-Ethoxyphenyl)ethenyl]-1-methylquinolinium 4-fluorobenzenesulfonate

**Hoong-Kun Fun, Thawanrat Kobkeatthawin, Pumsak Ruanwas, Ching Kheng Quah and Suchada Chantrapromma**

### S1. Comment

Quinolinium derivatives were reported to possess interesting bioactivities and pharmacological activities (Chanawanno *et al.*, 2010; Hopkins *et al.*, 2005; Musiol *et al.*, 2006; O′Donnell *et al.*, 2010), including non-linear optic properties (Ruanwas *et al.*, 2010). During the course of our research on the antibacterial activity of pyridinium and quinolinium salts, the title quinolinium salt (I) was synthesized in order to study the effect of the anion counter-part on its antibacterial activity because its starting quinolinium iodide salt (Chanawanno *et al.*, 2010) was found to be very active against the methicillin-resistant *Staphylococcus aureus* with a MIC value of 2.34 $\mu$g/ml. Herein the synthesis and crystal structure of (I) are reported.

In the title salt (Fig. 1), $C_{20}H_{20}NO^{+}.C_6H_4FSO_3^{-}$, the 4-(ethoxyphenyl)ethenyl unit is disordered over two positions with a refined site-occupancy ratio of 0.610 (6):0.390 (6). The cation exists in an *E* configuration with respect to the ethenyl bond [C10 ═C11 = 1.326 (18) Å for the major *A* component and 1.38 (3) Å for the minor *B* component] and torsion angle C9—C10—C11—C12 = -178.3 (12) ° for the major *A* component, and -179.0 (19)° for the minor *B* component. The 1-methylquinolinium ring system is planar with a *rms* deviation of 0.0199 (3) Å for the eleven non-H atoms. The cation is planar with dihedral angles between the N1/C1–C9 quinolinium and C12–C17 benzene rings of 6.7 (4) and 1.7 (7)° for the major *A* and minor *B* components, respectively. The ethoxy unit is disordered over two positions in such a way that the major *A* and minor *B* components are related by a 180° rotation. Moreover the ethoxy unit is co-planar with the attached benzene ring as indicated by the torsion angles C16–C15–O1–C18 = 2.5 (15)° and C15–O1–C18–C19 = 177.1 (8)° for the major *A* component. The corresponding values are 180.0 (14) and 177.8 (12)° for the minor *B* component. Bond distances in both cation and anion have normal values (Allen *et al.*, 1987) and are comparable to those observed in related structures (Chantrapromma *et al.*, 2011; Fun *et al.*, 2010; Ruanwas *et al.*, 2010).

In the crystal packing (Fig. 2), cations and anions are linked into chains along the *b* axis by C—H···$O_{sulfonyl}$ weak interactions. These chains are further connected into sheets parallel to the (001) plane by C—H···$O_{sulfonyl}$ weak interactions (Table 1), and these chains are also stacked by $\pi$–$\pi$ interactions involving quinolinium and benzene rings (Fig. 3) with separations $Cg_1\cdots Cg_3^{i}$ = 3.636 (5) Å in the major component *A* and $Cg_1\cdots Cg_4^{i}$ = 3.800 (9) Å in the minor component *B* (symmetry code as in Table 1); $Cg_1$, $Cg_3$ and $Cg_4$ are the centroids of the N1/C1/C6–C9, C12A–C17A and C12B–C17B rings, respectively. C—H···$\pi$ interactions (Table 1) are also present.

### S2. Experimental

The title compound was synthesized by dissolving silver(I) 4-fluorobenzenesulfonate (0.20 g, 0.71 mmol) in methanol (20 ml) which upon heating was added to a solution of 2-[(*E*)-2-(4-ethoxyphenyl)ethenyl]-1-methylquinolinium iodide

(Fun *et al.*, 2010) (0.29 g, 0.71 mmol) in hot methanol (30 ml). The mixture turned yellow and cloudy immediately. After stirring for 0.5 h, the precipitate of silver iodide which formed was filtered and the filtrate was evaporated to give a yellow solid. Yellow plate-shaped single crystals of the title compound suitable for X-ray structure determination were recrystallized from methanol by slow evaporation of the solvent at room temperature after a few weeks.

## S3. Refinement

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with d(C-H) = 0.93 Å for aromatic and CH, 0.97 Å for $CH_2$ and 0.96 Å for $CH_3$ atoms. The $U_{iso}$ values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups. The 4-(ethoxyphenyl)-ethenyl unit is disordered over two sites with refined site occupancies ratio 0.610 (6):0.390 (6). Similarity and simulation restraints were applied.

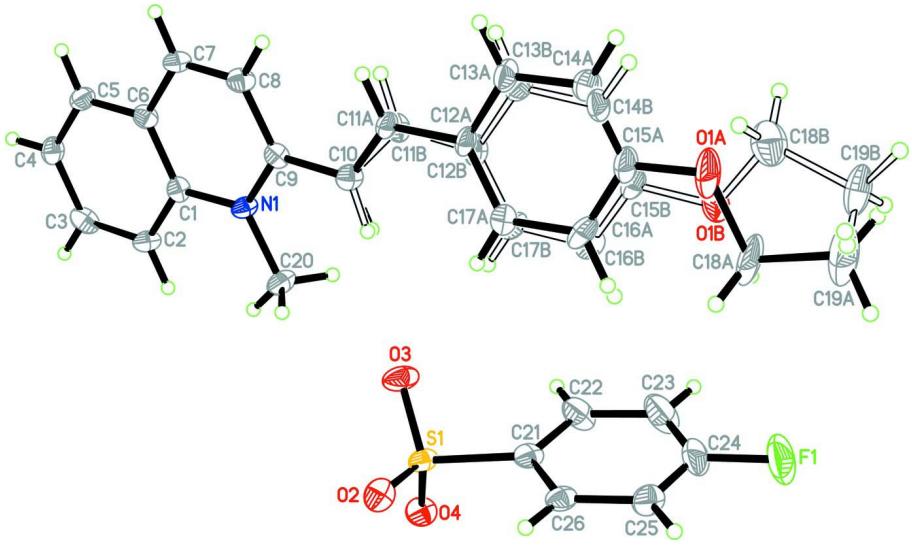


**Figure 1**

The structure of the title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme. Open bonds show the minor component.

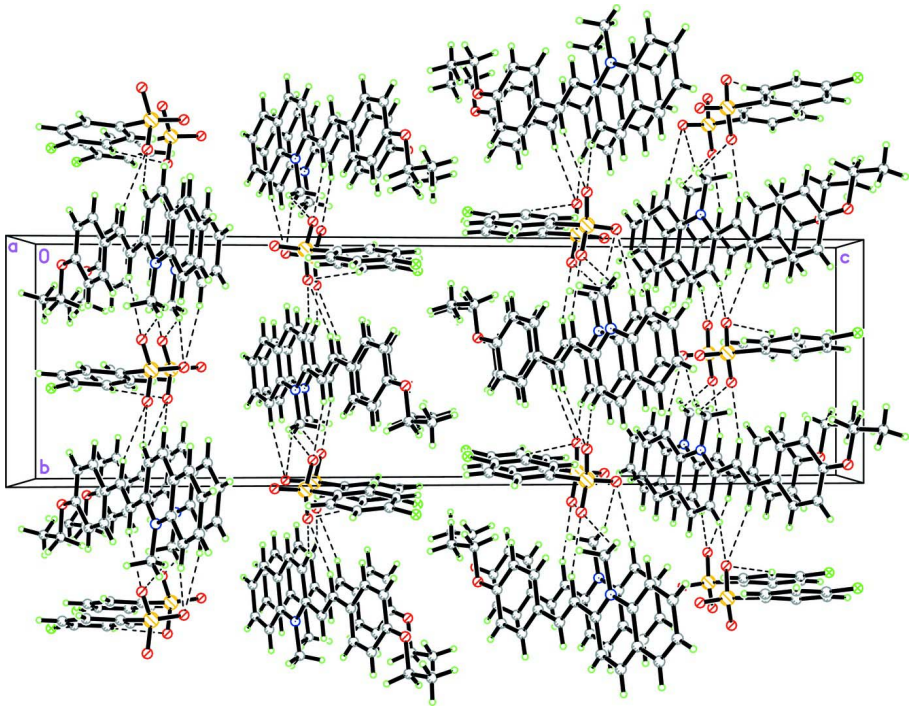


**Figure 2**

The crystal packing of the major component of the title compound viewed approximately along the *a* axis. Hydrogen bonds are drawn as dashed lines.

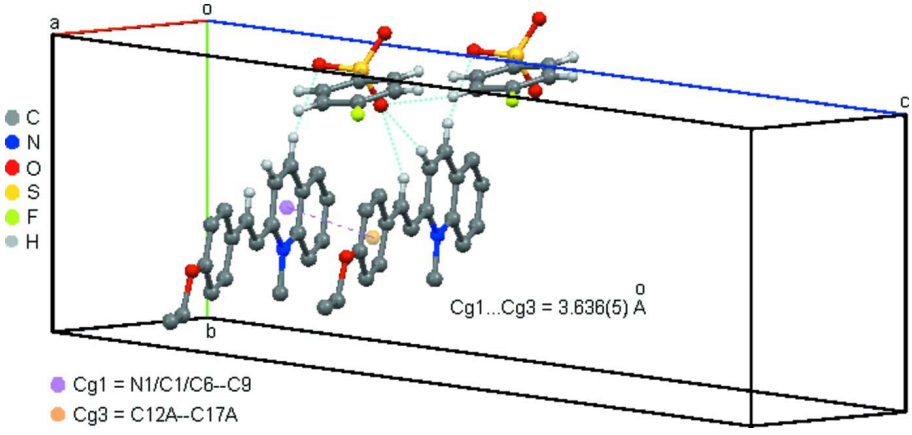


**Figure 3**

$\pi$–$\pi$ interaction between aromatic rings of the cations of the major component. H-atoms of the cations not involved in hydrogen bonds are omitted for clarity.

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*Crystal data*

$C_{20}H_{20}NO^{+}\cdot C_6H_4FO_3S^{-}$
$M_r$ = 465.52
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a$ = 6.4366 (3) Å
$b$ = 9.8909 (5) Å
$c$ = 34.3628 (15) Å
$\beta$ = 95.102 (2)°
$V$ = 2179.00 (18) Å$^3$
$Z$ = 4

$F(000) = 976$
$D_x = 1.419$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4993 reflections
$\theta = 2.1–27.5°$
$\mu = 0.19$ mm$^{-1}$
$T = 100$ K
Plate, yellow
0.37 × 0.12 × 0.05 mm

*Data collection*

Bruker APEXII CCD area-detector diffractometer
Radiation source: sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min} = 0.932$, $T_{max} = 0.991$
19050 measured reflections
4993 independent reflections
3609 reflections with $I > 2\sigma(I)$
$R_{int} = 0.060$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.1°$
$h = -8 \rightarrow 8$
$k = -12 \rightarrow 11$
$l = -44 \rightarrow 44$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.154$
$S = 1.09$
4993 reflections
392 parameters
418 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0388P)^2 + 4.3613P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.39$ e Å$^{-3}$
$\Delta\rho_{min} = -0.49$ e Å$^{-3}$

*Special details*

**Experimental**. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

**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 $F^2 > 2$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) |
|---|---|---|---|---|---|
| N1 | 1.5593 (3) | 0.1235 (2) | 0.18311 (7) | 0.0173 (5) | |
| C1 | 1.7488 (4) | 0.0857 (3) | 0.20382 (8) | 0.0170 (6) | |
| C2 | 1.8922 (4) | 0.1828 (3) | 0.22024 (9) | 0.0211 (6) | |
| H2A | 1.8655 | 0.2748 | 0.2173 | 0.025* | |
| C3 | 2.0733 (4) | 0.1375 (3) | 0.24085 (9) | 0.0233 (6) | |
| H3A | 2.1660 | 0.2008 | 0.2524 | 0.028* | |
| C4 | 2.1223 (4) | 0.0006 (3) | 0.24491 (9) | 0.0214 (6) | |
| H4A | 2.2460 | −0.0267 | 0.2587 | 0.026* | |
| C5 | 1.9854 (4) | −0.0928 (3) | 0.22830 (9) | 0.0205 (6) | |
| H5A | 2.0182 | −0.1842 | 0.2303 | 0.025* | |

| | | | | | |
|---|---|---|---|---|---|
| C6 | 1.7957 (4) | −0.0528 (3) | 0.20815 (8) | 0.0184 (6) | |
| C7 | 1.6492 (4) | −0.1489 (3) | 0.19255 (9) | 0.0191 (6) | |
| H7A | 1.6790 | −0.2406 | 0.1952 | 0.023* | |
| C8 | 1.4663 (4) | −0.1090 (3) | 0.17380 (9) | 0.0198 (6) | |
| H8A | 1.3698 | −0.1738 | 0.1644 | 0.024* | |
| C9 | 1.4191 (4) | 0.0300 (3) | 0.16827 (8) | 0.0163 (5) | |
| C10 | 1.2247 (4) | 0.0745 (3) | 0.14715 (9) | 0.0197 (6) | |
| H10A | 1.1959 | 0.1693 | 0.1442 | 0.024* | 0.610 (6) |
| H10B | 1.1971 | 0.1699 | 0.1466 | 0.024* | 0.390 (6) |
| C20 | 1.5120 (5) | 0.2681 (3) | 0.17700 (10) | 0.0245 (7) | |
| H20A | 1.4912 | 0.2865 | 0.1495 | 0.037* | |
| H20B | 1.6262 | 0.3213 | 0.1884 | 0.037* | |
| H20C | 1.3876 | 0.2905 | 0.1891 | 0.037* | |
| O1A | 0.3281 (9) | 0.0972 (6) | 0.04235 (19) | 0.0386 (14) | 0.610 (6) |
| C11A | 1.086 (2) | −0.0139 (19) | 0.1318 (6) | 0.0169 (19) | 0.610 (6) |
| H11A | 1.1156 | −0.1053 | 0.1356 | 0.020* | 0.610 (6) |
| C12A | 0.889 (2) | 0.0214 (10) | 0.1092 (4) | 0.0174 (16) | 0.610 (6) |
| C13A | 0.7675 (19) | −0.0815 (10) | 0.0915 (5) | 0.0235 (16) | 0.610 (6) |
| H13A | 0.8105 | −0.1708 | 0.0950 | 0.028* | 0.610 (6) |
| C14A | 0.5834 (16) | −0.0542 (8) | 0.0687 (4) | 0.0250 (16) | 0.610 (6) |
| H14A | 0.5068 | −0.1244 | 0.0565 | 0.030* | 0.610 (6) |
| C15A | 0.5146 (15) | 0.0770 (8) | 0.0641 (4) | 0.0246 (16) | 0.610 (6) |
| C16A | 0.6277 (15) | 0.1820 (9) | 0.0829 (3) | 0.0246 (18) | 0.610 (6) |
| H16A | 0.5789 | 0.2703 | 0.0805 | 0.030* | 0.610 (6) |
| C17A | 0.8141 (15) | 0.1543 (10) | 0.1054 (3) | 0.0190 (18) | 0.610 (6) |
| H17A | 0.8891 | 0.2246 | 0.1179 | 0.023* | 0.610 (6) |
| C18A | 0.2520 (9) | 0.2376 (8) | 0.03791 (18) | 0.0459 (17) | 0.610 (6) |
| H18A | 0.3556 | 0.2933 | 0.0267 | 0.055* | 0.610 (6) |
| H18B | 0.2271 | 0.2745 | 0.0632 | 0.055* | 0.610 (6) |
| C19A | 0.0542 (11) | 0.2371 (11) | 0.0117 (2) | 0.061 (2) | 0.610 (6) |
| H19A | −0.0053 | 0.3262 | 0.0108 | 0.092* | 0.610 (6) |
| H19B | −0.0423 | 0.1745 | 0.0216 | 0.092* | 0.610 (6) |
| H19C | 0.0832 | 0.2104 | −0.0141 | 0.092* | 0.610 (6) |
| O1B | 0.3343 (14) | 0.1530 (7) | 0.0407 (3) | 0.0238 (16) | 0.390 (6) |
| C11B | 1.076 (4) | −0.005 (3) | 0.1270 (9) | 0.019 (3) | 0.390 (6) |
| H11B | 1.1013 | −0.0980 | 0.1272 | 0.023* | 0.390 (6) |
| C12B | 0.883 (3) | 0.0408 (17) | 0.1054 (8) | 0.019 (2) | 0.390 (6) |
| C13B | 0.748 (3) | −0.0595 (16) | 0.0880 (7) | 0.022 (2) | 0.390 (6) |
| H13B | 0.7842 | −0.1504 | 0.0903 | 0.026* | 0.390 (6) |
| C14B | 0.561 (3) | −0.0215 (13) | 0.0672 (6) | 0.022 (2) | 0.390 (6) |
| H14B | 0.4692 | −0.0872 | 0.0564 | 0.027* | 0.390 (6) |
| C15B | 0.512 (2) | 0.1117 (12) | 0.0627 (6) | 0.0188 (19) | 0.390 (6) |
| C16B | 0.647 (2) | 0.2108 (13) | 0.0783 (5) | 0.020 (2) | 0.390 (6) |
| H16B | 0.6125 | 0.3016 | 0.0747 | 0.024* | 0.390 (6) |
| C17B | 0.831 (2) | 0.1749 (15) | 0.0993 (5) | 0.017 (2) | 0.390 (6) |
| H17B | 0.9209 | 0.2421 | 0.1094 | 0.020* | 0.390 (6) |
| C18B | 0.1886 (13) | 0.0568 (9) | 0.0237 (3) | 0.038 (2) | 0.390 (6) |
| H18C | 0.1379 | −0.0008 | 0.0436 | 0.046* | 0.390 (6) |

| | | | | | |
|---|---|---|---|---|---|
| H18D | 0.2530 | 0.0005 | 0.0050 | 0.046* | 0.390 (6) |
| C19B | 0.0125 (13) | 0.1380 (12) | 0.0035 (3) | 0.038 (2) | 0.390 (6) |
| H19D | −0.0927 | 0.0778 | −0.0080 | 0.057* | 0.390 (6) |
| H19E | 0.0647 | 0.1929 | −0.0165 | 0.057* | 0.390 (6) |
| H19F | −0.0468 | 0.1950 | 0.0223 | 0.057* | 0.390 (6) |
| S1 | 0.97568 (10) | 0.53446 (7) | 0.16403 (2) | 0.02062 (19) | |
| F1 | 0.3508 (3) | 0.6116 (3) | 0.03020 (7) | 0.0605 (7) | |
| O2 | 0.8648 (3) | 0.5242 (2) | 0.19899 (6) | 0.0262 (5) | |
| O3 | 1.0931 (3) | 0.4139 (2) | 0.15628 (7) | 0.0337 (6) | |
| O4 | 1.0969 (3) | 0.6584 (2) | 0.16254 (6) | 0.0261 (5) | |
| C21 | 0.7812 (4) | 0.5484 (3) | 0.12372 (9) | 0.0223 (6) | |
| C22 | 0.8393 (5) | 0.5361 (4) | 0.08595 (10) | 0.0331 (8) | |
| H22A | 0.9763 | 0.5141 | 0.0820 | 0.040* | |
| C23 | 0.6945 (5) | 0.5564 (4) | 0.05408 (11) | 0.0436 (10) | |
| H23A | 0.7320 | 0.5488 | 0.0287 | 0.052* | |
| C24 | 0.4930 (5) | 0.5883 (4) | 0.06144 (11) | 0.0378 (9) | |
| C25 | 0.4299 (5) | 0.5984 (3) | 0.09833 (10) | 0.0286 (7) | |
| H25A | 0.2922 | 0.6191 | 0.1021 | 0.034* | |
| C26 | 0.5755 (4) | 0.5771 (3) | 0.12989 (9) | 0.0212 (6) | |
| H26A | 0.5355 | 0.5819 | 0.1552 | 0.025* | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0140 (11) | 0.0130 (11) | 0.0247 (13) | −0.0001 (8) | 0.0009 (9) | −0.0002 (10) |
| C1 | 0.0109 (12) | 0.0221 (13) | 0.0181 (15) | 0.0002 (10) | 0.0016 (10) | −0.0002 (11) |
| C2 | 0.0206 (14) | 0.0151 (13) | 0.0278 (17) | 0.0008 (11) | 0.0031 (12) | −0.0024 (12) |
| C3 | 0.0174 (14) | 0.0256 (15) | 0.0268 (17) | −0.0101 (12) | 0.0008 (12) | −0.0069 (13) |
| C4 | 0.0159 (13) | 0.0268 (16) | 0.0209 (16) | 0.0014 (11) | −0.0011 (11) | 0.0017 (12) |
| C5 | 0.0213 (14) | 0.0170 (13) | 0.0231 (16) | 0.0033 (11) | 0.0013 (12) | 0.0011 (12) |
| C6 | 0.0154 (13) | 0.0196 (14) | 0.0201 (15) | −0.0023 (11) | 0.0006 (11) | −0.0027 (12) |
| C7 | 0.0193 (14) | 0.0132 (13) | 0.0250 (16) | −0.0004 (10) | 0.0031 (11) | −0.0014 (11) |
| C8 | 0.0167 (13) | 0.0167 (13) | 0.0261 (17) | −0.0056 (11) | 0.0019 (12) | −0.0019 (12) |
| C9 | 0.0100 (12) | 0.0209 (13) | 0.0184 (14) | −0.0014 (11) | 0.0026 (10) | −0.0023 (12) |
| C10 | 0.0144 (13) | 0.0206 (14) | 0.0242 (16) | 0.0010 (11) | 0.0020 (11) | −0.0012 (12) |
| C20 | 0.0198 (14) | 0.0152 (13) | 0.0371 (19) | 0.0009 (11) | −0.0052 (13) | 0.0016 (13) |
| O1A | 0.019 (2) | 0.065 (3) | 0.030 (2) | 0.010 (3) | −0.0085 (17) | 0.005 (3) |
| C11A | 0.013 (3) | 0.019 (3) | 0.018 (5) | 0.005 (2) | −0.001 (3) | 0.003 (3) |
| C12A | 0.013 (2) | 0.024 (3) | 0.015 (3) | 0.006 (2) | 0.000 (2) | 0.004 (3) |
| C13A | 0.018 (3) | 0.026 (3) | 0.026 (3) | 0.003 (2) | −0.006 (2) | 0.005 (3) |
| C14A | 0.019 (3) | 0.027 (3) | 0.029 (3) | 0.004 (3) | −0.003 (2) | −0.002 (3) |
| C15A | 0.016 (2) | 0.035 (4) | 0.022 (2) | 0.006 (3) | −0.003 (2) | 0.003 (3) |
| C16A | 0.019 (3) | 0.026 (4) | 0.030 (3) | 0.010 (3) | 0.005 (2) | 0.008 (3) |
| C17A | 0.014 (3) | 0.025 (4) | 0.018 (3) | 0.000 (2) | 0.004 (2) | 0.003 (3) |
| C18A | 0.029 (3) | 0.077 (4) | 0.030 (3) | 0.035 (3) | −0.002 (2) | 0.009 (3) |
| C19A | 0.037 (3) | 0.110 (6) | 0.036 (4) | 0.033 (4) | −0.006 (3) | 0.005 (4) |
| O1B | 0.021 (3) | 0.023 (3) | 0.026 (3) | −0.008 (3) | −0.006 (2) | 0.003 (3) |
| C11B | 0.018 (4) | 0.022 (5) | 0.017 (5) | −0.004 (4) | 0.000 (4) | 0.002 (4) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C12B | 0.011 (3) | 0.027 (4) | 0.019 (4) | 0.000 (3) | 0.001 (3) | 0.003 (3) |
| C13B | 0.021 (4) | 0.018 (4) | 0.025 (4) | 0.003 (3) | −0.001 (3) | 0.003 (4) |
| C14B | 0.019 (4) | 0.025 (4) | 0.021 (3) | −0.002 (4) | −0.008 (3) | 0.001 (4) |
| C15B | 0.015 (3) | 0.022 (4) | 0.019 (3) | 0.006 (3) | 0.002 (3) | 0.004 (3) |
| C16B | 0.015 (3) | 0.019 (4) | 0.026 (4) | −0.001 (3) | 0.005 (3) | 0.005 (3) |
| C17B | 0.013 (3) | 0.015 (4) | 0.023 (4) | −0.001 (3) | 0.004 (3) | 0.008 (3) |
| C18B | 0.031 (4) | 0.046 (4) | 0.036 (4) | −0.007 (3) | −0.002 (3) | 0.002 (4) |
| C19B | 0.017 (4) | 0.057 (5) | 0.039 (5) | 0.006 (4) | −0.007 (3) | 0.013 (4) |
| S1 | 0.0126 (3) | 0.0153 (3) | 0.0329 (4) | 0.0008 (3) | −0.0040 (3) | −0.0043 (3) |
| F1 | 0.0400 (13) | 0.096 (2) | 0.0406 (14) | 0.0180 (13) | −0.0222 (10) | −0.0190 (13) |
| O2 | 0.0217 (10) | 0.0269 (11) | 0.0298 (13) | −0.0014 (9) | 0.0015 (9) | 0.0035 (10) |
| O3 | 0.0195 (11) | 0.0232 (11) | 0.0559 (16) | 0.0070 (9) | −0.0105 (10) | −0.0129 (11) |
| O4 | 0.0193 (10) | 0.0225 (11) | 0.0355 (13) | −0.0056 (8) | −0.0031 (9) | −0.0040 (9) |
| C21 | 0.0145 (13) | 0.0160 (13) | 0.0353 (18) | −0.0031 (11) | −0.0050 (12) | −0.0068 (13) |
| C22 | 0.0163 (14) | 0.049 (2) | 0.0331 (19) | 0.0027 (14) | −0.0005 (13) | −0.0184 (17) |
| C23 | 0.0313 (18) | 0.067 (3) | 0.032 (2) | 0.0029 (18) | −0.0008 (15) | −0.0210 (19) |
| C24 | 0.0289 (17) | 0.048 (2) | 0.033 (2) | 0.0046 (16) | −0.0146 (15) | −0.0118 (17) |
| C25 | 0.0139 (14) | 0.0277 (16) | 0.043 (2) | 0.0015 (12) | −0.0054 (13) | −0.0066 (15) |
| C26 | 0.0171 (13) | 0.0151 (13) | 0.0311 (17) | −0.0014 (11) | 0.0011 (12) | −0.0029 (12) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N1—C9 | 1.359 (3) | C18A—H18A | 0.9700 |
| N1—C1 | 1.407 (3) | C18A—H18B | 0.9700 |
| N1—C20 | 1.473 (3) | C19A—H19A | 0.9600 |
| C1—C6 | 1.407 (4) | C19A—H19B | 0.9600 |
| C1—C2 | 1.414 (4) | C19A—H19C | 0.9600 |
| C2—C3 | 1.383 (4) | O1B—C15B | 1.375 (11) |
| C2—H2A | 0.9300 | O1B—C18B | 1.425 (10) |
| C3—C4 | 1.394 (4) | C11B—C12B | 1.463 (12) |
| C3—H3A | 0.9300 | C11B—H11B | 0.9300 |
| C4—C5 | 1.366 (4) | C12B—C17B | 1.380 (12) |
| C4—H4A | 0.9300 | C12B—C13B | 1.417 (12) |
| C5—C6 | 1.406 (4) | C13B—C14B | 1.399 (12) |
| C5—H5A | 0.9300 | C13B—H13B | 0.9300 |
| C6—C7 | 1.411 (4) | C14B—C15B | 1.360 (11) |
| C7—C8 | 1.350 (4) | C14B—H14B | 0.9300 |
| C7—H7A | 0.9300 | C15B—C16B | 1.385 (11) |
| C8—C9 | 1.418 (4) | C16B—C17B | 1.377 (11) |
| C8—H8A | 0.9300 | C16B—H16B | 0.9300 |
| C9—C10 | 1.457 (4) | C17B—H17B | 0.9300 |
| C10—C11A | 1.326 (18) | C18B—C19B | 1.506 (10) |
| C10—C11B | 1.38 (3) | C18B—H18C | 0.9700 |
| C10—H10A | 0.9600 | C18B—H18D | 0.9700 |
| C10—H10B | 0.9600 | C19B—H19D | 0.9600 |
| C20—H20A | 0.9600 | C19B—H19E | 0.9600 |
| C20—H20B | 0.9600 | C19B—H19F | 0.9600 |
| C20—H20C | 0.9600 | S1—O3 | 1.449 (2) |

| | | | |
|---|---|---|---|
| O1A—C15A | 1.371 (7) | S1—O2 | 1.454 (2) |
| O1A—C18A | 1.475 (8) | S1—O4 | 1.457 (2) |
| C11A—C12A | 1.467 (8) | S1—C21 | 1.788 (3) |
| C11A—H11A | 0.9300 | F1—C24 | 1.367 (4) |
| C12A—C13A | 1.392 (8) | C21—C22 | 1.388 (5) |
| C12A—C17A | 1.403 (8) | C21—C26 | 1.389 (4) |
| C13A—C14A | 1.388 (8) | C22—C23 | 1.388 (5) |
| C13A—H13A | 0.9300 | C22—H22A | 0.9300 |
| C14A—C15A | 1.375 (8) | C23—C24 | 1.380 (5) |
| C14A—H14A | 0.9300 | C23—H23A | 0.9300 |
| C15A—C16A | 1.394 (9) | C24—C25 | 1.369 (5) |
| C16A—C17A | 1.394 (8) | C25—C26 | 1.385 (4) |
| C16A—H16A | 0.9300 | C25—H25A | 0.9300 |
| C17A—H17A | 0.9300 | C26—H26A | 0.9300 |
| C18A—C19A | 1.492 (8) | | |

| | | | |
|---|---|---|---|
| C9—N1—C1 | 121.7 (2) | C12A—C17A—H17A | 119.7 |
| C9—N1—C20 | 119.0 (2) | O1A—C18A—C19A | 108.5 (6) |
| C1—N1—C20 | 119.3 (2) | O1A—C18A—H18A | 110.0 |
| C6—C1—N1 | 118.7 (2) | C19A—C18A—H18A | 110.0 |
| C6—C1—C2 | 119.5 (2) | O1A—C18A—H18B | 110.0 |
| N1—C1—C2 | 121.8 (2) | C19A—C18A—H18B | 110.0 |
| C3—C2—C1 | 118.3 (3) | H18A—C18A—H18B | 108.4 |
| C3—C2—H2A | 120.8 | C15B—O1B—C18B | 120.8 (8) |
| C1—C2—H2A | 120.8 | C10—C11B—C12B | 127 (2) |
| C2—C3—C4 | 122.6 (3) | C10—C11B—H11B | 116.7 |
| C2—C3—H3A | 118.7 | C12B—C11B—H11B | 116.7 |
| C4—C3—H3A | 118.7 | C17B—C12B—C13B | 118.3 (11) |
| C5—C4—C3 | 118.9 (3) | C17B—C12B—C11B | 124.3 (14) |
| C5—C4—H4A | 120.6 | C13B—C12B—C11B | 117.2 (13) |
| C3—C4—H4A | 120.6 | C14B—C13B—C12B | 119.8 (11) |
| C4—C5—C6 | 121.0 (3) | C14B—C13B—H13B | 120.1 |
| C4—C5—H5A | 119.5 | C12B—C13B—H13B | 120.1 |
| C6—C5—H5A | 119.5 | C15B—C14B—C13B | 119.8 (11) |
| C5—C6—C1 | 119.6 (2) | C15B—C14B—H14B | 120.1 |
| C5—C6—C7 | 121.3 (3) | C13B—C14B—H14B | 120.1 |
| C1—C6—C7 | 119.1 (2) | C14B—C15B—O1B | 121.5 (10) |
| C8—C7—C6 | 120.6 (3) | C14B—C15B—C16B | 120.8 (10) |
| C8—C7—H7A | 119.7 | O1B—C15B—C16B | 117.6 (9) |
| C6—C7—H7A | 119.7 | C17B—C16B—C15B | 120.0 (10) |
| C7—C8—C9 | 121.0 (2) | C17B—C16B—H16B | 120.0 |
| C7—C8—H8A | 119.5 | C15B—C16B—H16B | 120.0 |
| C9—C8—H8A | 119.5 | C16B—C17B—C12B | 121.1 (11) |
| N1—C9—C8 | 118.8 (2) | C16B—C17B—H17B | 119.5 |
| N1—C9—C10 | 119.6 (2) | C12B—C17B—H17B | 119.5 |
| C8—C9—C10 | 121.6 (2) | O1B—C18B—C19B | 105.9 (8) |
| C11A—C10—C9 | 121.2 (6) | O1B—C18B—H18C | 110.6 |
| C11B—C10—C9 | 127.1 (10) | C19B—C18B—H18C | 110.6 |

| | | | |
|---|---|---|---|
| C11A—C10—H10A | 119.1 | O1B—C18B—H18D | 110.6 |
| C11B—C10—H10A | 112.9 | C19B—C18B—H18D | 110.6 |
| C9—C10—H10A | 119.8 | H18C—C18B—H18D | 108.7 |
| C11A—C10—H10B | 121.6 | C18B—C19B—H19D | 109.5 |
| C11B—C10—H10B | 115.8 | C18B—C19B—H19E | 109.5 |
| C9—C10—H10B | 117.1 | H19D—C19B—H19E | 109.5 |
| N1—C20—H20A | 109.5 | C18B—C19B—H19F | 109.5 |
| N1—C20—H20B | 109.5 | H19D—C19B—H19F | 109.5 |
| H20A—C20—H20B | 109.5 | H19E—C19B—H19F | 109.5 |
| N1—C20—H20C | 109.5 | O3—S1—O2 | 113.38 (14) |
| H20A—C20—H20C | 109.5 | O3—S1—O4 | 113.36 (13) |
| H20B—C20—H20C | 109.5 | O2—S1—O4 | 113.04 (13) |
| C15A—O1A—C18A | 117.4 (6) | O3—S1—C21 | 105.18 (13) |
| C10—C11A—C12A | 124.9 (12) | O2—S1—C21 | 106.51 (13) |
| C10—C11A—H11A | 117.5 | O4—S1—C21 | 104.35 (13) |
| C12A—C11A—H11A | 117.5 | C22—C21—C26 | 120.1 (3) |
| C13A—C12A—C17A | 117.9 (7) | C22—C21—S1 | 119.3 (2) |
| C13A—C12A—C11A | 118.9 (8) | C26—C21—S1 | 120.6 (2) |
| C17A—C12A—C11A | 123.2 (9) | C21—C22—C23 | 120.5 (3) |
| C14A—C13A—C12A | 121.6 (7) | C21—C22—H22A | 119.8 |
| C14A—C13A—H13A | 119.2 | C23—C22—H22A | 119.8 |
| C12A—C13A—H13A | 119.2 | C24—C23—C22 | 117.7 (3) |
| C15A—C14A—C13A | 119.9 (7) | C24—C23—H23A | 121.2 |
| C15A—C14A—H14A | 120.0 | C22—C23—H23A | 121.2 |
| C13A—C14A—H14A | 120.0 | F1—C24—C25 | 118.8 (3) |
| O1A—C15A—C14A | 117.2 (7) | F1—C24—C23 | 118.0 (3) |
| O1A—C15A—C16A | 122.7 (7) | C25—C24—C23 | 123.2 (3) |
| C14A—C15A—C16A | 120.0 (7) | C24—C25—C26 | 118.5 (3) |
| C15A—C16A—C17A | 119.9 (7) | C24—C25—H25A | 120.7 |
| C15A—C16A—H16A | 120.0 | C26—C25—H25A | 120.7 |
| C17A—C16A—H16A | 120.0 | C25—C26—C21 | 120.0 (3) |
| C16A—C17A—C12A | 120.5 (7) | C25—C26—H26A | 120.0 |
| C16A—C17A—H17A | 119.7 | C21—C26—H26A | 120.0 |

| | | | |
|---|---|---|---|
| C9—N1—C1—C6 | 1.9 (4) | O1A—C15A—C16A—C17A | 178.5 (10) |
| C20—N1—C1—C6 | −177.6 (3) | C14A—C15A—C16A—C17A | 2.1 (16) |
| C9—N1—C1—C2 | −178.2 (3) | C15A—C16A—C17A—C12A | 0.1 (15) |
| C20—N1—C1—C2 | 2.4 (4) | C13A—C12A—C17A—C16A | −3.0 (17) |
| C6—C1—C2—C3 | −1.3 (4) | C11A—C12A—C17A—C16A | 178.8 (13) |
| N1—C1—C2—C3 | 178.7 (3) | C15A—O1A—C18A—C19A | 177.1 (8) |
| C1—C2—C3—C4 | 2.1 (5) | C11A—C10—C11B—C12B | 140 (19) |
| C2—C3—C4—C5 | −0.7 (5) | C9—C10—C11B—C12B | −179.0 (19) |
| C3—C4—C5—C6 | −1.5 (5) | C10—C11B—C12B—C17B | 6 (4) |
| C4—C5—C6—C1 | 2.2 (4) | C10—C11B—C12B—C13B | −178 (3) |
| C4—C5—C6—C7 | −176.9 (3) | C17B—C12B—C13B—C14B | −4 (3) |
| N1—C1—C6—C5 | 179.2 (3) | C11B—C12B—C13B—C14B | 180 (2) |
| C2—C1—C6—C5 | −0.8 (4) | C12B—C13B—C14B—C15B | 2 (3) |
| N1—C1—C6—C7 | −1.7 (4) | C13B—C14B—C15B—O1B | 176.6 (19) |

| | | | |
|---|---|---|---|
| C2—C1—C6—C7 | 178.4 (3) | C13B—C14B—C15B—C16B | 0 (3) |
| C5—C6—C7—C8 | 179.0 (3) | C18B—O1B—C15B—C14B | 3 (2) |
| C1—C6—C7—C8 | −0.2 (4) | C18B—O1B—C15B—C16B | 180.0 (14) |
| C6—C7—C8—C9 | 1.9 (5) | C14B—C15B—C16B—C17B | −1 (3) |
| C1—N1—C9—C8 | −0.2 (4) | O1B—C15B—C16B—C17B | −177.5 (16) |
| C20—N1—C9—C8 | 179.3 (3) | C15B—C16B—C17B—C12B | −1 (3) |
| C1—N1—C9—C10 | 179.8 (3) | C13B—C12B—C17B—C16B | 3 (3) |
| C20—N1—C9—C10 | −0.7 (4) | C11B—C12B—C17B—C16B | 180 (2) |
| C7—C8—C9—N1 | −1.7 (4) | C15B—O1B—C18B—C19B | 177.8 (12) |
| C7—C8—C9—C10 | 178.3 (3) | O3—S1—C21—C22 | −48.2 (3) |
| N1—C9—C10—C11A | 178.9 (12) | O2—S1—C21—C22 | −168.8 (2) |
| C8—C9—C10—C11A | −1.0 (12) | O4—S1—C21—C22 | 71.3 (3) |
| N1—C9—C10—C11B | 173 (2) | O3—S1—C21—C26 | 134.5 (2) |
| C8—C9—C10—C11B | −7 (2) | O2—S1—C21—C26 | 13.9 (3) |
| C11B—C10—C11A—C12A | −36 (15) | O4—S1—C21—C26 | −105.9 (2) |
| C9—C10—C11A—C12A | −178.3 (12) | C26—C21—C22—C23 | 2.0 (5) |
| C10—C11A—C12A—C13A | 173.8 (18) | S1—C21—C22—C23 | −175.2 (3) |
| C10—C11A—C12A—C17A | −8 (2) | C21—C22—C23—C24 | −0.3 (6) |
| C17A—C12A—C13A—C14A | 4 (2) | C22—C23—C24—F1 | 178.5 (3) |
| C11A—C12A—C13A—C14A | −177.8 (15) | C22—C23—C24—C25 | −1.1 (6) |
| C12A—C13A—C14A—C15A | −2 (2) | F1—C24—C25—C26 | −178.9 (3) |
| C18A—O1A—C15A—C14A | 179.0 (10) | C23—C24—C25—C26 | 0.7 (6) |
| C18A—O1A—C15A—C16A | 2.5 (15) | C24—C25—C26—C21 | 1.1 (4) |
| C13A—C14A—C15A—O1A | −177.8 (12) | C22—C21—C26—C25 | −2.4 (4) |
| C13A—C14A—C15A—C16A | −1.2 (18) | S1—C21—C26—C25 | 174.8 (2) |

*Hydrogen-bond geometry (Å, º)*

*Cg*4 and *Cg*5 are the centroids of the C12B–C17B and C21–C26 rings, respectively.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C2—H2*A*···O2$^{i}$ | 0.93 | 2.55 | 3.456 (4) | 166 |
| C8—H8*A*···O4$^{ii}$ | 0.93 | 2.41 | 3.306 (3) | 161 |
| C10—H10*A*···O3 | 0.96 | 2.55 | 3.483 (4) | 164 |
| C11*A*—H11*A*···O4$^{ii}$ | 0.93 | 2.52 | 3.408 (19) | 159 |
| C17*A*—H17*A*···O3 | 0.93 | 2.58 | 3.510 (10) | 177 |
| C20—H20*B*···O2$^{i}$ | 0.96 | 2.53 | 3.441 (4) | 158 |
| C20—H20*C*···O3 | 0.96 | 2.44 | 3.085 (4) | 124 |
| C25—H25*A*···O4$^{iii}$ | 0.93 | 2.55 | 3.264 (4) | 134 |
| C13*A*—H13*A*···*Cg*5$^{ii}$ | 0.93 | 2.82 | 3.575 (10) | 139 |
| C16*A*—H16*A*···*Cg*5 | 0.93 | 2.98 | 3.826 (9) | 151 |
| C19*A*—H19*B*···*Cg*4$^{iii}$ | 0.96 | 2.99 | 3.862 (11) | 152 |
| C13*B*—H13*B*···*Cg*5$^{ii}$ | 0.93 | 2.95 | 3.765 (16) | 147 |
| C16*B*—H16*B*···*Cg*5 | 0.93 | 2.70 | 3.562 (13) | 155 |

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