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# Fluphenazine dihydrochloride dimethanol solvate

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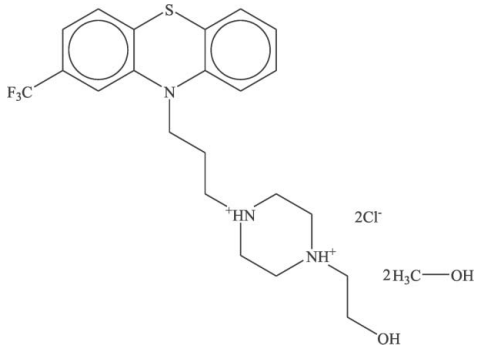
Key indicators: single-crystal X-ray study; $T$ = 85 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.084; $wR$ factor = 0.197; data-to-parameter ratio = 42.2.

In the title compound {systematic name: 1-(2-hydroxyethyl)-4-[3-(2-trifluoromethyl-10*H*-phenothiazin-10-yl)propyl]piperazine-1,4-diium dichloride dimethanol disolvate}, $C_{22}H_{28}F_3N_3OS^{2+}\cdot 2Cl^-\cdot 2CH_3OH$, the dihedral angle between the planes of the two outer benzene rings of the tricyclic phenothiazine system is 46.91 (13)°. The piperazine ring adopts a chair conformation. The crystal structure is stabilized by O—H···Cl, N—H···Cl, C—H···O, C—H···Cl and C—H···F hydrogen bonds and contacts.

## Related literature

For the properties of phenothiazines, see: Ford *et al.* (1988); Ohlow & Moosmann (2011); Tsakovska & Pajeva (2006) and for the biological properties of fluphenazine, see: Gasiorowski *et al.* (2001); Szabó *et al.* (1999). For related structures, see: Dahl *et al.* (1986); Dutkiewicz *et al.* (2010); McDowell (1978, 1980); Yathirajan *et al.* (2007). For puckering parameters, see: Cremer & Pople (1975);.




## Experimental

### Crystal data

$C_{22}H_{28}F_3N_3OS^{2+}\cdot 2Cl^-\cdot 2(CH_4O)$
$M_r$ = 574.53
Orthorhombic, $Pca2_1$
$a$ = 39.76 (2) Å
$b$ = 9.952 (8) Å
$c$ = 7.127 (5) Å
$V$ = 2820 (3) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.35 mm$^{-1}$
$T$ = 85 K
0.24 × 0.02 × 0.01 mm

### Data collection

Oxford Diffraction Xcalibur PX $\kappa$-geometry diffractometer with Onyx CCD camera
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2007)
$T_{min}$ = 0.850, $T_{max}$ = 1.000
43952 measured reflections
13922 independent reflections
10615 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.052

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.084
$wR(F^2)$ = 0.197
$S$ = 1.19
13922 reflections
330 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.25 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.85 e Å$^{-3}$
Absolute structure: Flack (1983), 5579 Friedel pairs
Flack parameter: 0.09 (7)

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N16—H16···Cl2 | 0.93 | 2.12 | 3.017 (3) | 161 |
| N18—H18···Cl1 | 0.93 | 2.16 | 3.078 (3) | 171 |
| O24—H24···Cl1 | 0.84 | 2.31 | 3.147 (3) | 172 |
| O22—H22···Cl2$^{i}$ | 0.84 | 2.27 | 3.065 (3) | 157 |
| O23—H23···Cl2$^{ii}$ | 0.84 | 2.36 | 3.169 (3) | 163 |
| C18—H18*A*···O22 | 0.99 | 2.23 | 2.924 (4) | 126 |
| C21—H21*A*···O23 | 0.99 | 2.39 | 3.266 (5) | 147 |
| C2—H2···F13*A*$^{iii}$ | 0.95 | 2.45 | 3.381 (4) | 165 |
| C14—H14*A*···O23$^{ii}$ | 0.99 | 2.51 | 3.482 (5) | 169 |
| C17—H17*A*···O24$^{iv}$ | 0.99 | 2.24 | 3.215 (4) | 166 |
| C17—H17*B*···O22$^{i}$ | 0.99 | 2.55 | 3.379 (5) | 141 |
| C16—H16*B*···Cl2$^{v}$ | 0.99 | 2.67 | 3.619 (4) | 161 |
| C19—H19*B*···Cl2$^{ii}$ | 0.99 | 2.75 | 3.529 (3) | 136 |

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2007); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); 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: BT5828).

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# supplementary materials

*Acta Cryst.* (2012). E**68**, o1004–o1005 [doi:10.1107/S1600536812008707]

## Fluphenazine dihydrochloride dimethanol solvate

**Joanna Petrus, Rafał Petrus and Bogusława Czarnik-Matusewicz**

### Comment

Fluphenazine (2-(4-(3-(2-(trifluoromethyl)-10*H*-phenothiazin-10-yl)propyl) piperazin-1-yl)ethanol)) (FPh) belongs to one of the oldest and the biggest family of antipsychotic drugs known as phenothiazines (Ohlow & Moosmann, 2011). Apart from its application in the treatment of many psychoses (mainly schizophrenia, mania and paranoid syndromes), it exhibits also a broad spectrum of biological effects, among them the anti-MDR (multidrug resistance) potency. (Gasiorowski *et al.*, 2001; Szabó *et al.*, 1999). Due to the anti-MDR activity of phenothiazines is strictly correlated with their structure (Tsakovska & Pajeva, 2006; Ford *et al.*, 1988), the aim of our work is to characterize the solid state structure of fluphenazine. In the crystal structure of I (Fig. 1), the dihedral angle between the planes of the two outer benzene rings of the phenothiazine system known as 'butterfly angle′, correlates with values find for phenothiazines with high biological activity (Dahl *et al.*, 1986; McDowell, 1978; Yathirajan *et al.*, 2007). The piperazine ring adopts a chair conformation, as in the case before reported fluphenazine dipicrate (Dutkiewicz *et al.*, 2010), described by the Cremer & Pople (1975) puckering parameters $q_2$ = 0.019 Å, $\varphi_2$ = 13.9°, $q_3$ = -0.593 Å, $Q$ = 0.593 Å, $\theta$ = 178.2°. The crystal structure is stabilized by O—H···Cl, N—H···Cl, C—H···O, C—H···Cl and C—H···F hydrogen bonds and contacts (Table 1 and Fig. 2), that are very similar to those in trifluperazine dihydrochloride (McDowell, 1980).

### Experimental

The $FPh^{2+}\cdot 2Cl^{-}\cdot 2CH_3OH$ crystals were obtained by slow evaporation of methanol solution of dihydrochloride fluphenazine (Jelfa) at -15°C.

### Refinement

All H atoms were found in difference Fourier maps. In the final refinement cycles, all H atoms were positioned geometrically and treated as riding atoms, with C—H = 0.95–0.99 Å, N—H = 0.93 Å and O—H = 0.84 Å, and with $U_{iso}$(H) = 1.2$U_{eq}$(C, Nsp$^3$) or 1.5$U_{eq}$(O, $C_{methyl}$).

### Computing details

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2007); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2007); data reduction: *CrysAlis RED* (Oxford Diffraction, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

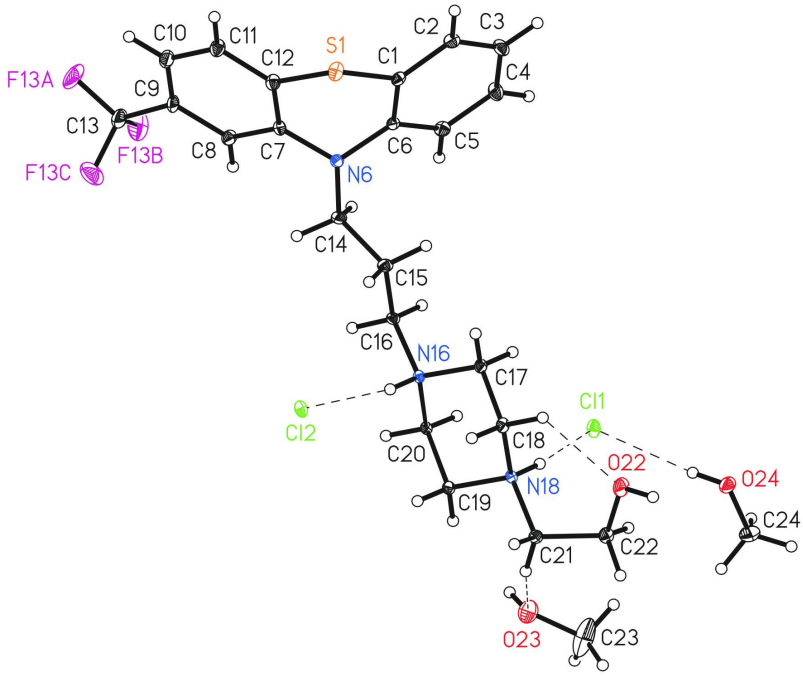


**Figure 1**

The structures and atom-numbering schemes for the FPh dication, chloride anions and solvent molecules joined by hydrogen bonds (dashed lines) in the asymmetric unit of I. Displacement ellipsoids are drawn at the 30% probability level.

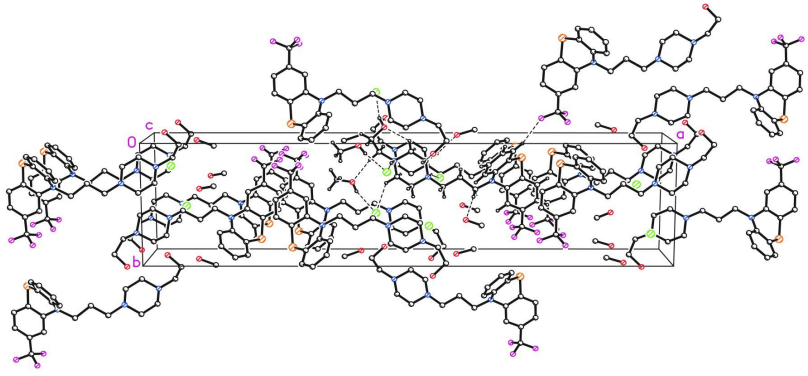


**Figure 2**

Packing diagram of the title compound viewed along the *c* axis.

## 1-(2-hydroxyethyl)-4-[3-(2-trifluoromethyl-10*H*-phenothiazin- 10-yl)propyl]piperazine-1,4-diium dichloride dimethanol disolvate

*Crystal data*

$C_{22}H_{28}F_3N_3OS^{2+}\cdot 2Cl^-\cdot 2(CH_4O)$
$M_r = 574.53$
Orthorhombic, $Pca2_1$
Hall symbol: P 2c -2ac
$a = 39.76$ (2) Å
$b = 9.952$ (8) Å
$c = 7.127$ (5) Å
$V = 2820$ (3) Å$^3$
$Z = 4$
$F(000) = 1208$
$D_x = 1.353$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 13454 reflections
$\theta = 4.8$–$38.5°$
$\mu = 0.35$ mm$^{-1}$
$T = 85$ K
Needle, colourless
0.24 × 0.02 × 0.01 mm

*Data collection*

Oxford Diffraction Xcalibur PX $\kappa$-geometry diffractometer with CCD Onyx camera
Radiation source: fine-focus sealed tube
Graphite/ monochromator
$\omega$ and $\varphi$ scans
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2007)
$T_{min} = 0.850$, $T_{max} = 1.000$
43952 measured reflections
13922 independent reflections
10615 reflections with $I > 2\sigma(I)$
$R_{int} = 0.052$
$\theta_{max} = 38.6°$, $\theta_{min} = 4.8°$
$h = -69\rightarrow 64$
$k = -17\rightarrow 15$
$l = -12\rightarrow 10$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.084$
$wR(F^2) = 0.197$
$S = 1.19$
13922 reflections
330 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.062P)^2 + 4.274P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.25$ e Å$^{-3}$
$\Delta\rho_{min} = -0.85$ e Å$^{-3}$
Absolute structure: Flack (1983), 5579 Friedel pairs
Flack parameter: 0.09 (7)

*Special details*

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

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

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | 0.716794 (19) | 0.15955 (8) | 0.47197 (13) | 0.01988 (15) |
| C1 | 0.69059 (7) | 0.1626 (3) | 0.2720 (4) | 0.0141 (5) |

| | | | | |
|---|---|---|---|---|
| C2 | 0.69591 (8) | 0.0733 (3) | 0.1245 (5) | 0.0186 (6) |
| H2 | 0.7139 | 0.0109 | 0.1301 | 0.022* |
| C3 | 0.67489 (8) | 0.0756 (3) | −0.0306 (5) | 0.0217 (6) |
| H3 | 0.6781 | 0.0136 | −0.1303 | 0.026* |
| C4 | 0.64910 (8) | 0.1692 (3) | −0.0392 (5) | 0.0218 (5) |
| H4 | 0.6347 | 0.1709 | −0.1456 | 0.026* |
| C5 | 0.64397 (7) | 0.2604 (3) | 0.1050 (5) | 0.0178 (5) |
| H5 | 0.6265 | 0.3251 | 0.0956 | 0.021* |
| C6 | 0.66444 (7) | 0.2571 (3) | 0.2641 (4) | 0.0131 (4) |
| N6 | 0.66034 (6) | 0.3468 (2) | 0.4191 (4) | 0.0154 (5) |
| C7 | 0.68982 (7) | 0.4120 (3) | 0.4788 (4) | 0.0146 (5) |
| C8 | 0.69142 (8) | 0.5497 (3) | 0.5105 (5) | 0.0176 (5) |
| H8 | 0.6722 | 0.6045 | 0.4893 | 0.021* |
| C9 | 0.72137 (8) | 0.6071 (3) | 0.5736 (5) | 0.0204 (6) |
| C10 | 0.75000 (9) | 0.5322 (3) | 0.5989 (5) | 0.0219 (6) |
| H10 | 0.7702 | 0.5731 | 0.6409 | 0.026* |
| C11 | 0.74876 (8) | 0.3957 (3) | 0.5618 (5) | 0.0208 (6) |
| H11 | 0.7686 | 0.3432 | 0.5746 | 0.025* |
| C12 | 0.71912 (7) | 0.3344 (3) | 0.5065 (4) | 0.0174 (5) |
| C13 | 0.72107 (9) | 0.7549 (4) | 0.6146 (6) | 0.0288 (8) |
| F13A | 0.75142 (7) | 0.8068 (2) | 0.6355 (6) | 0.0553 (10) |
| F13B | 0.70499 (8) | 0.8260 (2) | 0.4841 (5) | 0.0470 (7) |
| F13C | 0.70460 (9) | 0.7820 (3) | 0.7761 (5) | 0.0500 (8) |
| C14 | 0.62843 (7) | 0.4191 (3) | 0.4335 (5) | 0.0178 (5) |
| H14A | 0.6242 | 0.4684 | 0.3151 | 0.021* |
| H14B | 0.6299 | 0.4858 | 0.5363 | 0.021* |
| C15 | 0.59930 (7) | 0.3231 (3) | 0.4715 (5) | 0.0163 (5) |
| H15A | 0.5998 | 0.2482 | 0.3802 | 0.020* |
| H15B | 0.6013 | 0.2850 | 0.5993 | 0.020* |
| C16 | 0.56640 (6) | 0.4003 (2) | 0.4535 (5) | 0.0133 (4) |
| H16A | 0.5691 | 0.4910 | 0.5085 | 0.016* |
| H16B | 0.5607 | 0.4111 | 0.3192 | 0.016* |
| N16 | 0.53816 (6) | 0.3281 (2) | 0.5520 (3) | 0.0112 (4) |
| H16 | 0.5436 | 0.3238 | 0.6788 | 0.013* |
| C17 | 0.53309 (7) | 0.1872 (3) | 0.4842 (4) | 0.0142 (5) |
| H17A | 0.5541 | 0.1354 | 0.5009 | 0.017* |
| H17B | 0.5276 | 0.1882 | 0.3488 | 0.017* |
| C18 | 0.50516 (7) | 0.1208 (3) | 0.5911 (5) | 0.0146 (5) |
| H18A | 0.5022 | 0.0278 | 0.5447 | 0.018* |
| H18B | 0.5113 | 0.1160 | 0.7256 | 0.018* |
| N18 | 0.47276 (6) | 0.1961 (2) | 0.5704 (3) | 0.0108 (4) |
| H18 | 0.4676 | 0.2015 | 0.4433 | 0.013* |
| C19 | 0.47808 (7) | 0.3362 (3) | 0.6430 (4) | 0.0132 (4) |
| H19A | 0.4840 | 0.3327 | 0.7779 | 0.016* |
| H19B | 0.4570 | 0.3885 | 0.6296 | 0.016* |
| C20 | 0.50609 (6) | 0.4044 (2) | 0.5348 (4) | 0.0101 (4) |
| H20A | 0.4997 | 0.4111 | 0.4008 | 0.012* |
| H20B | 0.5093 | 0.4967 | 0.5835 | 0.012* |
| C21 | 0.44365 (7) | 0.1309 (3) | 0.6692 (4) | 0.0150 (5) |

| | | | | |
|---|---|---|---|---|
| H21A | 0.4259 | 0.1990 | 0.6905 | 0.018* |
| H21B | 0.4512 | 0.0981 | 0.7933 | 0.018* |
| C22 | 0.42902 (8) | 0.0152 (3) | 0.5600 (5) | 0.0170 (5) |
| H22A | 0.4094 | −0.0220 | 0.6277 | 0.020* |
| H22B | 0.4214 | 0.0468 | 0.4355 | 0.020* |
| O22 | 0.45382 (7) | −0.0860 (2) | 0.5376 (4) | 0.0262 (5) |
| H22 | 0.4445 | −0.1616 | 0.5324 | 0.039* |
| O23 | 0.39180 (8) | 0.3725 (3) | 0.5576 (5) | 0.0333 (6) |
| H23 | 0.4001 | 0.4451 | 0.5190 | 0.050* |
| C23 | 0.36311 (18) | 0.3410 (7) | 0.4515 (13) | 0.071 (2) |
| H23A | 0.3484 | 0.4200 | 0.4441 | 0.106* |
| H23B | 0.3510 | 0.2669 | 0.5115 | 0.106* |
| H23C | 0.3699 | 0.3142 | 0.3247 | 0.106* |
| O24 | 0.40656 (6) | 0.0214 (2) | 0.0563 (4) | 0.0233 (5) |
| H24 | 0.4190 | 0.0875 | 0.0808 | 0.035* |
| C24 | 0.37321 (9) | 0.0675 (5) | 0.0181 (6) | 0.0335 (9) |
| H24A | 0.3639 | 0.1101 | 0.1306 | 0.050* |
| H24B | 0.3591 | −0.0090 | −0.0179 | 0.050* |
| H24C | 0.3738 | 0.1328 | −0.0847 | 0.050* |
| Cl1 | 0.459485 (19) | 0.24652 (8) | 0.15078 (11) | 0.01844 (13) |
| Cl2 | 0.562033 (17) | 0.38032 (7) | 0.94712 (10) | 0.01575 (12) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0209 (3) | 0.0132 (3) | 0.0255 (4) | 0.0016 (2) | −0.0071 (3) | 0.0018 (3) |
| C1 | 0.0136 (11) | 0.0084 (10) | 0.0203 (13) | −0.0007 (9) | −0.0001 (9) | 0.0013 (9) |
| C2 | 0.0164 (11) | 0.0105 (11) | 0.0289 (17) | 0.0025 (9) | 0.0035 (11) | −0.0022 (10) |
| C3 | 0.0259 (13) | 0.0161 (12) | 0.0230 (15) | 0.0017 (10) | 0.0032 (13) | −0.0057 (12) |
| C4 | 0.0257 (13) | 0.0220 (13) | 0.0175 (13) | 0.0010 (11) | −0.0047 (12) | −0.0027 (13) |
| C5 | 0.0160 (12) | 0.0170 (12) | 0.0203 (14) | 0.0028 (10) | −0.0017 (10) | −0.0001 (10) |
| C6 | 0.0135 (10) | 0.0061 (9) | 0.0198 (13) | −0.0007 (8) | 0.0023 (9) | −0.0009 (9) |
| N6 | 0.0128 (9) | 0.0112 (9) | 0.0223 (13) | 0.0003 (8) | 0.0018 (8) | −0.0045 (8) |
| C7 | 0.0162 (10) | 0.0123 (10) | 0.0153 (12) | 0.0000 (8) | −0.0009 (9) | −0.0019 (9) |
| C8 | 0.0164 (11) | 0.0110 (11) | 0.0253 (15) | −0.0001 (9) | 0.0000 (10) | −0.0015 (10) |
| C9 | 0.0211 (13) | 0.0143 (12) | 0.0258 (15) | −0.0048 (10) | −0.0021 (12) | −0.0064 (11) |
| C10 | 0.0200 (13) | 0.0205 (14) | 0.0253 (16) | −0.0021 (11) | −0.0043 (12) | −0.0034 (12) |
| C11 | 0.0181 (12) | 0.0198 (14) | 0.0246 (15) | 0.0008 (10) | −0.0057 (11) | −0.0019 (12) |
| C12 | 0.0163 (11) | 0.0175 (12) | 0.0184 (14) | 0.0012 (10) | −0.0041 (10) | −0.0004 (10) |
| C13 | 0.0258 (15) | 0.0192 (15) | 0.041 (2) | −0.0062 (12) | 0.0014 (14) | −0.0085 (15) |
| F13A | 0.0261 (11) | 0.0234 (11) | 0.116 (3) | −0.0107 (10) | 0.0014 (16) | −0.0251 (15) |
| F13B | 0.0628 (17) | 0.0125 (9) | 0.066 (2) | −0.0013 (10) | −0.0150 (16) | −0.0016 (11) |
| F13C | 0.066 (2) | 0.0277 (13) | 0.0561 (19) | −0.0046 (13) | 0.0231 (16) | −0.0180 (13) |
| C14 | 0.0139 (10) | 0.0171 (12) | 0.0224 (14) | 0.0014 (9) | 0.0035 (11) | −0.0034 (11) |
| C15 | 0.0135 (10) | 0.0122 (10) | 0.0233 (14) | 0.0015 (8) | 0.0036 (10) | −0.0010 (11) |
| C16 | 0.0135 (10) | 0.0110 (10) | 0.0153 (11) | 0.0004 (7) | 0.0025 (10) | −0.0001 (10) |
| N16 | 0.0117 (9) | 0.0114 (9) | 0.0104 (9) | 0.0011 (7) | −0.0015 (7) | −0.0008 (8) |
| C17 | 0.0143 (10) | 0.0095 (10) | 0.0187 (13) | 0.0009 (8) | −0.0010 (9) | −0.0022 (9) |
| C18 | 0.0161 (11) | 0.0062 (9) | 0.0216 (13) | 0.0014 (8) | −0.0022 (10) | −0.0014 (9) |
| N18 | 0.0133 (9) | 0.0072 (8) | 0.0118 (10) | 0.0012 (7) | −0.0008 (8) | 0.0006 (7) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C19 | 0.0155 (10) | 0.0084 (9) | 0.0156 (11) | −0.0011 (8) | 0.0013 (9) | −0.0011 (9) |
| C20 | 0.0140 (10) | 0.0037 (9) | 0.0127 (11) | 0.0007 (7) | −0.0002 (8) | 0.0011 (7) |
| C21 | 0.0168 (11) | 0.0126 (11) | 0.0157 (12) | −0.0032 (9) | 0.0031 (9) | 0.0008 (9) |
| C22 | 0.0212 (12) | 0.0118 (11) | 0.0179 (13) | −0.0033 (9) | 0.0008 (10) | 0.0016 (10) |
| O22 | 0.0263 (11) | 0.0120 (9) | 0.0404 (15) | −0.0039 (8) | 0.0014 (11) | −0.0060 (10) |
| O23 | 0.0375 (15) | 0.0228 (13) | 0.0394 (17) | −0.0038 (11) | −0.0043 (13) | 0.0034 (12) |
| C23 | 0.080 (4) | 0.055 (3) | 0.077 (4) | −0.036 (3) | −0.038 (4) | 0.013 (3) |
| O24 | 0.0189 (10) | 0.0157 (10) | 0.0353 (14) | −0.0021 (8) | 0.0012 (10) | −0.0041 (10) |
| C24 | 0.0175 (14) | 0.045 (2) | 0.038 (2) | 0.0028 (15) | 0.0015 (14) | −0.0069 (17) |
| Cl1 | 0.0254 (3) | 0.0182 (3) | 0.0118 (2) | −0.0055 (3) | −0.0041 (3) | 0.0017 (2) |
| Cl2 | 0.0212 (3) | 0.0131 (2) | 0.0129 (3) | 0.0022 (2) | −0.0034 (2) | −0.0012 (2) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—C12 | 1.760 (4) | C16—H16B | 0.9900 |
| S1—C1 | 1.766 (3) | N16—C20 | 1.489 (3) |
| C1—C2 | 1.393 (4) | N16—C17 | 1.497 (4) |
| C1—C6 | 1.403 (4) | N16—H16 | 0.9300 |
| C2—C3 | 1.386 (5) | C17—C18 | 1.500 (4) |
| C2—H2 | 0.9500 | C17—H17A | 0.9900 |
| C3—C4 | 1.387 (4) | C17—H17B | 0.9900 |
| C3—H3 | 0.9500 | C18—N18 | 1.498 (4) |
| C4—C5 | 1.386 (5) | C18—H18A | 0.9900 |
| C4—H4 | 0.9500 | C18—H18B | 0.9900 |
| C5—C6 | 1.396 (4) | N18—C19 | 1.502 (4) |
| C5—H5 | 0.9500 | N18—C21 | 1.502 (4) |
| C6—N6 | 1.430 (4) | N18—H18 | 0.9300 |
| N6—C7 | 1.406 (4) | C19—C20 | 1.516 (4) |
| N6—C14 | 1.462 (4) | C19—H19A | 0.9900 |
| C7—C8 | 1.390 (4) | C19—H19B | 0.9900 |
| C7—C12 | 1.412 (4) | C20—H20A | 0.9900 |
| C8—C9 | 1.395 (4) | C20—H20B | 0.9900 |
| C8—H8 | 0.9500 | C21—C22 | 1.507 (4) |
| C9—C10 | 1.373 (5) | C21—H21A | 0.9900 |
| C9—C13 | 1.499 (5) | C21—H21B | 0.9900 |
| C10—C11 | 1.385 (5) | C22—O22 | 1.418 (4) |
| C10—H10 | 0.9500 | C22—H22A | 0.9900 |
| C11—C12 | 1.385 (4) | C22—H22B | 0.9900 |
| C11—H11 | 0.9500 | O22—H22 | 0.8400 |
| C13—F13A | 1.321 (4) | O23—C23 | 1.404 (7) |
| C13—F13B | 1.332 (5) | O23—H23 | 0.8400 |
| C13—F13C | 1.352 (5) | C23—H23A | 0.9800 |
| C14—C15 | 1.526 (4) | C23—H23B | 0.9800 |
| C14—H14A | 0.9900 | C23—H23C | 0.9800 |
| C14—H14B | 0.9900 | O24—C24 | 1.429 (4) |
| C15—C16 | 1.522 (4) | O24—H24 | 0.8400 |
| C15—H15A | 0.9900 | C24—H24A | 0.9800 |
| C15—H15B | 0.9900 | C24—H24B | 0.9800 |
| C16—N16 | 1.507 (4) | C24—H24C | 0.9800 |
| C16—H16A | 0.9900 | | |

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|---|---|---|---|
| C12—S1—C1 | 97.29 (14) | C20—N16—C17 | 109.6 (2) |
| C2—C1—C6 | 120.6 (3) | C20—N16—C16 | 110.9 (2) |
| C2—C1—S1 | 120.6 (2) | C17—N16—C16 | 113.4 (2) |
| C6—C1—S1 | 118.8 (2) | C20—N16—H16 | 107.6 |
| C3—C2—C1 | 120.0 (3) | C17—N16—H16 | 107.6 |
| C3—C2—H2 | 120.0 | C16—N16—H16 | 107.6 |
| C1—C2—H2 | 120.0 | N16—C17—C18 | 110.4 (2) |
| C2—C3—C4 | 119.5 (3) | N16—C17—H17A | 109.6 |
| C2—C3—H3 | 120.3 | C18—C17—H17A | 109.6 |
| C4—C3—H3 | 120.3 | N16—C17—H17B | 109.6 |
| C5—C4—C3 | 121.1 (3) | C18—C17—H17B | 109.6 |
| C5—C4—H4 | 119.5 | H17A—C17—H17B | 108.1 |
| C3—C4—H4 | 119.5 | N18—C18—C17 | 111.5 (2) |
| C4—C5—C6 | 120.1 (3) | N18—C18—H18A | 109.3 |
| C4—C5—H5 | 120.0 | C17—C18—H18A | 109.3 |
| C6—C5—H5 | 120.0 | N18—C18—H18B | 109.3 |
| C5—C6—C1 | 118.7 (3) | C17—C18—H18B | 109.3 |
| C5—C6—N6 | 123.1 (3) | H18A—C18—H18B | 108.0 |
| C1—C6—N6 | 118.2 (3) | C18—N18—C19 | 108.0 (2) |
| C7—N6—C6 | 115.3 (2) | C18—N18—C21 | 113.6 (2) |
| C7—N6—C14 | 118.4 (2) | C19—N18—C21 | 110.3 (2) |
| C6—N6—C14 | 117.4 (2) | C18—N18—H18 | 108.2 |
| C8—C7—N6 | 122.8 (3) | C19—N18—H18 | 108.2 |
| C8—C7—C12 | 118.6 (3) | C21—N18—H18 | 108.2 |
| N6—C7—C12 | 118.6 (3) | N18—C19—C20 | 110.1 (2) |
| C7—C8—C9 | 119.7 (3) | N18—C19—H19A | 109.6 |
| C7—C8—H8 | 120.2 | C20—C19—H19A | 109.6 |
| C9—C8—H8 | 120.2 | N18—C19—H19B | 109.6 |
| C10—C9—C8 | 121.9 (3) | C20—C19—H19B | 109.6 |
| C10—C9—C13 | 120.9 (3) | H19A—C19—H19B | 108.1 |
| C8—C9—C13 | 117.2 (3) | N16—C20—C19 | 111.0 (2) |
| C9—C10—C11 | 118.6 (3) | N16—C20—H20A | 109.4 |
| C9—C10—H10 | 120.7 | C19—C20—H20A | 109.4 |
| C11—C10—H10 | 120.7 | N16—C20—H20B | 109.4 |
| C12—C11—C10 | 121.1 (3) | C19—C20—H20B | 109.4 |
| C12—C11—H11 | 119.5 | H20A—C20—H20B | 108.0 |
| C10—C11—H11 | 119.5 | N18—C21—C22 | 112.7 (2) |
| C11—C12—C7 | 120.1 (3) | N18—C21—H21A | 109.1 |
| C11—C12—S1 | 121.3 (2) | C22—C21—H21A | 109.1 |
| C7—C12—S1 | 118.6 (2) | N18—C21—H21B | 109.1 |
| F13A—C13—F13B | 108.0 (4) | C22—C21—H21B | 109.1 |
| F13A—C13—F13C | 105.6 (3) | H21A—C21—H21B | 107.8 |
| F13B—C13—F13C | 104.8 (3) | O22—C22—C21 | 109.4 (3) |
| F13A—C13—C9 | 113.5 (3) | O22—C22—H22A | 109.8 |
| F13B—C13—C9 | 112.9 (3) | C21—C22—H22A | 109.8 |
| F13C—C13—C9 | 111.4 (3) | O22—C22—H22B | 109.8 |
| N6—C14—C15 | 111.3 (2) | C21—C22—H22B | 109.8 |
| N6—C14—H14A | 109.4 | H22A—C22—H22B | 108.2 |

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|---|---|---|---|
| C15—C14—H14A | 109.4 | C22—O22—H22 | 109.5 |
| N6—C14—H14B | 109.4 | C23—O23—H23 | 109.5 |
| C15—C14—H14B | 109.4 | O23—C23—H23A | 109.5 |
| H14A—C14—H14B | 108.0 | O23—C23—H23B | 109.5 |
| C16—C15—C14 | 108.8 (2) | H23A—C23—H23B | 109.5 |
| C16—C15—H15A | 109.9 | O23—C23—H23C | 109.5 |
| C14—C15—H15A | 109.9 | H23A—C23—H23C | 109.5 |
| C16—C15—H15B | 109.9 | H23B—C23—H23C | 109.5 |
| C14—C15—H15B | 109.9 | C24—O24—H24 | 109.5 |
| H15A—C15—H15B | 108.3 | O24—C24—H24A | 109.5 |
| N16—C16—C15 | 111.1 (2) | O24—C24—H24B | 109.5 |
| N16—C16—H16A | 109.4 | H24A—C24—H24B | 109.5 |
| C15—C16—H16A | 109.4 | O24—C24—H24C | 109.5 |
| N16—C16—H16B | 109.4 | H24A—C24—H24C | 109.5 |
| C15—C16—H16B | 109.4 | H24B—C24—H24C | 109.5 |
| H16A—C16—H16B | 108.0 | | |

| | | | |
|---|---|---|---|
| C12—S1—C1—C2 | 141.0 (3) | N6—C7—C12—C11 | 178.8 (3) |
| C12—S1—C1—C6 | −38.6 (3) | C8—C7—C12—S1 | 178.4 (2) |
| C6—C1—C2—C3 | −1.2 (4) | N6—C7—C12—S1 | −2.0 (4) |
| S1—C1—C2—C3 | 179.2 (2) | C1—S1—C12—C11 | −141.1 (3) |
| C1—C2—C3—C4 | 1.4 (5) | C1—S1—C12—C7 | 39.8 (3) |
| C2—C3—C4—C5 | −0.1 (5) | C10—C9—C13—F13A | 13.7 (6) |
| C3—C4—C5—C6 | −1.4 (5) | C8—C9—C13—F13A | −166.8 (4) |
| C4—C5—C6—C1 | 1.6 (4) | C10—C9—C13—F13B | 137.1 (4) |
| C4—C5—C6—N6 | −179.2 (3) | C8—C9—C13—F13B | −43.4 (5) |
| C2—C1—C6—C5 | −0.3 (4) | C10—C9—C13—F13C | −105.3 (4) |
| S1—C1—C6—C5 | 179.3 (2) | C8—C9—C13—F13C | 74.2 (5) |
| C2—C1—C6—N6 | −179.5 (3) | C7—N6—C14—C15 | −148.0 (3) |
| S1—C1—C6—N6 | 0.1 (4) | C6—N6—C14—C15 | 66.1 (4) |
| C5—C6—N6—C7 | −130.1 (3) | N6—C14—C15—C16 | −170.9 (3) |
| C1—C6—N6—C7 | 49.0 (4) | C14—C15—C16—N16 | −160.3 (3) |
| C5—C6—N6—C14 | 16.7 (4) | C15—C16—N16—C20 | 179.7 (2) |
| C1—C6—N6—C14 | −164.1 (3) | C15—C16—N16—C17 | −56.5 (3) |
| C6—N6—C7—C8 | 131.6 (3) | C20—N16—C17—C18 | −56.8 (3) |
| C14—N6—C7—C8 | −14.9 (5) | C16—N16—C17—C18 | 178.8 (2) |
| C6—N6—C7—C12 | −47.9 (4) | N16—C17—C18—N18 | 59.1 (3) |
| C14—N6—C7—C12 | 165.6 (3) | C17—C18—N18—C19 | −59.3 (3) |
| N6—C7—C8—C9 | 178.6 (3) | C17—C18—N18—C21 | 177.9 (2) |
| C12—C7—C8—C9 | −1.9 (5) | C18—N18—C19—C20 | 58.7 (3) |
| C7—C8—C9—C10 | 2.6 (5) | C21—N18—C19—C20 | −176.6 (2) |
| C7—C8—C9—C13 | −177.0 (3) | C17—N16—C20—C19 | 57.3 (3) |
| C8—C9—C10—C11 | −0.6 (6) | C16—N16—C20—C19 | −176.8 (2) |
| C13—C9—C10—C11 | 179.0 (3) | N18—C19—C20—N16 | −59.3 (3) |
| C9—C10—C11—C12 | −2.1 (6) | C18—N18—C21—C22 | −79.5 (3) |
| C10—C11—C12—C7 | 2.8 (5) | C19—N18—C21—C22 | 159.0 (2) |
| C10—C11—C12—S1 | −176.4 (3) | N18—C21—C22—O22 | 61.8 (3) |
| C8—C7—C12—C11 | −0.7 (5) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N16—H16···Cl2 | 0.93 | 2.12 | 3.017 (3) | 161 |
| N18—H18···Cl1 | 0.93 | 2.16 | 3.078 (3) | 171 |
| O24—H24···Cl1 | 0.84 | 2.31 | 3.147 (3) | 172 |
| O22—H22···Cl2$^{i}$ | 0.84 | 2.27 | 3.065 (3) | 157 |
| O23—H23···Cl2$^{ii}$ | 0.84 | 2.36 | 3.169 (3) | 163 |
| C18—H18*A*···O22 | 0.99 | 2.23 | 2.924 (4) | 126 |
| C21—H21*A*···O23 | 0.99 | 2.39 | 3.266 (5) | 147 |
| C2—H2···F13*A*$^{iii}$ | 0.95 | 2.45 | 3.381 (4) | 165 |
| C14—H14*A*···O23$^{ii}$ | 0.99 | 2.51 | 3.482 (5) | 169 |
| C17—H17*A*···O24$^{iv}$ | 0.99 | 2.24 | 3.215 (4) | 166 |
| C17—H17*B*···O22$^{i}$ | 0.99 | 2.55 | 3.379 (5) | 141 |
| C16—H16*B*···Cl2$^{v}$ | 0.99 | 2.67 | 3.619 (4) | 161 |
| C19—H19*B*···Cl2$^{ii}$ | 0.99 | 2.75 | 3.529 (3) | 136 |

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