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1-[2-(2-Thienylcarbonyl)ethyl]-4-[3-(trifluoromethyl)phenyl]piperazin-1-ium chloride

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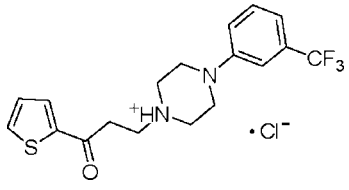
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.007 Å; disorder in main residue; R factor = 0.062; wR factor = 0.187; data-to-parameter ratio = 15.0.

The title compound, $C_{18}H_{20}F_3N_2OS^+\cdot Cl^-$, was synthesized from 2-acetothiophene, 1-[3-(trifluoromethyl)phenyl]-piperazine and paraformaldehyde. There are two independent ion pairs in the asymmetric unit. In each cation, the piperazine ring adopts a chair conformation and the F atoms of the trifluoromethyl groups are disordered over two sites with a ratio of major and minor occupancy of approximately 0.6:0.4. Two intermolecular N—H···Cl hydrogen bonds help to stabilize the crystal structure.

Related literature

For background information, see: Oficialdegui *et al.* (2000); Martinez *et al.* (2001); Esparza *et al.* (2001); Orus *et al.* (2002).



Experimental

Crystal data

$C_{18}H_{20}F_3N_2OS^+\cdot Cl^-$
M_r = 404.89
Monoclinic, $P2_1/n$
a = 10.5715 (14) Å
b = 10.4989 (14) Å
c = 34.966 (8) Å
β = 90.040 (6)°
V = 3880.8 (12) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.34 mm^{-1}
T = 294 (2) K
0.20 × 0.14 × 0.12 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.935, T_{max} = 0.960
21590 measured reflections
7997 independent reflections
3476 reflections with $I > 2\sigma(I)$
R_{int} = 0.061

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.187
S = 1.00
7997 reflections
533 parameters
156 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···Cl2	0.94 (3)	2.10 (3)	3.025 (3)	168 (4)
N1—H1A···Cl1^{i}	0.93 (3)	2.11 (3)	3.033 (3)	174 (3)

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2431).

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supplementary materials

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1-[2-(2-Thienylcarbonyl)ethyl]-4-[3-(trifluoromethyl)phenyl]piperazin-1-ium chloride

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Comment

Mannich bases can be used as anti-corrosion reagents or as a class of selective antidepressants with a low incidence of side effects (Oficialdegui *et al.*, 2000; Martinez *et al.*, 2001; Esparza *et al.*, 2001; Orus *et al.*, 2002). The title compound, was synthesized from 2-acetothiophene, 1-(3-Trifluoromethyl-phenyl)-piperazineand and paraformaldehyde. The molecular structure of the title compound is illustrated in Fig. 1. The asymmetric unit contains two independent molecules. Two intermolecular N—H···Cl hydrogen bonds help to consolidate the crystal packing. In each molecule, the piperazine ring has a chair conformation and the F atoms of the CF_3 groups are disordered over two sites.

Experimental

A mixture of 2-acetothiophene 1.26 (10 mmol), 1-(3-Trifluoromethyl-phenyl)-piperazine 2.3 g (15 mmol) and concentrated hydrochloric acid (10 ml) in absolute ethanol(20 ml) was refluxed. Paraformaldehyde 0.9 g (45 mmol) was added over a period of 20 min. The reaction mixture was refluxed for another 12 h, cooled and the solid was precipitated. 20 ml acetone was added to the mixture and stirred for 30 min. The mixture was filtrated, washed with ethyl ether, dried in vacuum to give the title compound. The title compound was dissolved in absolute methanol; the solution was kept at ambient temperature for 15 d by natural evaporation to give single crystals suitable for X-ray analysis.

Refinement

Carbon-bound H atoms were positioned geometrically, with C—H = 0.93–0.97 Å and U_{iso}(H) = 1.2 U_{eq}(C), The nitrogen-bound H atoms were refined freely.

Figures

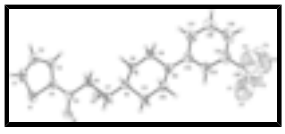


Fig. 1. The molecular structure of one of the cations drawn with 30% probability ellipsoids.

1-[2-(2-Thienylcarbonyl)ethyl]-4-[3-(trifluoromethyl)phenyl]piperazin-1-ium chloride

Crystal data

$C_{18}H_{20}F_3N_2OS^+\cdot Cl^-$	F_{000} = 1680
M_r = 404.89	D_x = 1.386 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 3440 reflections

a = 10.5715 (14) Å	θ = 2.3–22.6º
b = 10.4989 (14) Å	μ = 0.34 mm^{-1}
c = 34.966 (8) Å	T = 294 (2) K
β = 90.040 (6)º	Block, colorless
V = 3880.8 (12) $Å^3$	0.20 × 0.14 × 0.12 mm
Z = 8	

Data collection

Bruker SMART CCD diffractometer	7997 independent reflections
Radiation source: fine-focus sealed tube	3476 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.061
T = 294(2) K	θ_{max} = 26.5º
φ and ω scans	θ_{min} = 1.2º
Absorption correction: multi-scan (SADABS; Bruker, 1997)	$h = -13 \rightarrow 8$
T_{min} = 0.935, T_{max} = 0.960	$k = -11 \rightarrow 13$
21590 measured reflections	$l = -43 \rightarrow 33$

Refinement

Refinement on F^2	156 restraints
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.062	$w = 1/[\sigma^2(F_o^2) + (0.04P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.187	$(\Delta/\sigma)_{max} < 0.001$
S = 1.00	$\Delta\rho_{max}$ = 0.42 e $Å^{-3}$
7997 reflections	$\Delta\rho_{min}$ = −0.29 e $Å^{-3}$
533 parameters	Extinction correction: none

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 $F^2 > 2$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)
S1	0.87631 (13)	0.26060 (17)	0.11320 (4)	0.0945 (5)	

S2	0.13158 (12)	0.17445 (15)	0.39331 (4)	0.0845 (5)	
Cl1	0.95342 (8)	0.50699 (9)	0.22049 (3)	0.0532 (3)	
Cl2	0.45144 (9)	0.43229 (10)	0.22268 (3)	0.0567 (3)	
F4	0.6149 (10)	0.3307 (14)	0.0024 (4)	0.160 (5)	0.59 (2)
F5	0.4496 (17)	0.2631 (12)	−0.0181 (4)	0.154 (4)	0.59 (2)
F6	0.4203 (15)	0.3934 (11)	0.0185 (3)	0.137 (5)	0.59 (2)
F4'	0.570 (2)	0.260 (2)	−0.0160 (5)	0.160 (7)	0.41 (2)
F5'	0.376 (2)	0.2455 (18)	−0.0130 (5)	0.152 (7)	0.41 (2)
F6'	0.503 (2)	0.3963 (16)	0.0258 (4)	0.129 (6)	0.41 (2)
O1	0.9179 (3)	0.1508 (3)	0.19011 (9)	0.0654 (9)	
O2	0.0775 (3)	0.2832 (3)	0.31653 (8)	0.0616 (8)	
N1	0.7286 (3)	0.2251 (3)	0.29390 (9)	0.0439 (8)	
H1A	0.669 (3)	0.163 (3)	0.2889 (11)	0.064 (13)*	
N2	0.6407 (3)	0.3079 (3)	0.36817 (10)	0.0554 (9)	
N3	0.2638 (3)	0.2183 (3)	0.21098 (9)	0.0397 (8)	
H3A	0.330 (3)	0.276 (4)	0.2164 (12)	0.086 (16)*	
N4	0.3515 (3)	0.1331 (3)	0.13693 (9)	0.0492 (9)	
C1	0.7761 (6)	0.3490 (6)	0.08685 (14)	0.100 (2)	
H1	0.7895	0.3684	0.0612	0.120*	
C2	0.6737 (5)	0.3892 (5)	0.10693 (15)	0.0781 (15)	
H2	0.6084	0.4377	0.0966	0.094*	
C3	0.6785 (4)	0.3486 (4)	0.14507 (12)	0.0557 (11)	
H3	0.6167	0.3681	0.1630	0.067*	
C4	0.7836 (4)	0.2773 (4)	0.15315 (12)	0.0538 (11)	
C5	0.8246 (4)	0.2186 (4)	0.18879 (12)	0.0492 (11)	
C6	0.7438 (4)	0.2431 (4)	0.22355 (11)	0.0540 (11)	
H6A	0.7150	0.3308	0.2232	0.065*	
H6B	0.6699	0.1885	0.2227	0.065*	
C7	0.8153 (3)	0.2185 (4)	0.26006 (11)	0.0489 (11)	
H7A	0.8542	0.1350	0.2589	0.059*	
H7B	0.8820	0.2813	0.2629	0.059*	
C8	0.6711 (4)	0.3527 (4)	0.29960 (12)	0.0545 (11)	
H8A	0.6261	0.3777	0.2766	0.065*	
H8B	0.7372	0.4148	0.3043	0.065*	
C9	0.5804 (4)	0.3512 (4)	0.33305 (12)	0.0583 (12)	
H9A	0.5472	0.4363	0.3369	0.070*	
H9B	0.5098	0.2955	0.3271	0.070*	
C10	0.7027 (4)	0.1857 (4)	0.36287 (12)	0.0602 (12)	
H10A	0.6392	0.1208	0.3583	0.072*	
H10B	0.7479	0.1634	0.3861	0.072*	
C11	0.7940 (4)	0.1878 (4)	0.32990 (12)	0.0551 (11)	
H11A	0.8616	0.2478	0.3353	0.066*	
H11B	0.8314	0.1040	0.3268	0.066*	
C12	0.5690 (4)	0.3231 (5)	0.40204 (14)	0.0648 (13)	
C13	0.6025 (5)	0.2608 (5)	0.43516 (15)	0.0832 (16)	
H13A	0.6717	0.2062	0.4350	0.100*	
C14	0.5359 (6)	0.2777 (7)	0.46829 (18)	0.102 (2)	
C15	0.4349 (7)	0.3582 (8)	0.4698 (2)	0.131 (3)	
H15A	0.3931	0.3724	0.4927	0.157*	

C16	0.3960 (6)	0.4176 (7)	0.4372 (2)	0.133 (3)	
H16A	0.3242	0.4688	0.4375	0.159*	
C17	0.4637 (6)	0.4017 (6)	0.40344 (18)	0.108 (2)	
H17A	0.4379	0.4445	0.3815	0.129*	
C18	0.5752 (7)	0.2091 (8)	0.5042 (3)	0.152 (3)	
F1	0.6814 (12)	0.1474 (16)	0.5034 (5)	0.212 (10)	0.40
F2	0.4798 (13)	0.1113 (12)	0.5086 (4)	0.222 (8)	0.40
F3	0.5516 (15)	0.2816 (12)	0.5349 (3)	0.191 (6)	0.40
F1'	0.6351 (9)	0.0965 (7)	0.4965 (2)	0.139 (3)	0.60
F2'	0.4833 (10)	0.1807 (13)	0.5273 (3)	0.231 (5)	0.60
F3'	0.6644 (7)	0.2900 (8)	0.5220 (2)	0.184 (4)	0.60
C19	0.2390 (5)	0.0884 (5)	0.41821 (14)	0.0835 (16)	
H19	0.2303	0.0700	0.4441	0.100*	
C20	0.3376 (4)	0.0491 (4)	0.39733 (13)	0.0668 (13)	
H20	0.4048	0.0012	0.4067	0.080*	
C21	0.3257 (4)	0.0905 (4)	0.35889 (12)	0.0516 (11)	
H21	0.3852	0.0728	0.3400	0.062*	
C22	0.2179 (4)	0.1591 (4)	0.35233 (11)	0.0476 (10)	
C23	0.1737 (4)	0.2176 (4)	0.31697 (11)	0.0460 (10)	
C24	0.2519 (3)	0.1999 (4)	0.28165 (11)	0.0505 (11)	
H24A	0.2840	0.1134	0.2811	0.061*	
H24B	0.3239	0.2570	0.2827	0.061*	
C25	0.1788 (3)	0.2249 (4)	0.24523 (11)	0.0442 (10)	
H25A	0.1118	0.1624	0.2427	0.053*	
H25B	0.1401	0.3086	0.2465	0.053*	
C26	0.3225 (4)	0.0914 (4)	0.20559 (11)	0.0498 (11)	
H26A	0.2571	0.0285	0.2010	0.060*	
H26B	0.3676	0.0673	0.2286	0.060*	
C27	0.4123 (4)	0.0936 (4)	0.17252 (11)	0.0532 (11)	
H27A	0.4812	0.1516	0.1782	0.064*	
H27B	0.4480	0.0092	0.1692	0.064*	
C28	0.2864 (4)	0.2552 (4)	0.14174 (12)	0.0530 (11)	
H28A	0.2398	0.2750	0.1186	0.064*	
H28B	0.3486	0.3219	0.1457	0.064*	
C29	0.1965 (4)	0.2530 (4)	0.17514 (11)	0.0532 (11)	
H29A	0.1579	0.3363	0.1781	0.064*	
H29B	0.1297	0.1918	0.1703	0.064*	
C30	0.4274 (4)	0.1209 (4)	0.10424 (12)	0.0562 (11)	
C31	0.5111 (5)	0.0189 (5)	0.10076 (15)	0.0978 (19)	
H31A	0.5173	−0.0409	0.1203	0.117*	
C32	0.5852 (6)	0.0065 (7)	0.0684 (2)	0.126 (2)	
H32A	0.6419	−0.0610	0.0669	0.152*	
C33	0.5778 (6)	0.0897 (7)	0.03866 (17)	0.104 (2)	
H33A	0.6267	0.0794	0.0168	0.125*	
C34	0.4960 (5)	0.1886 (6)	0.04236 (15)	0.0806 (15)	
C35	0.4210 (4)	0.2047 (5)	0.07409 (13)	0.0644 (13)	
H35A	0.3653	0.2731	0.0752	0.077*	
C36	0.4935 (10)	0.2888 (9)	0.0119 (2)	0.124 (3)	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0841 (10)	0.1419 (15)	0.0576 (9)	0.0016 (9)	0.0215 (7)	−0.0117 (9)
S2	0.0848 (10)	0.1166 (12)	0.0520 (8)	0.0144 (8)	0.0242 (7)	−0.0045 (8)
Cl1	0.0477 (6)	0.0448 (7)	0.0672 (8)	0.0020 (5)	0.0027 (5)	0.0029 (5)
Cl2	0.0492 (6)	0.0460 (7)	0.0749 (8)	−0.0021 (5)	0.0017 (5)	−0.0066 (6)
F4	0.144 (7)	0.195 (8)	0.142 (8)	−0.050 (6)	0.040 (6)	0.054 (6)
F5	0.176 (10)	0.189 (7)	0.097 (6)	−0.023 (8)	−0.038 (7)	0.022 (5)
F6	0.173 (9)	0.129 (6)	0.108 (6)	0.029 (6)	0.059 (6)	0.047 (5)
F4'	0.167 (10)	0.201 (11)	0.111 (9)	−0.018 (8)	0.069 (8)	0.027 (7)
F5'	0.152 (10)	0.177 (10)	0.127 (9)	0.019 (8)	−0.020 (8)	0.025 (7)
F6'	0.169 (11)	0.117 (8)	0.100 (7)	−0.012 (8)	0.004 (8)	0.035 (6)
O1	0.0550 (18)	0.066 (2)	0.075 (2)	0.0076 (16)	0.0098 (16)	−0.0073 (17)
O2	0.0543 (18)	0.064 (2)	0.066 (2)	0.0113 (16)	0.0070 (15)	−0.0064 (16)
N1	0.0398 (19)	0.039 (2)	0.053 (2)	−0.0027 (17)	0.0023 (16)	0.0015 (17)
N2	0.056 (2)	0.057 (2)	0.053 (2)	0.0002 (18)	0.0019 (18)	−0.0038 (19)
N3	0.0378 (18)	0.039 (2)	0.042 (2)	0.0011 (16)	0.0020 (15)	0.0046 (15)
N4	0.059 (2)	0.050 (2)	0.038 (2)	0.0023 (17)	0.0003 (17)	0.0005 (16)
C1	0.119 (5)	0.144 (6)	0.037 (3)	−0.031 (4)	0.010 (3)	0.002 (3)
C2	0.087 (4)	0.086 (4)	0.061 (4)	−0.018 (3)	−0.008 (3)	0.005 (3)
C3	0.063 (3)	0.055 (3)	0.049 (3)	−0.013 (2)	0.007 (2)	0.000 (2)
C4	0.049 (3)	0.064 (3)	0.048 (3)	−0.014 (2)	0.007 (2)	−0.009 (2)
C5	0.043 (2)	0.048 (3)	0.057 (3)	−0.012 (2)	0.005 (2)	−0.010 (2)
C6	0.046 (2)	0.063 (3)	0.054 (3)	0.000 (2)	0.009 (2)	0.001 (2)
C7	0.040 (2)	0.050 (3)	0.057 (3)	0.0002 (19)	0.002 (2)	0.004 (2)
C8	0.060 (3)	0.038 (3)	0.066 (3)	0.005 (2)	0.006 (2)	0.002 (2)
C9	0.063 (3)	0.047 (3)	0.065 (3)	0.009 (2)	0.007 (2)	0.006 (2)
C10	0.066 (3)	0.070 (3)	0.045 (3)	0.010 (2)	−0.002 (2)	0.011 (2)
C11	0.055 (2)	0.062 (3)	0.048 (3)	0.010 (2)	−0.006 (2)	0.011 (2)
C12	0.057 (3)	0.075 (3)	0.062 (3)	−0.012 (2)	0.006 (2)	−0.015 (3)
C13	0.072 (3)	0.122 (4)	0.056 (3)	−0.004 (3)	0.008 (3)	0.001 (3)
C14	0.094 (4)	0.153 (6)	0.061 (4)	−0.004 (4)	0.009 (3)	−0.003 (4)
C15	0.125 (5)	0.179 (7)	0.088 (5)	0.010 (5)	0.029 (4)	−0.033 (5)
C16	0.117 (5)	0.161 (6)	0.120 (6)	0.040 (4)	0.031 (5)	−0.021 (5)
C17	0.116 (4)	0.119 (5)	0.088 (4)	0.039 (4)	0.023 (3)	−0.013 (4)
C18	0.120 (6)	0.194 (8)	0.142 (7)	0.012 (6)	0.045 (6)	−0.013 (6)
F1	0.210 (12)	0.231 (13)	0.194 (12)	−0.015 (9)	−0.021 (8)	0.046 (9)
F2	0.255 (12)	0.207 (11)	0.205 (12)	0.011 (9)	0.029 (9)	−0.011 (9)
F3	0.234 (10)	0.224 (10)	0.114 (8)	−0.061 (9)	0.019 (8)	0.005 (7)
F1'	0.169 (6)	0.177 (7)	0.070 (4)	−0.007 (6)	0.020 (4)	0.049 (4)
F2'	0.253 (8)	0.299 (10)	0.141 (7)	0.016 (8)	0.117 (6)	0.045 (7)
F3'	0.177 (6)	0.250 (8)	0.126 (6)	0.073 (6)	−0.054 (5)	−0.071 (5)
C19	0.111 (4)	0.099 (4)	0.040 (3)	−0.001 (3)	0.001 (3)	0.003 (3)
C20	0.078 (3)	0.069 (3)	0.054 (3)	0.001 (3)	−0.006 (3)	0.000 (3)
C21	0.057 (3)	0.052 (3)	0.045 (3)	−0.010 (2)	0.006 (2)	−0.004 (2)
C22	0.048 (2)	0.050 (3)	0.045 (3)	−0.003 (2)	0.0085 (19)	−0.004 (2)

C23	0.044 (2)	0.042 (3)	0.052 (3)	−0.006 (2)	0.004 (2)	−0.004 (2)
C24	0.043 (2)	0.066 (3)	0.042 (3)	0.004 (2)	0.0051 (19)	0.005 (2)
C25	0.041 (2)	0.045 (3)	0.047 (3)	0.0006 (18)	0.0030 (19)	0.0085 (19)
C26	0.062 (3)	0.045 (3)	0.042 (3)	0.008 (2)	0.003 (2)	0.009 (2)
C27	0.061 (3)	0.050 (3)	0.048 (3)	0.013 (2)	0.002 (2)	0.002 (2)
C28	0.056 (3)	0.056 (3)	0.047 (3)	0.008 (2)	−0.001 (2)	0.010 (2)
C29	0.048 (2)	0.062 (3)	0.050 (3)	0.008 (2)	−0.006 (2)	0.011 (2)
C30	0.067 (3)	0.056 (3)	0.046 (3)	−0.004 (2)	0.004 (2)	−0.008 (2)
C31	0.140 (5)	0.089 (4)	0.064 (4)	0.036 (4)	0.034 (3)	0.004 (3)
C32	0.147 (6)	0.146 (7)	0.086 (5)	0.052 (5)	0.033 (4)	−0.014 (5)
C33	0.118 (5)	0.131 (6)	0.063 (4)	0.009 (4)	0.037 (4)	−0.013 (4)
C34	0.093 (4)	0.095 (4)	0.053 (4)	−0.012 (3)	0.019 (3)	−0.003 (3)
C35	0.072 (3)	0.074 (3)	0.047 (3)	−0.002 (2)	0.007 (2)	0.003 (3)
C36	0.169 (9)	0.149 (8)	0.054 (5)	−0.003 (7)	0.031 (5)	−0.002 (5)

Geometric parameters (Å, °)

S1—C1	1.683 (6)	C12—C17	1.387 (6)
S1—C4	1.716 (4)	C13—C14	1.367 (7)
S2—C19	1.692 (5)	C13—H13A	0.9300
S2—C22	1.707 (4)	C14—C15	1.363 (8)
F4—C36	1.397 (14)	C14—C18	1.505 (10)
F5—C36	1.178 (13)	C15—C16	1.363 (9)
F6—C36	1.363 (13)	C15—H15A	0.9300
F4'—C36	1.303 (15)	C16—C17	1.389 (8)
F5'—C36	1.58 (2)	C16—H16A	0.9300
F6'—C36	1.234 (17)	C17—H17A	0.9300
O1—C5	1.218 (4)	C18—F1	1.296 (9)
O2—C23	1.228 (4)	C18—F2'	1.298 (8)
N1—C8	1.484 (5)	C18—F3	1.340 (9)
N1—C11	1.488 (5)	C18—F1'	1.368 (8)
N1—C7	1.499 (5)	C18—F3'	1.413 (8)
N1—H1A	0.93 (3)	C18—F2	1.448 (9)
N2—C12	1.416 (5)	C19—C20	1.339 (6)
N2—C10	1.452 (5)	C19—H19	0.9300
N2—C9	1.456 (5)	C20—C21	1.418 (6)
N3—C26	1.482 (5)	C20—H20	0.9300
N3—C29	1.486 (5)	C21—C22	1.367 (5)
N3—C25	1.499 (4)	C21—H21	0.9300
N3—H3A	0.94 (3)	C22—C23	1.458 (5)
N4—C30	1.403 (5)	C23—C24	1.498 (5)
N4—C27	1.461 (5)	C24—C25	1.512 (5)
N4—C28	1.465 (5)	C24—H24A	0.9700
C1—C2	1.358 (6)	C24—H24B	0.9700
C1—H1	0.9300	C25—H25A	0.9700
C2—C3	1.401 (6)	C25—H25B	0.9700
C2—H2	0.9300	C26—C27	1.497 (5)
C3—C4	1.369 (5)	C26—H26A	0.9700
C3—H3	0.9300	C26—H26B	0.9700

C4—C5	1.456 (6)	C27—H27A	0.9700
C5—C6	1.508 (5)	C27—H27B	0.9700
C6—C7	1.505 (5)	C28—C29	1.507 (5)
C6—H6A	0.9700	C28—H28A	0.9700
C6—H6B	0.9700	C28—H28B	0.9700
C7—H7A	0.9700	C29—H29A	0.9700
C7—H7B	0.9700	C29—H29B	0.9700
C8—C9	1.513 (5)	C30—C35	1.375 (6)
C8—H8A	0.9700	C30—C31	1.395 (6)
C8—H8B	0.9700	C31—C32	1.384 (7)
C9—H9A	0.9700	C31—H31A	0.9300
C9—H9B	0.9700	C32—C33	1.359 (8)
C10—C11	1.504 (5)	C32—H32A	0.9300
C10—H10A	0.9700	C33—C34	1.357 (7)
C10—H10B	0.9700	C33—H33A	0.9300
C11—H11A	0.9700	C34—C35	1.375 (6)
C11—H11B	0.9700	C34—C36	1.498 (9)
C12—C13	1.376 (6)	C35—H35A	0.9300
C1—S1—C4	91.7 (3)	F2'—C18—F2	41.3 (7)
C19—S2—C22	91.3 (2)	F3—C18—F2	100.7 (9)
C8—N1—C11	108.3 (3)	F1'—C18—F2	74.4 (8)
C8—N1—C7	113.5 (3)	F3'—C18—F2	147.5 (11)
C11—N1—C7	111.8 (3)	F1—C18—C14	117.4 (9)
C8—N1—H1A	112 (2)	F2'—C18—C14	115.0 (8)
C11—N1—H1A	107 (2)	F3—C18—C14	110.2 (8)
C7—N1—H1A	104 (2)	F1'—C18—C14	112.1 (7)
C12—N2—C10	116.7 (4)	F3'—C18—C14	105.2 (7)
C12—N2—C9	115.8 (4)	F2—C18—C14	103.7 (8)
C10—N2—C9	111.5 (3)	C20—C19—S2	114.0 (4)
C26—N3—C29	108.3 (3)	C20—C19—H19	123.0
C26—N3—C25	113.3 (3)	S2—C19—H19	123.0
C29—N3—C25	112.0 (3)	C19—C20—C21	110.7 (4)
C26—N3—H3A	107 (3)	C19—C20—H20	124.6
C29—N3—H3A	111 (3)	C21—C20—H20	124.6
C25—N3—H3A	105 (3)	C22—C21—C20	113.2 (4)
C30—N4—C27	114.6 (3)	C22—C21—H21	123.4
C30—N4—C28	116.3 (3)	C20—C21—H21	123.4
C27—N4—C28	110.9 (3)	C21—C22—C23	129.1 (4)
C2—C1—S1	112.9 (4)	C21—C22—S2	110.8 (3)
C2—C1—H1	123.5	C23—C22—S2	120.1 (3)
S1—C1—H1	123.5	O2—C23—C22	120.8 (4)
C1—C2—C3	111.7 (5)	O2—C23—C24	121.1 (4)
C1—C2—H2	124.2	C22—C23—C24	118.1 (4)
C3—C2—H2	124.2	C23—C24—C25	113.0 (3)
C4—C3—C2	113.1 (4)	C23—C24—H24A	109.0
C4—C3—H3	123.5	C25—C24—H24A	109.0
C2—C3—H3	123.5	C23—C24—H24B	109.0
C3—C4—C5	130.5 (4)	C25—C24—H24B	109.0
C3—C4—S1	110.6 (3)	H24A—C24—H24B	107.8

C5—C4—S1	118.9 (3)	N3—C25—C24	111.0 (3)
O1—C5—C4	121.4 (4)	N3—C25—H25A	109.4
O1—C5—C6	121.9 (4)	C24—C25—H25A	109.4
C4—C5—C6	116.7 (4)	N3—C25—H25B	109.4
C7—C6—C5	111.7 (3)	C24—C25—H25B	109.4
C7—C6—H6A	109.3	H25A—C25—H25B	108.0
C5—C6—H6A	109.3	N3—C26—C27	110.5 (3)
C7—C6—H6B	109.3	N3—C26—H26A	109.5
C5—C6—H6B	109.3	C27—C26—H26A	109.5
H6A—C6—H6B	107.9	N3—C26—H26B	109.5
N1—C7—C6	110.8 (3)	C27—C26—H26B	109.5
N1—C7—H7A	109.5	H26A—C26—H26B	108.1
C6—C7—H7A	109.5	N4—C27—C26	112.5 (3)
N1—C7—H7B	109.5	N4—C27—H27A	109.1
C6—C7—H7B	109.5	C26—C27—H27A	109.1
H7A—C7—H7B	108.1	N4—C27—H27B	109.1
N1—C8—C9	110.7 (3)	C26—C27—H27B	109.1
N1—C8—H8A	109.5	H27A—C27—H27B	107.8
C9—C8—H8A	109.5	N4—C28—C29	111.9 (3)
N1—C8—H8B	109.5	N4—C28—H28A	109.2
C9—C8—H8B	109.5	C29—C28—H28A	109.2
H8A—C8—H8B	108.1	N4—C28—H28B	109.2
N2—C9—C8	112.2 (3)	C29—C28—H28B	109.2
N2—C9—H9A	109.2	H28A—C28—H28B	107.9
C8—C9—H9A	109.2	N3—C29—C28	110.8 (3)
N2—C9—H9B	109.2	N3—C29—H29A	109.5
C8—C9—H9B	109.2	C28—C29—H29A	109.5
H9A—C9—H9B	107.9	N3—C29—H29B	109.5
N2—C10—C11	112.0 (3)	C28—C29—H29B	109.5
N2—C10—H10A	109.2	H29A—C29—H29B	108.1
C11—C10—H10A	109.2	C35—C30—C31	117.1 (4)
N2—C10—H10B	109.2	C35—C30—N4	122.6 (4)
C11—C10—H10B	109.2	C31—C30—N4	120.3 (4)
H10A—C10—H10B	107.9	C32—C31—C30	120.2 (5)
N1—C11—C10	110.7 (3)	C32—C31—H31A	119.9
N1—C11—H11A	109.5	C30—C31—H31A	119.9
C10—C11—H11A	109.5	C33—C32—C31	122.2 (6)
N1—C11—H11B	109.5	C33—C32—H32A	118.9
C10—C11—H11B	109.5	C31—C32—H32A	118.9
H11A—C11—H11B	108.1	C34—C33—C32	117.1 (5)
C13—C12—C17	117.3 (5)	C34—C33—H33A	121.5
C13—C12—N2	120.8 (5)	C32—C33—H33A	121.5
C17—C12—N2	121.8 (5)	C33—C34—C35	122.6 (5)
C14—C13—C12	121.3 (6)	C33—C34—C36	118.7 (6)
C14—C13—H13A	119.4	C35—C34—C36	118.5 (6)
C12—C13—H13A	119.4	C30—C35—C34	120.8 (5)
C15—C14—C13	121.1 (7)	C30—C35—H35A	119.6
C15—C14—C18	118.7 (6)	C34—C35—H35A	119.6
C13—C14—C18	120.2 (6)	F5—C36—F6'	126.5 (13)

C14—C15—C16	119.2 (7)	F5—C36—F4'	61.7 (9)
C14—C15—H15A	120.4	F6'—C36—F4'	117.1 (15)
C16—C15—H15A	120.4	F5—C36—F6	96.4 (12)
C15—C16—C17	120.0 (7)	F6'—C36—F6	41.0 (7)
C15—C16—H16A	120.0	F4'—C36—F6	131.8 (10)
C17—C16—H16A	120.0	F5—C36—F4	102.9 (9)
C12—C17—C16	121.0 (6)	F6'—C36—F4	74.2 (12)
C12—C17—H17A	119.5	F4'—C36—F4	47.5 (9)
C16—C17—H17A	119.5	F6—C36—F4	108.0 (11)
F1—C18—F2'	123.1 (13)	F5—C36—C34	118.6 (11)
F1—C18—F3	117.5 (11)	F6'—C36—C34	111.1 (9)
F2'—C18—F3	59.5 (7)	F4'—C36—C34	111.0 (10)
F1—C18—F1'	33.2 (9)	F6—C36—C34	117.1 (7)
F2'—C18—F1'	105.7 (8)	F4—C36—C34	111.9 (8)
F3—C18—F1'	137.3 (11)	F5—C36—F5'	30.2 (10)
F1—C18—F3'	74.4 (9)	F6'—C36—F5'	123.2 (15)
F2'—C18—F3'	111.4 (10)	F4'—C36—F5'	90.5 (14)
F3—C18—F3'	55.3 (7)	F6—C36—F5'	83.0 (12)
F1'—C18—F3'	107.3 (8)	F4—C36—F5'	133.0 (10)
F1—C18—F2	104.5 (10)	C34—C36—F5'	101.7 (10)
C4—S1—C1—C2	−1.0 (4)	C22—S2—C19—C20	0.5 (4)
S1—C1—C2—C3	1.1 (6)	S2—C19—C20—C21	−0.2 (6)
C1—C2—C3—C4	−0.7 (6)	C19—C20—C21—C22	−0.3 (6)
C2—C3—C4—C5	179.4 (4)	C20—C21—C22—C23	179.5 (4)
C2—C3—C4—S1	−0.1 (5)	C20—C21—C22—S2	0.6 (4)
C1—S1—C4—C3	0.6 (4)	C19—S2—C22—C21	−0.6 (3)
C1—S1—C4—C5	−178.9 (4)	C19—S2—C22—C23	−179.6 (3)
C3—C4—C5—O1	175.2 (4)	C21—C22—C23—O2	−175.4 (4)
S1—C4—C5—O1	−5.4 (5)	S2—C22—C23—O2	3.3 (5)
C3—C4—C5—C6	−3.2 (6)	C21—C22—C23—C24	2.1 (6)
S1—C4—C5—C6	176.3 (3)	S2—C22—C23—C24	−179.2 (3)
O1—C5—C6—C7	21.4 (6)	O2—C23—C24—C25	−21.7 (5)
C4—C5—C6—C7	−160.3 (3)	C22—C23—C24—C25	160.8 (3)
C8—N1—C7—C6	−63.1 (4)	C26—N3—C25—C24	61.9 (4)
C11—N1—C7—C6	174.0 (3)	C29—N3—C25—C24	−175.3 (3)
C5—C6—C7—N1	−171.9 (3)	C23—C24—C25—N3	174.6 (3)
C11—N1—C8—C9	−57.8 (4)	C29—N3—C26—C27	58.7 (4)
C7—N1—C8—C9	177.5 (3)	C25—N3—C26—C27	−176.4 (3)
C12—N2—C9—C8	170.1 (3)	C30—N4—C27—C26	−172.1 (3)
C10—N2—C9—C8	−53.4 (5)	C28—N4—C27—C26	53.7 (4)
N1—C8—C9—N2	56.2 (5)	N3—C26—C27—N4	−57.4 (4)
C12—N2—C10—C11	−169.9 (3)	C30—N4—C28—C29	173.5 (3)
C9—N2—C10—C11	54.0 (5)	C27—N4—C28—C29	−53.1 (4)
C8—N1—C11—C10	58.5 (4)	C26—N3—C29—C28	−58.7 (4)
C7—N1—C11—C10	−175.8 (3)	C25—N3—C29—C28	175.7 (3)
N2—C10—C11—N1	−57.4 (5)	N4—C28—C29—N3	56.8 (4)
C10—N2—C12—C13	30.9 (6)	C27—N4—C30—C35	−145.6 (4)
C9—N2—C12—C13	165.2 (4)	C28—N4—C30—C35	−14.0 (6)
C10—N2—C12—C17	−149.8 (5)	C27—N4—C30—C31	36.2 (6)

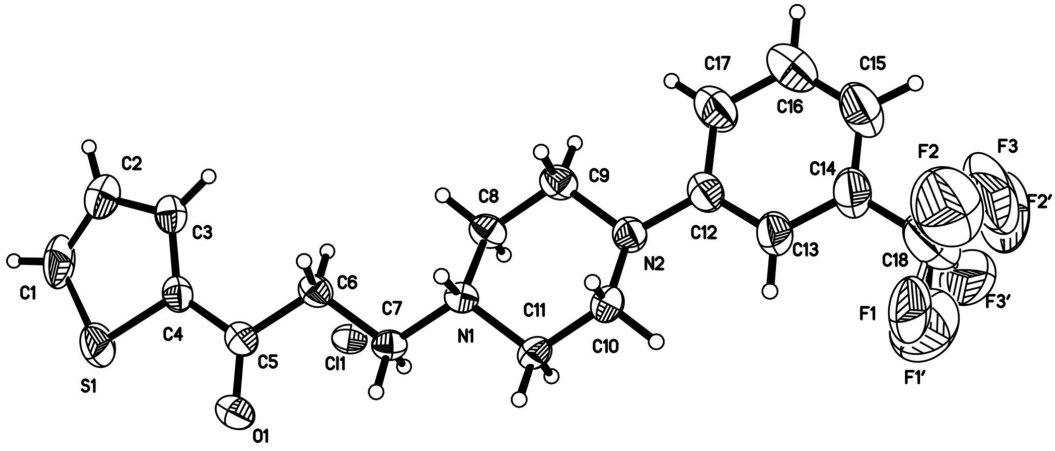
C9—N2—C12—C17	−15.5 (6)	C28—N4—C30—C31	167.8 (4)
C17—C12—C13—C14	−1.2 (8)	C35—C30—C31—C32	1.2 (8)
N2—C12—C13—C14	178.1 (5)	N4—C30—C31—C32	179.5 (5)
C12—C13—C14—C15	−0.9 (9)	C30—C31—C32—C33	−1.5 (11)
C12—C13—C14—C18	−179.6 (5)	C31—C32—C33—C34	1.5 (11)
C13—C14—C15—C16	3.4 (11)	C32—C33—C34—C35	−1.3 (9)
C18—C14—C15—C16	−177.9 (7)	C32—C33—C34—C36	174.6 (7)
C14—C15—C16—C17	−3.8 (12)	C31—C30—C35—C34	−1.0 (7)
C13—C12—C17—C16	0.7 (9)	N4—C30—C35—C34	−179.3 (4)
N2—C12—C17—C16	−178.6 (5)	C33—C34—C35—C30	1.1 (8)
C15—C16—C17—C12	1.8 (11)	C36—C34—C35—C30	−174.8 (6)
C15—C14—C18—F1	−170.6 (11)	C33—C34—C36—F5	71.9 (16)
C13—C14—C18—F1	8.2 (13)	C35—C34—C36—F5	−112.1 (14)
C15—C14—C18—F2'	32.4 (12)	C33—C34—C36—F6'	−128.6 (14)
C13—C14—C18—F2'	−148.8 (9)	C35—C34—C36—F6'	47.5 (17)
C15—C14—C18—F3	−32.4 (11)	C33—C34—C36—F4'	4(2)
C13—C14—C18—F3	146.4 (9)	C35—C34—C36—F4'	179.6 (17)
C15—C14—C18—F1'	153.2 (8)	C33—C34—C36—F6	−173.2 (12)
C13—C14—C18—F1'	−28.0 (9)	C35—C34—C36—F6	2.9 (15)
C15—C14—C18—F3'	−90.5 (8)	C33—C34—C36—F4	−47.8 (14)
C13—C14—C18—F3'	88.3 (8)	C35—C34—C36—F4	128.3 (11)
C15—C14—C18—F2	74.7 (10)	C33—C34—C36—F5'	98.7 (10)
C13—C14—C18—F2	−106.5 (9)	C35—C34—C36—F5'	−85.3 (9)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3A···Cl2	0.94 (3)	2.10 (3)	3.025 (3)	168 (4)
N1—H1A···Cl1^{i}	0.93 (3)	2.11 (3)	3.033 (3)	174 (3)

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

Fig. 1


C17
C15
C16
C2
C9
C14
F2
F3
C8
C12
F2'
C3
N2
C13
C18
C1
C6
C7
C11
F1
F3'
C4
N1
C10
C5
Cl1
S1
F1'
O1