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1,4-Dibenzyl-1,4-diazoniabicyclo[2.2.2]-octane bis(trifluoromethanesulfonate)

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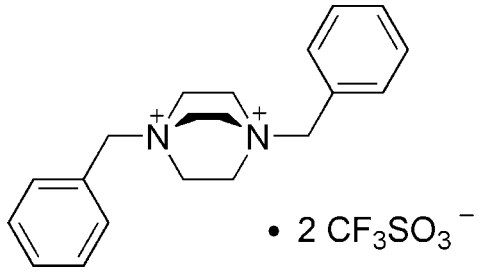
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.009 Å; disorder in solvent or counterion; R factor = 0.059; wR factor = 0.173; data-to-parameter ratio = 11.4.

In the title compound, $C_{20}H_{26}N_2^{2+}\cdot 2CF_3SO_3^-$, the dihedral angle between the phenyl rings of the dication is 9.4 (3)°. The $-CF_3$ groups of both anions are disordered over two sets of positions in ratios of 0.920 (10):0.080 (10) and 0.76 (3):0.24 (3). In the crystal structure, C—H···O interactions link the molecules into columns.

Related literature

For ammonium cations containing a 1,4-diazabicyclo[2.2.2]-octane core, see: Chantrapromma *et al.* (2002); Mansikkamäki *et al.* (2005).



Experimental

Crystal data

$C_{20}H_{26}N_2^{2+}\cdot 2CF_3O_3S^-$
M_r = 592.57
Orthorhombic, *Pbca*
a = 13.759 (3) Å
b = 14.334 (3) Å
c = 26.195 (6) Å
V = 5166 (2) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.29 mm^{-1}
T = 294 (2) K
0.18 × 0.16 × 0.10 mm

Data collection

Bruker SMART 1K CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.950, T_{max} = 0.972
24969 measured reflections
4560 independent reflections
1952 reflections with $I > 2\sigma(I)$
R_{int} = 0.149

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.173
S = 1.00
4560 reflections
399 parameters
144 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7A···O6^{i}	0.97	2.54	3.380 (6)	145
C9—H9A···O1^{ii}	0.97	2.56	3.416 (6)	148
C9—H9B···O2^{iii}	0.97	2.38	3.227 (6)	146
C10—H10B···O1^{iii}	0.97	2.43	3.347 (6)	156
C11—H11A···O4^{iii}	0.97	2.38	3.255 (6)	150
C11—H11B···O6	0.97	2.37	3.191 (5)	142
C12—H12B···O5	0.97	2.38	3.287 (6)	155
C14—H14A···O1^{ii}	0.97	2.50	3.357 (7)	148
C14—H14B···O4^{iii}	0.97	2.54	3.394 (7)	147

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

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: HB2583).

References

Bruker (1997). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
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supplementary materials

Acta Cryst. (2007). E**63**, o4464 [**doi:10.1107/S1600536807052063**]

1,4-Dibenzyl-1,4-diazoniabicyclo[2.2.2]octane bis(trifluoromethanesulfonate)

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Comment

In the dication of the title compound, (I), (Fig. 1), the dihedral angle between the terminal phenyl rings is 9.4 (3)°, indicating a *trans* conformation.

A network of C—H···O interactions (Table 1) between cation and anions link the molecules into columns. The adjacent columns are stabilized by weak aromatic interactions.

For related structures, see: Chantrapromma *et al.* (2002); Mansikkamäki *et al.* (2005).

Experimental

The title compound was obtained, in a 75% yield, from counterion exchange of 1,4-dibenzyl-1,4-diazoniabicyclo[2.2.2]octane dibromide (4.52 mg, 10 mmol) with silver trifluoromethanesulfonate (2.83 mg, 11 mmol) in methanol/acetonitrile (10 ml, *v*/*v* 1:4). Colourless blocks of (I) were grown by natural evaporation of a CH_3CN/CH_2Cl_2 (*v*/*v* 1:1) solution.

Refinement

All H atoms were positioned geometrically (C—H = 0.93 and 0.97 Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$. The $–CF_3$ groups of the two trifluoromethanesulfonate anions were found to be disordered. The site occupancies of one trilate ion was refined to 0.920 (10):0.080 (10) and that of the other to 0.76 (3):0.24 (3).

Figures

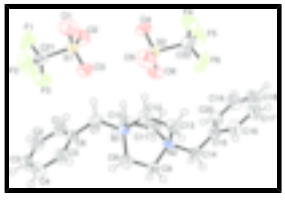


Fig. 1. The molecular structure of (I) with displacement ellipsoids drawn at the 50% probability level (arbitrary spheres for the H atoms). Only one orientation of each trifluoromethanesulfonate ion is shown.

1,4-Dibenzyl-1,4-diazoniabicyclo[2.2.2]octane bis(trifluoromethanesulfonate)

Crystal data

$C_{20}H_{26}N_2^{2+}\cdot 2CF_3O_3S^-$	F_{000} = 2448
M_r = 592.57	D_x = 1.524 Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation

	λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 1753 reflections
a = 13.759 (3) Å	θ = 2.6–19.6º
b = 14.334 (3) Å	μ = 0.29 mm^{-1}
c = 26.195 (6) Å	T = 294 (2) K
V = 5166 (2) $Å^3$	Block, colourless
Z = 8	0.18 × 0.16 × 0.10 mm

Data collection

Bruker SMART 1K CCD diffractometer	4560 independent reflections
Radiation source: fine-focus sealed tube	1952 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.149
T = 294(2) K	θ_{max} = 25.0º
ω scans	θ_{min} = 1.6º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −16→16
T_{min} = 0.950, T_{max} = 0.972	k = −12→17
24969 measured reflections	l = −31→23

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.059	H-atom parameters constrained
$wR(F^2)$ = 0.173	$w = 1/[\sigma^2(F_o^2) + (0.07P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.01	$(\Delta/\sigma)_{max}$ = 0.002
4560 reflections	$\Delta\rho_{max}$ = 0.29 e $Å^{-3}$
399 parameters	$\Delta\rho_{min}$ = −0.27 e $Å^{-3}$
144 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
N1	0.5039 (3)	0.1698 (2)	0.28420 (15)	0.0310 (10)	
N2	0.5071 (3)	0.0692 (2)	0.20271 (15)	0.0305 (10)	
C1	0.3890 (4)	0.1821 (3)	0.3991 (2)	0.0489 (15)	
H1	0.3366	0.2006	0.3790	0.059*	
C2	0.3725 (5)	0.1458 (4)	0.4476 (3)	0.0670 (19)	
H2	0.3094	0.1403	0.4599	0.080*	
C3	0.4484 (6)	0.1187 (4)	0.4768 (2)	0.070 (2)	
H3	0.4370	0.0941	0.5091	0.084*	
C4	0.5424 (6)	0.1269 (4)	0.4593 (3)	0.070 (2)	
H4	0.5941	0.1082	0.4797	0.084*	
C5	0.5592 (4)	0.1626 (4)	0.4119 (2)	0.0530 (16)	
H5	0.6229	0.1682	0.4003	0.064*	
C6	0.4831 (4)	0.1908 (3)	0.3804 (2)	0.0359 (13)	
C7	0.5038 (4)	0.2349 (3)	0.33045 (19)	0.0399 (14)	
H7A	0.5668	0.2650	0.3325	0.048*	
H7B	0.4558	0.2834	0.3247	0.048*	
C8	0.5599 (4)	0.0811 (3)	0.29332 (19)	0.0405 (14)	
H8A	0.5336	0.0489	0.3228	0.049*	
H8B	0.6274	0.0958	0.3003	0.049*	
C9	0.5532 (4)	0.0187 (3)	0.24675 (19)	0.0410 (14)	
H9A	0.6178	−0.0018	0.2371	0.049*	
H9B	0.5150	−0.0362	0.2550	0.049*	
C10	0.4022 (3)	0.1436 (3)	0.2681 (2)	0.0383 (14)	
H10A	0.3628	0.1995	0.2650	0.046*	
H10B	0.3730	0.1040	0.2939	0.046*	
C11	0.4043 (3)	0.0927 (3)	0.2176 (2)	0.0425 (14)	
H11A	0.3665	0.0357	0.2202	0.051*	
H11B	0.3751	0.1315	0.1914	0.051*	
C12	0.5512 (3)	0.2205 (3)	0.24066 (18)	0.0385 (14)	
H12A	0.6148	0.2425	0.2512	0.046*	
H12B	0.5123	0.2745	0.2316	0.046*	
C13	0.5621 (4)	0.1581 (3)	0.1945 (2)	0.0455 (15)	
H13A	0.5374	0.1897	0.1644	0.055*	
H13B	0.6303	0.1443	0.1889	0.055*	
C14	0.5103 (4)	0.0051 (3)	0.15592 (19)	0.0394 (14)	
H14A	0.5752	−0.0209	0.1532	0.047*	
H14B	0.4658	−0.0464	0.1616	0.047*	
C15	0.4850 (4)	0.0501 (3)	0.1062 (2)	0.0407 (14)	
C16	0.5581 (4)	0.0838 (4)	0.0752 (2)	0.0556 (16)	
H16	0.6223	0.0811	0.0862	0.067*	
C17	0.5370 (6)	0.1213 (4)	0.0284 (3)	0.076 (2)	
H17	0.5870	0.1441	0.0080	0.091*	
C18	0.4437 (7)	0.1253 (4)	0.0115 (3)	0.081 (2)	
H18	0.4297	0.1507	−0.0203	0.097*	
C19	0.3693 (6)	0.0912 (5)	0.0421 (3)	0.080 (2)	

H19	0.3053	0.0940	0.0308	0.096*	
C20	0.3901 (5)	0.0532 (4)	0.0893 (2)	0.0575 (17)	
H20	0.3403	0.0298	0.1095	0.069*	
S1	0.20596 (10)	0.40619 (9)	0.33002 (5)	0.0408 (4)	
F1	0.1019 (5)	0.4374 (3)	0.4105 (2)	0.107 (2)	0.920 (10)
F2	0.2488 (4)	0.3995 (5)	0.42608 (18)	0.130 (3)	0.920 (10)
F3	0.1492 (5)	0.2968 (3)	0.4033 (2)	0.0942 (19)	0.920 (10)
F1'	0.101 (3)	0.323 (3)	0.397 (3)	0.078 (19)	0.080 (10)
F2'	0.170 (4)	0.4667 (15)	0.4217 (13)	0.040 (13)	0.080 (10)
F3'	0.256 (2)	0.339 (3)	0.4140 (7)	0.17 (2)	0.080 (10)
O1	0.2473 (3)	0.4983 (2)	0.33005 (16)	0.0668 (12)	
O2	0.1144 (3)	0.4028 (3)	0.30509 (16)	0.0753 (13)	
O3	0.2700 (4)	0.3325 (3)	0.31787 (18)	0.1060 (18)	
C21	0.1763 (4)	0.3850 (4)	0.3955 (3)	0.0635 (19)	
S2	0.28188 (10)	0.33629 (9)	0.17470 (5)	0.0390 (4)	
F4	0.3532 (11)	0.4358 (5)	0.1020 (4)	0.085 (3)	0.76 (3)
F5	0.2433 (8)	0.3406 (12)	0.0780 (3)	0.100 (3)	0.76 (3)
F6	0.3860 (9)	0.2898 (7)	0.0955 (5)	0.104 (4)	0.76 (3)
F4'	0.4084 (12)	0.3128 (19)	0.1073 (7)	0.064 (8)	0.24 (3)
F5'	0.322 (2)	0.4388 (9)	0.0912 (14)	0.076 (10)	0.24 (3)
F6'	0.268 (2)	0.299 (2)	0.0755 (8)	0.071 (8)	0.24 (3)
O4	0.2075 (3)	0.4040 (3)	0.18113 (17)	0.0884 (16)	
O5	0.3699 (3)	0.3521 (3)	0.20207 (16)	0.0749 (13)	
O6	0.2488 (2)	0.2410 (2)	0.17770 (15)	0.0563 (11)	
C22	0.3177 (4)	0.3502 (4)	0.1094 (2)	0.0621 (18)	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.029 (2)	0.023 (2)	0.041 (3)	−0.0019 (18)	−0.004 (2)	0.007 (2)
N2	0.032 (3)	0.024 (2)	0.036 (3)	−0.0010 (18)	0.002 (2)	0.0068 (19)
C1	0.051 (4)	0.043 (3)	0.053 (4)	0.005 (3)	0.000 (3)	−0.008 (3)
C2	0.084 (5)	0.058 (4)	0.059 (5)	−0.003 (4)	0.030 (4)	−0.002 (4)
C3	0.126 (7)	0.056 (4)	0.028 (4)	0.012 (4)	0.014 (5)	0.000 (3)
C4	0.093 (6)	0.075 (5)	0.042 (5)	0.015 (4)	−0.010 (4)	0.007 (3)
C5	0.049 (4)	0.068 (4)	0.042 (4)	0.005 (3)	−0.007 (3)	0.000 (3)
C6	0.043 (4)	0.027 (3)	0.038 (3)	0.000 (2)	0.003 (3)	0.005 (2)
C7	0.043 (3)	0.030 (3)	0.047 (4)	−0.006 (2)	−0.007 (3)	−0.006 (3)
C8	0.041 (3)	0.035 (3)	0.045 (4)	0.007 (3)	−0.003 (3)	0.016 (3)
C9	0.045 (4)	0.037 (3)	0.042 (3)	0.014 (2)	−0.001 (3)	0.010 (3)
C10	0.024 (3)	0.037 (3)	0.054 (4)	−0.002 (2)	0.003 (3)	0.000 (3)
C11	0.025 (3)	0.042 (3)	0.061 (4)	0.007 (2)	−0.004 (3)	−0.007 (3)
C12	0.043 (3)	0.028 (3)	0.044 (4)	−0.014 (2)	−0.001 (3)	0.007 (3)
C13	0.055 (4)	0.040 (3)	0.041 (4)	−0.020 (3)	0.001 (3)	0.013 (3)
C14	0.045 (4)	0.031 (3)	0.042 (4)	0.004 (2)	−0.002 (3)	−0.005 (3)
C15	0.050 (4)	0.037 (3)	0.035 (3)	−0.004 (3)	−0.003 (3)	−0.005 (3)
C16	0.058 (4)	0.064 (4)	0.045 (4)	−0.008 (3)	0.003 (3)	−0.003 (3)
C17	0.118 (7)	0.062 (5)	0.047 (5)	−0.021 (4)	0.013 (5)	0.003 (4)

C18	0.150 (8)	0.051 (4)	0.040 (5)	0.014 (5)	−0.021 (5)	−0.001 (3)
C19	0.099 (6)	0.080 (5)	0.060 (5)	0.018 (5)	−0.034 (5)	−0.003 (4)
C20	0.057 (5)	0.059 (4)	0.057 (4)	−0.002 (3)	−0.014 (4)	−0.008 (3)
S1	0.0411 (9)	0.0445 (9)	0.0369 (9)	0.0073 (7)	−0.0002 (7)	0.0014 (7)
F1	0.139 (5)	0.086 (3)	0.097 (4)	0.002 (3)	0.069 (4)	−0.015 (3)
F2	0.181 (5)	0.146 (5)	0.064 (3)	−0.049 (4)	−0.062 (3)	0.012 (3)
F3	0.132 (5)	0.060 (3)	0.090 (4)	−0.013 (3)	0.032 (3)	0.023 (3)
F1'	0.08 (2)	0.07 (2)	0.08 (2)	−0.004 (10)	0.007 (10)	0.005 (10)
F2'	0.045 (16)	0.041 (15)	0.035 (15)	0.002 (10)	−0.003 (10)	−0.005 (9)
F3'	0.17 (2)	0.17 (2)	0.17 (2)	0.0001 (11)	−0.0001 (11)	0.0000 (11)
O1	0.054 (3)	0.063 (3)	0.084 (3)	−0.021 (2)	−0.002 (2)	0.014 (2)
O2	0.070 (3)	0.074 (3)	0.081 (3)	−0.010 (2)	−0.043 (3)	−0.001 (2)
O3	0.138 (4)	0.103 (3)	0.078 (3)	0.087 (3)	0.044 (3)	0.023 (3)
C21	0.070 (5)	0.047 (4)	0.073 (5)	−0.004 (3)	0.014 (4)	−0.011 (4)
S2	0.0394 (9)	0.0416 (8)	0.0360 (8)	0.0076 (7)	0.0030 (7)	−0.0011 (7)
F4	0.110 (6)	0.062 (4)	0.083 (5)	−0.016 (3)	0.032 (5)	0.021 (3)
F5	0.149 (6)	0.096 (7)	0.056 (4)	−0.002 (5)	−0.032 (4)	0.001 (4)
F6	0.134 (6)	0.081 (5)	0.099 (5)	0.005 (5)	0.066 (5)	−0.033 (4)
F4'	0.043 (10)	0.074 (12)	0.074 (11)	−0.001 (7)	0.012 (7)	−0.018 (8)
F5'	0.073 (12)	0.072 (12)	0.082 (12)	0.006 (7)	0.009 (8)	0.029 (8)
F6'	0.091 (12)	0.074 (11)	0.047 (9)	−0.004 (8)	−0.002 (7)	−0.010 (8)
O4	0.101 (4)	0.083 (3)	0.081 (3)	0.061 (3)	0.042 (3)	0.023 (3)
O5	0.065 (3)	0.081 (3)	0.079 (3)	−0.019 (2)	−0.038 (3)	−0.003 (2)
O6	0.051 (2)	0.045 (2)	0.073 (3)	−0.0096 (18)	−0.002 (2)	0.012 (2)
C22	0.069 (5)	0.052 (5)	0.065 (5)	−0.002 (4)	0.017 (4)	−0.007 (4)

Geometric parameters (Å, °)

N1—C12	1.501 (6)	C13—H13B	0.9700
N1—C8	1.506 (6)	C14—C15	1.494 (7)
N1—C10	1.509 (5)	C14—H14A	0.9700
N1—C7	1.529 (6)	C14—H14B	0.9700
N2—C13	1.497 (5)	C15—C16	1.380 (7)
N2—C9	1.503 (6)	C15—C20	1.380 (7)
N2—C11	1.506 (5)	C16—C17	1.371 (8)
N2—C14	1.533 (6)	C16—H16	0.9300
C1—C6	1.389 (7)	C17—C18	1.360 (9)
C1—C2	1.391 (8)	C17—H17	0.9300
C1—H1	0.9300	C18—C19	1.389 (9)
C2—C3	1.352 (8)	C18—H18	0.9300
C2—H2	0.9300	C19—C20	1.379 (8)
C3—C4	1.377 (9)	C19—H19	0.9300
C3—H3	0.9300	C20—H20	0.9300
C4—C5	1.363 (8)	S1—O3	1.411 (4)
C4—H4	0.9300	S1—O2	1.420 (4)
C5—C6	1.393 (7)	S1—O1	1.438 (4)
C5—H5	0.9300	S1—C21	1.788 (7)
C6—C7	1.482 (7)	F1—C21	1.330 (6)
C7—H7A	0.9700	F2—C21	1.296 (6)

C7—H7B	0.9700	F3—C21	1.334 (6)
C8—C9	1.516 (7)	F1'—C21	1.367 (11)
C8—H8A	0.9700	F2'—C21	1.359 (10)
C8—H8B	0.9700	F3'—C21	1.370 (10)
C9—H9A	0.9700	S2—O4	1.421 (4)
C9—H9B	0.9700	S2—O5	1.425 (4)
C10—C11	1.513 (7)	S2—O6	1.442 (3)
C10—H10A	0.9700	S2—C22	1.791 (6)
C10—H10B	0.9700	F4—C22	1.334 (7)
C11—H11A	0.9700	F5—C22	1.321 (7)
C11—H11B	0.9700	F6—C22	1.329 (7)
C12—C13	1.512 (6)	F4'—C22	1.359 (9)
C12—H12A	0.9700	F5'—C22	1.358 (10)
C12—H12B	0.9700	F6'—C22	1.347 (10)
C13—H13A	0.9700		
C12—N1—C8	107.9 (4)	C15—C14—H14B	108.4
C12—N1—C10	108.1 (4)	N2—C14—H14B	108.4
C8—N1—C10	108.0 (4)	H14A—C14—H14B	107.5
C12—N1—C7	107.9 (3)	C16—C15—C20	119.3 (5)
C8—N1—C7	112.9 (4)	C16—C15—C14	119.6 (5)
C10—N1—C7	111.9 (3)	C20—C15—C14	121.0 (5)
C13—N2—C9	107.9 (4)	C17—C16—C15	120.7 (6)
C13—N2—C11	108.8 (4)	C17—C16—H16	119.7
C9—N2—C11	107.8 (4)	C15—C16—H16	119.7
C13—N2—C14	112.4 (4)	C18—C17—C16	120.5 (7)
C9—N2—C14	108.2 (3)	C18—C17—H17	119.8
C11—N2—C14	111.5 (4)	C16—C17—H17	119.8
C6—C1—C2	120.4 (6)	C17—C18—C19	119.6 (7)
C6—C1—H1	119.8	C17—C18—H18	120.2
C2—C1—H1	119.8	C19—C18—H18	120.2
C3—C2—C1	120.0 (6)	C20—C19—C18	120.2 (7)
C3—C2—H2	120.0	C20—C19—H19	119.9
C1—C2—H2	120.0	C18—C19—H19	119.9
C2—C3—C4	120.7 (6)	C19—C20—C15	119.8 (6)
C2—C3—H3	119.6	C19—C20—H20	120.1
C4—C3—H3	119.6	C15—C20—H20	120.1
C5—C4—C3	119.7 (6)	O3—S1—O2	115.1 (3)
C5—C4—H4	120.1	O3—S1—O1	116.1 (3)
C3—C4—H4	120.1	O2—S1—O1	112.5 (2)
C4—C5—C6	121.3 (6)	O3—S1—C21	103.4 (3)
C4—C5—H5	119.4	O2—S1—C21	103.5 (3)
C6—C5—H5	119.4	O1—S1—C21	104.2 (3)
C1—C6—C5	117.8 (5)	F2—C21—F1	108.5 (6)
C1—C6—C7	121.9 (5)	F2—C21—F3	105.7 (6)
C5—C6—C7	120.1 (5)	F1—C21—F3	105.9 (5)
C6—C7—N1	116.1 (4)	F2—C21—F2'	66.1 (17)
C6—C7—H7A	108.3	F1—C21—F2'	47.0 (19)
N1—C7—H7A	108.3	F3—C21—F2'	136.2 (19)
C6—C7—H7B	108.3	F2—C21—F1'	132 (2)

N1—C7—H7B	108.3	F1—C21—F1'	77 (2)
H7A—C7—H7B	107.4	F3—C21—F1'	34 (2)
N1—C8—C9	109.9 (4)	F2'—C21—F1'	120 (3)
N1—C8—H8A	109.7	F2—C21—F3'	40.4 (18)
C9—C8—H8A	109.7	F1—C21—F3'	141.6 (11)
N1—C8—H8B	109.7	F3—C21—F3'	73.6 (19)
C9—C8—H8B	109.7	F2'—C21—F3'	106.3 (10)
H8A—C8—H8B	108.2	F1'—C21—F3'	106.9 (10)
N2—C9—C8	111.0 (4)	F2—C21—S1	113.0 (5)
N2—C9—H9A	109.4	F1—C21—S1	111.4 (5)
C8—C9—H9A	109.4	F3—C21—S1	111.9 (4)
N2—C9—H9B	109.4	F2'—C21—S1	110.6 (17)
C8—C9—H9B	109.4	F1'—C21—S1	108 (3)
H9A—C9—H9B	108.0	F3'—C21—S1	103.7 (8)
N1—C10—C11	110.3 (4)	O4—S2—O5	116.3 (3)
N1—C10—H10A	109.6	O4—S2—O6	114.4 (3)
C11—C10—H10A	109.6	O5—S2—O6	113.0 (2)
N1—C10—H10B	109.6	O4—S2—C22	103.6 (3)
C11—C10—H10B	109.6	O5—S2—C22	103.2 (3)
H10A—C10—H10B	108.1	O6—S2—C22	104.2 (2)
N2—C11—C10	110.6 (4)	F5—C22—F6	108.1 (7)
N2—C11—H11A	109.5	F5—C22—F4	106.8 (8)
C10—C11—H11A	109.5	F6—C22—F4	107.5 (7)
N2—C11—H11B	109.5	F5—C22—F6'	30.1 (11)
C10—C11—H11B	109.5	F6—C22—F6'	79.8 (12)
H11A—C11—H11B	108.1	F4—C22—F6'	126.7 (13)
N1—C12—C13	111.4 (4)	F5—C22—F5'	84.9 (12)
N1—C12—H12A	109.4	F6—C22—F5'	118.8 (14)
C13—C12—H12A	109.4	F4—C22—F5'	22.1 (13)
N1—C12—H12B	109.4	F6'—C22—F5'	107.7 (10)
C13—C12—H12B	109.4	F5—C22—F4'	130.2 (11)
H12A—C12—H12B	108.0	F6—C22—F4'	23.4 (11)
N2—C13—C12	109.8 (4)	F4—C22—F4'	91.2 (12)
N2—C13—H13A	109.7	F6'—C22—F4'	103.1 (14)
C12—C13—H13A	109.7	F5'—C22—F4'	108.4 (9)
N2—C13—H13B	109.7	F5—C22—S2	111.7 (6)
C12—C13—H13B	109.7	F6—C22—S2	112.6 (6)
H13A—C13—H13B	108.2	F4—C22—S2	110.0 (6)
C15—C14—N2	115.6 (4)	F6'—C22—S2	115.3 (11)
C15—C14—H14A	108.4	F5'—C22—S2	116.9 (15)
N2—C14—H14A	108.4	F4'—C22—S2	104.3 (7)
C6—C1—C2—C3	−0.2 (8)	C16—C17—C18—C19	0.1 (10)
C1—C2—C3—C4	0.4 (9)	C17—C18—C19—C20	−0.2 (10)
C2—C3—C4—C5	−0.2 (9)	C18—C19—C20—C15	0.7 (9)
C3—C4—C5—C6	−0.2 (9)	C16—C15—C20—C19	−1.0 (8)
C2—C1—C6—C5	−0.3 (7)	C14—C15—C20—C19	−176.9 (5)
C2—C1—C6—C7	−175.8 (5)	O3—S1—C21—F2	69.7 (6)
C4—C5—C6—C1	0.5 (8)	O2—S1—C21—F2	−170.0 (5)
C4—C5—C6—C7	176.1 (5)	O1—S1—C21—F2	−52.2 (5)

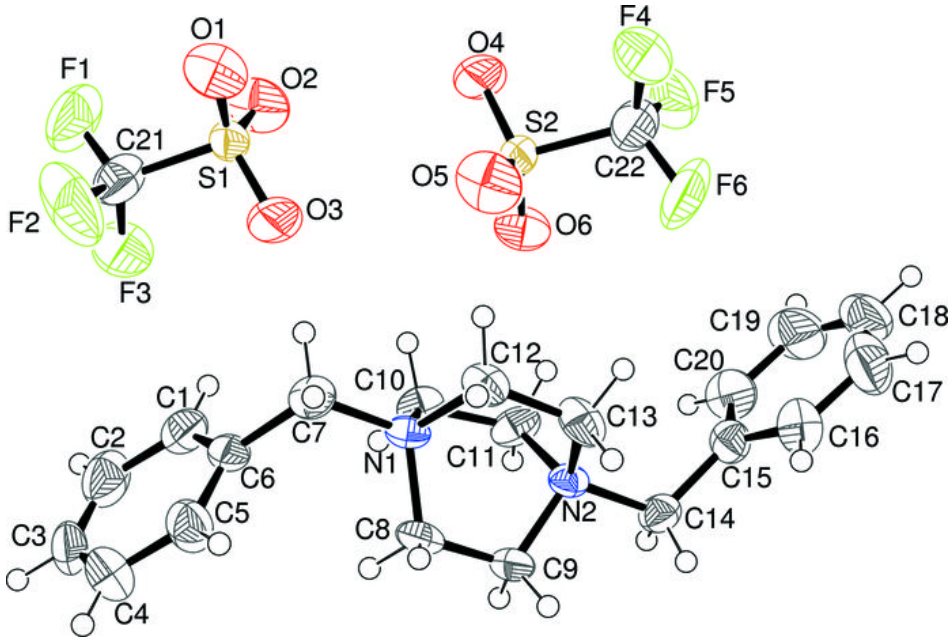
C1—C6—C7—N1	−89.4 (6)	O3—S1—C21—F1	−167.8 (5)
C5—C6—C7—N1	95.2 (5)	O2—S1—C21—F1	−47.5 (5)
C12—N1—C7—C6	−165.2 (4)	O1—S1—C21—F1	70.3 (5)
C8—N1—C7—C6	−46.1 (6)	O3—S1—C21—F3	−49.5 (6)
C10—N1—C7—C6	76.0 (5)	O2—S1—C21—F3	70.9 (5)
C12—N1—C8—C9	−63.2 (5)	O1—S1—C21—F3	−171.4 (5)
C10—N1—C8—C9	53.4 (5)	O3—S1—C21—F2'	142 (2)
C7—N1—C8—C9	177.7 (4)	O2—S1—C21—F2'	−98 (2)
C13—N2—C9—C8	53.6 (5)	O1—S1—C21—F2'	20 (2)
C11—N2—C9—C8	−63.8 (5)	O3—S1—C21—F1'	−85 (2)
C14—N2—C9—C8	175.4 (4)	O2—S1—C21—F1'	35 (2)
N1—C8—C9—N2	9.1 (6)	O1—S1—C21—F1'	153 (2)
C12—N1—C10—C11	52.1 (5)	O3—S1—C21—F3'	28 (2)
C8—N1—C10—C11	−64.4 (5)	O2—S1—C21—F3'	148 (2)
C7—N1—C10—C11	170.7 (4)	O1—S1—C21—F3'	−94 (2)
C13—N2—C11—C10	−64.0 (5)	O4—S2—C22—F5	−56.3 (9)
C9—N2—C11—C10	52.8 (5)	O5—S2—C22—F5	−178.0 (9)
C14—N2—C11—C10	171.5 (4)	O6—S2—C22—F5	63.7 (9)
N1—C10—C11—N2	9.4 (5)	O4—S2—C22—F6	−178.1 (9)
C8—N1—C12—C13	52.9 (5)	O5—S2—C22—F6	60.2 (9)
C10—N1—C12—C13	−63.7 (5)	O6—S2—C22—F6	−58.1 (9)
C7—N1—C12—C13	175.2 (4)	O4—S2—C22—F4	62.1 (9)
C9—N2—C13—C12	−64.1 (5)	O5—S2—C22—F4	−59.6 (9)
C11—N2—C13—C12	52.6 (5)	O6—S2—C22—F4	−177.9 (9)
C14—N2—C13—C12	176.6 (4)	O4—S2—C22—F6'	−89.0 (19)
N1—C12—C13—N2	9.7 (6)	O5—S2—C22—F6'	149.3 (19)
C13—N2—C14—C15	−49.7 (6)	O6—S2—C22—F6'	31.0 (19)
C9—N2—C14—C15	−168.8 (4)	O4—S2—C22—F5'	39.1 (16)
C11—N2—C14—C15	72.8 (5)	O5—S2—C22—F5'	−82.6 (16)
N2—C14—C15—C16	95.0 (6)	O6—S2—C22—F5'	159.1 (16)
N2—C14—C15—C20	−89.1 (6)	O4—S2—C22—F4'	158.8 (14)
C20—C15—C16—C17	0.9 (8)	O5—S2—C22—F4'	37.0 (14)
C14—C15—C16—C17	176.8 (5)	O6—S2—C22—F4'	−81.2 (14)
C15—C16—C17—C18	−0.4 (9)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7A···O6^{i}	0.97	2.54	3.380 (6)	145
C9—H9A···O1^{ii}	0.97	2.56	3.416 (6)	148
C9—H9B···O2^{iii}	0.97	2.38	3.227 (6)	146
C10—H10B···O1^{iii}	0.97	2.43	3.347 (6)	156
C11—H11A···O4^{iii}	0.97	2.38	3.255 (6)	150
C11—H11B···O6	0.97	2.37	3.191 (5)	142
C12—H12B···O5	0.97	2.38	3.287 (6)	155
C14—H14A···O1^{ii}	0.97	2.50	3.357 (7)	148
C14—H14B···O4^{iii}	0.97	2.54	3.394 (7)	147

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

Fig. 1


O1
F1
O2
C21
S1
O3
F2
F3
O4
F4
F5
S2
C22
O5
F6
O6
C19
C18
C20
C17
C12
C10
C1
C13
C7
C16
C2
N1
C11
C6
C15
C5
N2
C14
C3
C8
C9
C4