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(+)-(2*S*,3*S*,4*S*,5*S*)-3-[3,5-Bis(trifluoromethyl)benzyloxy]-*N*-(*tert*-butoxycarbonyl)-4,5-dimethyl-2-phenylpyrrolidine, (I), and (+)-(2*S*,3*S*,4*S*,5*S*)-3-[3,5-bis(trifluoromethyl)benzyloxy]-4,5-dimethyl-2-phenylpyrrolidinium *p*-toluenesulfonate, (II), at 134K

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Abstract

The structures of the title compounds, *tert*-butyl (+)-(2*S*,3S,4*S*,5S)-3-[3,5-bis(trifluoromethyl)benzyloxy]- 4,5-dimethyl-2-phenylpyrrolidine-1-carboxylate, $C_{26}H_{29}F_6NO_3$, and (+)-(2*S*,3S,4*S*,5S)-3-[3,5-bis(trifluoromethyl)benzyloxy]-4,5-dimethyl-2- phenylpyrrolidinium *p*-toluenesulfonate, $C_{21}H_{22}F_6NO^+\cdot C_7H_7O_3S^-$, were determined at 134 K. The pyrrolidine rings have similar twist conformations in both compounds. The molecules of the *p*-toluenesulfonate complex are connected by intermolecular N—H···O hydrogen bonds into zigzag chains. Both crystal structures contain weak intermolecular C—H···O interactions. The *p*-toluenesulfonate complex also shows intermolecular C—H···π_{arene} interactions.

Comment

The five-membered pyrrolidine ring has a C2,C3 twist conformation in both compounds. The ring puckering parameters defined by Cremer & Pople (1975) are q = 0.371Å and φ = 86.4° in (I) and q = 0.400Å and φ = 91.2° in (II). The methyl group attached to C2 is in an equatorial position, the ether oxygen attached to C3 is in an axial position and the methyl and phenyl groups attached to C1 and C4 are both in pseudo-equatorial positions with respect to the pyrrolidine ring. The N—C1 and N—C4 bonds are about 0.04Å longer in the protonated compound (II) compared to (I). The C1—C2 and C3—C4 bonds in (II) are about 0.02%A shorter than those in (I). The amide bond in (I) shows a small deviation from planarity: the angle between the planes of the N atom and the carboxylate group is 15°. Molecule (I) shows an intramolecular C12—H12A···*Cg* contact (*Cg* is the centroid of the phenyl group labeled C21–C26), with a H···*Cg* distance of 2.78 Å. The largest differences between (I) and (II) are found for the orientations of the bis(trifluoro)phenyl groups and for the phenyl groups attached to C4. These differences may be attributed to crystal-packing effects. The shortest intermolecular contact in (I) is a C16—H16···O1(1 − *x*, 1/2 + *y*, 1 − *z*) contact with a H···O distance of only 2.37 Å. The crystal packing in (II) shows two intermolecular hydrogen bonds between the NH_2^+ group and O atoms of two symmetry-related *p*-toluenesulfonate groups. The hydrogen bonding results in chains of molecules in the crystallographic b direction which corresponds to the long dimension of the crystal. The S—3 and S—O4 bonds are slightly longer than the S—O2 bond due to the involvement of O3 and O4 in the hydrogen-bonding system. The crystal structure of (II) contains a short intermolecular C5—H5B···O3(1 − *x*, *y* − 1/2, −*z*) contact with a H···O distance of only 2.37 Å, a number of additional intermolecular C—H···O contacts with H···O distances between 2.44 and 2.49Å and two intermolecular C—H···*Cg* contacts with H···*Cg* distances of 2.84 and 2.88 Å.

Experimental

(2*S*,3S,4*S*,5S)-*N*-(*tert*-Butoxycarbonyl)-3-hydroxy-4,5-dimethyl-2-phenyl- pyrrolidine was synthesized as described by Reggelin & Heinrich (1998). Reaction of this compound with potassium hexamethyldisilazide and bis-(3,5-trifluoromethyl)benzyl bromide resulted in (I). Crystals of (I) were obtained from a solution in diethyl ether. Removal of

the t-butoxycarbonyl protecting group with *p*-toluenesulfonic acid resulted in the salt (II), which was recrystallized from ethyl acetate.

Refinement

The absolute configuration of (II) was determined using 3881 unique reflections and 3081 Friedel opposites. As compound (I) is a precursor of (II) it should have the same absolute configuration. The H atoms were determined from a difference Fourier synthesis and were refined with fixed individual displacement parameters [U(H) = 1.2U_{eq}(C), U(H) = 1.5U_{eq}(C_{methyl}) and U(H) = 1.2U_{eq}(N)] using a riding model with fixed distances: *H*—*C*(methyl) = 0.98 Å, H—C(aromatic) = 0.96 Å, H—C(secondary) = 0.99 Å, H—C(tertiary) = 1.00Å and H—N = 0.90 Å. Torsion angles about the C—C bond of the methyl groups were allowed to refine.

Computing details

For both compounds, data collection: *SMART* (Siemens, 1995); cell refinement: *SMART* (Siemens, 1995); data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1996); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 1996); software used to prepare material for publication: CIF in *SHELXL97* (Sheldrick, 1997)

(+)-(2*S*,3S,4*S*,5S)-*N*-(*tert*-butoxycarbonyl)-3-[3,5-bis-(trifluoromethyl)- phenylmethoxy]-4,5-dimethyl-2-phenyl-pyrrolidine

Crystal data

$C_{26}H_{29}F_6NO_3$	V = 1281.5 (3) Å^3
M_r = 517.50	Z = 2
Monoclinic, $P2_1$	Mo $K\alpha$
a = 9.0696 (15) Å	μ = 0.12 mm^{-1}
b = 16.809 (2) Å	T = 134 (2) K
c = 9.2603 (11) Å	0.90 × 0.90 × 0.45 mm
β = 114.804 (12)º	

Data collection

Siemens SMART diffractometer	6888 independent reflections
Absorption correction: numerical (SHELXTL; Sheldrick, 1996)	6656 reflections with $I > 2\sigma(I)$
T_{min} = 0.901, T_{max} = 0.951	R_{int} = 0.052
21131 measured reflections	

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047	H-atom parameters constrained
$wR(F^2)$ = 0.130	$\Delta\rho_{max}$ = 0.31 e Å^{-3}

S = 1.68	$\Delta\rho_{min}$ = −0.22 e Å^{-3}
6888 reflections	Absolute structure: Flack (1983)
330 parameters	Flack parameter: 0.1 (4)
1 restraint	

(+)-(2*S*,3S,4*S*,5S)-3-[3,5-bis-(trifluoromethyl)-phenylmethoxy]-4,5-dimethyl-2- phenyl-pyrrolidinyl *p*-toluenesulfonate

Crystal data

$C_{21}H_{22}F_6NO^{1+}\cdot C_7H_7O_3S^{1-}$	V = 1418.2 (4) Å^3
M_r = 589.58	Z = 2
Monoclinic, $P2_1$	Mo $K\alpha$
a = 9.8727 (18) Å	μ = 0.19 mm^{-1}
b = 8.8917 (11) Å	T = 134 (2) K
c = 16.527 (2) Å	0.85 × 0.40 × 0.18 mm
β = 102.162 (12)º	

Data collection

Siemens SMART diffractometer	6962 independent reflections
Absorption correction: numerical (SHELXTL; Sheldrick, 1996)	5978 reflections with $I > 2\sigma(I)$
T_{min} = 0.881, T_{max} = 0.968	R_{int} = 0.068
22132 measured reflections	

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047	H-atom parameters constrained
$wR(F^2)$ = 0.113	$\Delta\rho_{max}$ = 0.44 e Å^{-3}
S = 1.29	$\Delta\rho_{min}$ = −0.36 e Å^{-3}
6962 reflections	Absolute structure: Flack (1983)
364 parameters	Flack parameter: −0.04 (6)
1 restraint	

Table 1
Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H02···O3^{i}	0.90	1.95	2.801 (2)	158
N—H01···O4^{ii}	0.90	1.88	2.771 (2)	173

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

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CIF access

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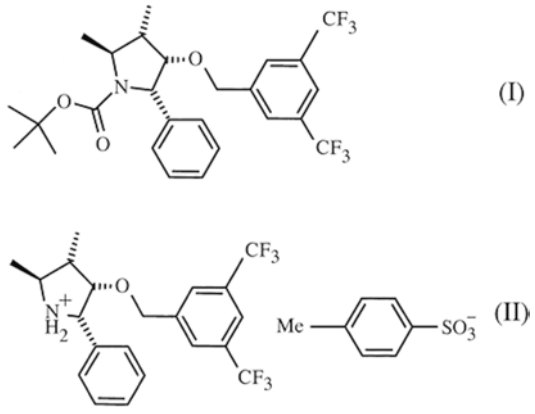
Scheme 1



supplementary materials

(+)-(2*S*,3*S*,4*S*,5*S*)-*N*-(*tert*-butoxycarbonyl)-3-[3,5-bis-(trifluoromethyl)- phenylmethoxy]-4,5-dimethyl-2-phenyl-pyrrolidine

Crystal data

$C_{26}H_{29}F_6NO_3$	F_{000} = 540
M_r = 517.50	D_x = 1.341 Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 9.0696 (15) Å	Cell parameters from 264 reflections
b = 16.809 (2) Å	θ = 3–23º
c = 9.2603 (11) Å	μ = 0.12 mm^{-1}
β = 114.804 (12)º	T = 134 (2) K
V = 1281.5 (3) Å^3	Block, yellow
Z = 2	0.90 × 0.90 × 0.45 mm

Data collection

Siemens SMART diffractometer	6888 independent reflections
Radiation source: fine-focus sealed tube	6656 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.052
T = 134(2) K	θ_{max} = 31.7º
ω scans	θ_{min} = 2.4º
Absorption correction: numerical (SHELXTL; Sheldrick, 1996)	h = −12→13
T_{min} = 0.901, T_{max} = 0.951	k = −22→24
21131 measured reflections	l = −13→13

Refinement

Refinement on F^2	Hydrogen site location: difference Fourier map
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.047	$w = 1/[\sigma^2(F_o^2) + (0.06P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.130	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.68	$\Delta\rho_{max}$ = 0.31 e Å^{-3}
6888 reflections	$\Delta\rho_{min}$ = −0.22 e Å^{-3}
330 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983)
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.1 (4)
Secondary atom site location: difference Fourier map	

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}
O3	0.16619 (12)	0.06171 (6)	0.12244 (11)	0.01714 (19)
F6	0.49686 (13)	0.06964 (6)	0.71888 (11)	0.0295 (2)
O2	−0.18058 (13)	−0.14851 (6)	0.15462 (14)	0.0241 (2)
F5	0.68772 (15)	0.04823 (7)	0.64605 (14)	0.0400 (3)
F1	0.46907 (15)	0.39766 (7)	0.24255 (14)	0.0409 (3)
O1	0.05232 (14)	−0.21959 (7)	0.21696 (14)	0.0262 (2)
C15	0.49299 (17)	0.27872 (8)	0.38668 (16)	0.0187 (2)
N	0.00469 (15)	−0.10279 (7)	0.07934 (15)	0.0198 (2)
C18	0.37692 (17)	0.12896 (8)	0.40689 (17)	0.0196 (3)
H18	0.3383	0.0766	0.4144	0.023*
C1	0.14088 (17)	−0.10960 (8)	0.03221 (16)	0.0192 (2)
H1	0.2464	−0.1039	0.1280	0.023*
C3	0.03766 (16)	0.02511 (8)	−0.00940 (15)	0.0171 (2)
H3	−0.0249	0.0654	−0.0917	0.020*
C4	−0.07692 (16)	−0.02443 (8)	0.04228 (16)	0.0176 (2)
H4	−0.1850	−0.0301	−0.0503	0.021*
F3	0.70913 (16)	0.35498 (8)	0.38243 (19)	0.0525 (4)
F4	0.70461 (16)	0.14736 (7)	0.79914 (13)	0.0470 (3)
C13	0.29318 (17)	0.17535 (8)	0.27243 (16)	0.0174 (2)
C16	0.57745 (17)	0.23302 (8)	0.52229 (16)	0.0194 (2)
H16	0.6746	0.2526	0.6072	0.023*
C17	0.51643 (17)	0.15828 (8)	0.53041 (16)	0.0184 (2)
C5	−0.03298 (18)	−0.16197 (8)	0.15760 (17)	0.0205 (3)
C14	0.35188 (18)	0.25092 (8)	0.26241 (16)	0.0190 (2)
H14	0.2955	0.2836	0.1705	0.023*
C19	0.5597 (2)	0.35937 (9)	0.3769 (2)	0.0274 (3)
C2	0.11350 (18)	−0.03708 (8)	−0.07760 (16)	0.0190 (2)
H2	0.0311	−0.0520	−0.1861	0.023*
C21	−0.10006 (17)	0.01418 (8)	0.17915 (16)	0.0201 (3)
C12	0.14052 (17)	0.14405 (8)	0.14093 (16)	0.0193 (3)
H12A	0.0470	0.1510	0.1686	0.023*
H12B	0.1176	0.1731	0.0407	0.023*
C20	0.60164 (18)	0.10634 (9)	0.67360 (17)	0.0235 (3)

C6	−0.2393 (2)	−0.19430 (10)	0.2558 (2)	0.0281 (3)
F2	0.5724 (2)	0.40652 (7)	0.49753 (17)	0.0546 (4)
C22	−0.2381 (2)	0.06005 (10)	0.1475 (2)	0.0286 (3)
H22	−0.3239	0.0613	0.0421	0.034*
C10	0.1401 (2)	−0.18718 (9)	−0.0537 (2)	0.0283 (3)
H10A	0.2243	−0.1851	−0.0938	0.042*
H10C	0.0336	−0.1944	−0.1430	0.042*
H10B	0.1618	−0.2318	0.0205	0.042*
C11	0.2659 (2)	−0.00887 (10)	−0.0927 (2)	0.0274 (3)
H11A	0.3511	0.0024	0.0133	0.041*
H11C	0.2414	0.0396	−0.1574	0.041*
H11B	0.3033	−0.0504	−0.1437	0.041*
C26	0.0195 (2)	0.00951 (10)	0.33418 (18)	0.0251 (3)
H26	0.1126	−0.0240	0.3579	0.030*
C24	−0.1302 (3)	0.10184 (13)	0.4219 (2)	0.0414 (4)
H24	−0.1383	0.1334	0.5046	0.050*
C25	0.0044 (3)	0.05330 (12)	0.4550 (2)	0.0355 (4)
H25	0.0873	0.0500	0.5617	0.043*
C9	−0.1219 (3)	−0.18664 (13)	0.4297 (2)	0.0404 (4)
H9A	−0.0194	−0.2131	0.4474	0.061*
H9B	−0.1690	−0.2116	0.4962	0.061*
H9C	−0.1013	−0.1302	0.4576	0.061*
C23	−0.2517 (3)	0.10440 (13)	0.2698 (3)	0.0402 (4)
H23	−0.3462	0.1366	0.2474	0.048*
C8	−0.2654 (3)	−0.28101 (12)	0.2024 (3)	0.0402 (4)
H8A	−0.3166	−0.3096	0.2616	0.060*
H8B	−0.1605	−0.3056	0.2230	0.060*
H8C	−0.3360	−0.2835	0.0883	0.060*
C7	−0.3992 (3)	−0.15346 (15)	0.2220 (3)	0.0481 (5)
H7C	−0.4661	−0.1526	0.1070	0.072*
H7B	−0.3786	−0.0988	0.2623	0.072*
H7A	−0.4565	−0.1825	0.2748	0.072*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0162 (4)	0.0132 (4)	0.0179 (4)	−0.0002 (3)	0.0031 (4)	−0.0009 (3)
F6	0.0312 (5)	0.0289 (5)	0.0310 (5)	0.0021 (4)	0.0155 (4)	0.0107 (4)
O2	0.0184 (5)	0.0234 (5)	0.0325 (5)	−0.0002 (4)	0.0127 (4)	0.0089 (4)
F5	0.0369 (6)	0.0435 (6)	0.0430 (6)	0.0218 (5)	0.0200 (5)	0.0193 (5)
F1	0.0403 (6)	0.0259 (5)	0.0408 (6)	−0.0093 (4)	0.0017 (5)	0.0138 (4)
O1	0.0238 (6)	0.0223 (5)	0.0331 (6)	0.0039 (4)	0.0125 (5)	0.0075 (4)
C15	0.0180 (6)	0.0152 (6)	0.0220 (6)	−0.0027 (4)	0.0075 (5)	−0.0013 (4)
N	0.0185 (5)	0.0158 (5)	0.0277 (6)	0.0012 (4)	0.0124 (5)	0.0027 (4)
C18	0.0190 (6)	0.0157 (5)	0.0217 (6)	−0.0013 (4)	0.0064 (5)	0.0012 (5)
C1	0.0185 (6)	0.0172 (6)	0.0233 (6)	−0.0014 (5)	0.0101 (5)	−0.0009 (5)
C3	0.0159 (6)	0.0165 (6)	0.0160 (5)	−0.0014 (4)	0.0040 (4)	0.0010 (4)
C4	0.0149 (6)	0.0165 (6)	0.0198 (5)	0.0004 (4)	0.0058 (5)	0.0012 (4)

F3	0.0308 (6)	0.0455 (7)	0.0789 (10)	−0.0082 (5)	0.0207 (6)	0.0224 (6)
F4	0.0529 (8)	0.0342 (6)	0.0257 (5)	−0.0161 (5)	−0.0113 (5)	0.0080 (4)
C13	0.0165 (6)	0.0153 (5)	0.0187 (6)	−0.0010 (4)	0.0057 (5)	−0.0011 (4)
C16	0.0179 (6)	0.0187 (6)	0.0207 (6)	−0.0024 (5)	0.0072 (5)	−0.0014 (4)
C17	0.0182 (6)	0.0172 (6)	0.0180 (6)	0.0003 (5)	0.0058 (5)	0.0016 (4)
C5	0.0180 (6)	0.0193 (6)	0.0234 (6)	−0.0016 (5)	0.0079 (5)	0.0012 (5)
C14	0.0204 (6)	0.0154 (5)	0.0195 (6)	0.0003 (4)	0.0067 (5)	0.0020 (4)
C19	0.0289 (8)	0.0191 (6)	0.0298 (7)	−0.0068 (5)	0.0079 (6)	0.0038 (5)
C2	0.0208 (6)	0.0180 (6)	0.0184 (6)	−0.0030 (5)	0.0083 (5)	−0.0031 (4)
C21	0.0191 (6)	0.0197 (6)	0.0238 (6)	0.0002 (5)	0.0113 (5)	0.0018 (5)
C12	0.0170 (6)	0.0138 (6)	0.0217 (6)	0.0016 (4)	0.0029 (5)	0.0013 (4)
C20	0.0226 (7)	0.0195 (6)	0.0235 (6)	−0.0021 (5)	0.0048 (5)	0.0040 (5)
C6	0.0271 (8)	0.0269 (7)	0.0371 (8)	−0.0010 (6)	0.0201 (7)	0.0084 (6)
F2	0.0914 (11)	0.0254 (5)	0.0473 (7)	−0.0234 (6)	0.0294 (7)	−0.0115 (5)
C22	0.0235 (7)	0.0315 (8)	0.0348 (8)	0.0070 (6)	0.0160 (6)	0.0054 (6)
C10	0.0329 (8)	0.0191 (6)	0.0382 (8)	−0.0016 (5)	0.0201 (7)	−0.0045 (6)
C11	0.0325 (8)	0.0239 (7)	0.0350 (8)	−0.0034 (6)	0.0233 (7)	−0.0012 (5)
C26	0.0252 (7)	0.0267 (7)	0.0235 (7)	−0.0028 (5)	0.0104 (6)	0.0012 (5)
C24	0.0518 (12)	0.0448 (10)	0.0425 (10)	−0.0026 (9)	0.0345 (9)	−0.0103 (8)
C25	0.0415 (10)	0.0427 (10)	0.0253 (7)	−0.0073 (8)	0.0170 (7)	−0.0045 (7)
C9	0.0479 (11)	0.0462 (11)	0.0316 (8)	−0.0051 (9)	0.0212 (8)	0.0038 (7)
C23	0.0403 (10)	0.0427 (10)	0.0514 (11)	0.0093 (8)	0.0328 (9)	−0.0015 (8)
C8	0.0450 (11)	0.0311 (9)	0.0515 (11)	−0.0133 (8)	0.0273 (9)	0.0014 (7)
C7	0.0339 (10)	0.0524 (12)	0.0720 (14)	0.0095 (9)	0.0362 (10)	0.0236 (11)

Geometric parameters (Å, °)

O3—C12	1.4256 (16)	C3—C4	1.5554 (18)
O3—C3	1.4262 (15)	C4—C21	1.5149 (18)
F6—C20	1.3394 (18)	F3—C19	1.337 (2)
O2—C5	1.3464 (17)	F4—C20	1.3372 (18)
O2—C6	1.4738 (17)	C13—C14	1.3954 (18)
F5—C20	1.3401 (19)	C13—C12	1.5040 (19)
F1—C19	1.3337 (19)	C16—C17	1.3874 (19)
O1—C5	1.2174 (18)	C17—C20	1.5026 (19)
C15—C14	1.3946 (19)	C19—F2	1.335 (2)
C15—C16	1.3953 (19)	C2—C11	1.522 (2)
C15—C19	1.5025 (19)	C21—C26	1.392 (2)
N—C5	1.3561 (18)	C21—C22	1.393 (2)
N—C1	1.4770 (17)	C6—C7	1.514 (3)
N—C4	1.4791 (17)	C6—C9	1.518 (3)
C18—C17	1.3921 (19)	C6—C8	1.525 (3)
C18—C13	1.3935 (18)	C22—C23	1.404 (2)
C1—C10	1.526 (2)	C26—C25	1.393 (2)
C1—C2	1.5395 (19)	C24—C23	1.377 (3)
C3—C2	1.5262 (18)	C24—C25	1.392 (3)
C12—O3—C3	113.93 (10)	F1—C19—F3	106.29 (14)
C5—O2—C6	121.36 (12)	F2—C19—F3	105.68 (15)
C14—C15—C16	121.70 (12)	F1—C19—C15	112.69 (13)

C14—C15—C19	120.37 (13)	F2—C19—C15	112.37 (13)
C16—C15—C19	117.92 (13)	F3—C19—C15	112.04 (14)
C5—N—C1	122.04 (11)	C11—C2—C3	114.14 (12)
C5—N—C4	124.29 (11)	C11—C2—C1	113.97 (13)
C1—N—C4	113.55 (11)	C3—C2—C1	104.17 (10)
C17—C18—C13	120.19 (13)	C26—C21—C22	119.41 (14)
N—C1—C10	113.49 (12)	C26—C21—C4	121.15 (13)
N—C1—C2	102.16 (11)	C22—C21—C4	119.27 (13)
C10—C1—C2	111.60 (12)	O3—C12—C13	107.19 (11)
O3—C3—C2	107.54 (11)	F4—C20—F6	107.08 (12)
O3—C3—C4	112.28 (10)	F4—C20—F5	106.93 (14)
C2—C3—C4	103.65 (10)	F6—C20—F5	105.69 (12)
N—C4—C21	114.76 (11)	F4—C20—C17	112.38 (12)
N—C4—C3	102.37 (10)	F6—C20—C17	111.97 (12)
C21—C4—C3	111.91 (11)	F5—C20—C17	112.37 (12)
C18—C13—C14	119.26 (12)	O2—C6—C7	101.65 (13)
C18—C13—C12	119.95 (12)	O2—C6—C9	110.50 (14)
C14—C13—C12	120.79 (12)	C7—C6—C9	111.46 (17)
C17—C16—C15	117.80 (13)	O2—C6—C8	109.98 (14)
C16—C17—C18	121.45 (13)	C7—C6—C8	111.16 (17)
C16—C17—C20	120.31 (13)	C9—C6—C8	111.67 (16)
C18—C17—C20	118.23 (12)	C21—C22—C23	119.98 (16)
O1—C5—O2	125.52 (13)	C21—C26—C25	120.12 (16)
O1—C5—N	124.75 (13)	C23—C24—C25	119.71 (16)
O2—C5—N	109.70 (12)	C24—C25—C26	120.36 (17)
C15—C14—C13	119.59 (12)	C24—C23—C22	120.30 (17)
F1—C19—F2	107.29 (15)		

C5—N—C1—C10	49.32 (19)	C16—C15—C19—F2	−58.5 (2)
C4—N—C1—C10	−134.41 (13)	C14—C15—C19—F3	−119.04 (16)
C5—N—C1—C2	169.60 (13)	C16—C15—C19—F3	60.30 (18)
C4—N—C1—C2	−14.13 (14)	O3—C3—C2—C11	−43.93 (15)
C12—O3—C3—C2	145.36 (11)	C4—C3—C2—C11	−163.00 (12)
C12—O3—C3—C4	−101.25 (13)	O3—C3—C2—C1	80.98 (12)
C5—N—C4—C21	45.76 (19)	C4—C3—C2—C1	−38.10 (13)
C1—N—C4—C21	−130.41 (13)	N—C1—C2—C11	156.88 (12)
C5—N—C4—C3	167.21 (13)	C10—C1—C2—C11	−81.53 (15)
C1—N—C4—C3	−8.96 (14)	N—C1—C2—C3	31.87 (13)
O3—C3—C4—N	−87.12 (12)	C10—C1—C2—C3	153.46 (12)
C2—C3—C4—N	28.64 (13)	N—C4—C21—C26	39.01 (18)
O3—C3—C4—C21	36.27 (15)	C3—C4—C21—C26	−77.08 (16)
C2—C3—C4—C21	152.03 (11)	N—C4—C21—C22	−145.80 (14)
C17—C18—C13—C14	0.8 (2)	C3—C4—C21—C22	98.11 (15)
C17—C18—C13—C12	−178.67 (13)	C3—O3—C12—C13	−173.96 (11)
C14—C15—C16—C17	−0.1 (2)	C18—C13—C12—O3	−39.50 (16)
C19—C15—C16—C17	−179.43 (13)	C14—C13—C12—O3	141.07 (12)
C15—C16—C17—C18	0.9 (2)	C16—C17—C20—F4	17.6 (2)
C15—C16—C17—C20	−179.81 (12)	C18—C17—C20—F4	−163.06 (14)
C13—C18—C17—C16	−1.3 (2)	C16—C17—C20—F6	138.19 (13)
C13—C18—C17—C20	179.44 (12)	C18—C17—C20—F6	−42.49 (18)

C6—O2—C5—O1	14.0 (2)	C16—C17—C20—F5	−103.04 (16)
C6—O2—C5—N	−168.06 (13)	C18—C17—C20—F5	76.28 (17)
C1—N—C5—O1	11.5 (2)	C5—O2—C6—C7	175.15 (16)
C4—N—C5—O1	−164.33 (14)	C5—O2—C6—C9	56.7 (2)
C1—N—C5—O2	−166.38 (12)	C5—O2—C6—C8	−67.0 (2)
C4—N—C5—O2	17.76 (19)	C26—C21—C22—C23	3.4 (2)
C16—C15—C14—C13	−0.4 (2)	C4—C21—C22—C23	−171.90 (15)
C19—C15—C14—C13	178.96 (13)	C22—C21—C26—C25	−2.9 (2)
C18—C13—C14—C15	0.02 (19)	C4—C21—C26—C25	172.25 (14)
C12—C13—C14—C15	179.46 (13)	C23—C24—C25—C26	2.3 (3)
C14—C15—C19—F1	0.8 (2)	C21—C26—C25—C24	0.1 (3)
C16—C15—C19—F1	−179.88 (14)	C25—C24—C23—C22	−1.8 (3)
C14—C15—C19—F2	122.14 (16)	C21—C22—C23—C24	−1.0 (3)

(+)-(2*S*,3S,4*S*,5S)-3-[3,5-bis-(trifluoromethyl)-phenylmethoxy]-4,5-dimethyl-2- phenyl-pyrrolidinyl *p*-toluenesulfonate

Crystal data

$C_{21}H_{22}F_6NO^{1+}\cdot C_7H_7O_3S^{1-}$	F_{000} = 612
M_r = 589.58	D_x = 1.381 Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 9.8727 (18) Å	Cell parameters from 183 reflections
b = 8.8917 (11) Å	θ = 3–23º
c = 16.527 (2) Å	μ = 0.19 mm^{-1}
β = 102.162 (12)º	T = 134 (2) K
V = 1418.2 (4) Å^3	Ruler, colorless
Z = 2	0.85 × 0.40 × 0.18 mm

Data collection

Siemens SMART diffractometer	6962 independent reflections
Radiation source: fine-focus sealed tube	5978 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.068
T = 134(2) K	θ_{max} = 29.7º
ω scans	θ_{min} = 2.1º
Absorption correction: numerical (SHELXTL; Sheldrick, 1996)	h = −13→13
T_{min} = 0.881, T_{max} = 0.968	k = −12→12
22132 measured reflections	l = −21→22

Refinement

Refinement on F^2	Hydrogen site location: difference Fourier map
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.047	$w = 1/[\sigma^2(F_o^2) + (0.05P)^2]$

	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.113$	$(\Delta/\sigma)_{max} = 0.010$
$S = 1.29$	$\Delta\rho_{max} = 0.44$ e Å^{-3}
6962 reflections	$\Delta\rho_{min} = -0.36$ e Å^{-3}
364 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983)
Primary atom site location: structure-invariant direct methods	Flack parameter: −0.04 (6)
Secondary atom site location: difference Fourier map	

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}
S	0.87045 (5)	0.34522 (5)	0.12279 (3)	0.02393 (12)
O1	0.37951 (14)	0.38294 (15)	0.15830 (9)	0.0260 (3)
C22	0.9063 (2)	0.3431 (3)	0.23270 (12)	0.0261 (4)
O4	0.95892 (17)	0.22819 (17)	0.09864 (10)	0.0314 (4)
N	0.14346 (17)	0.53302 (18)	0.03048 (10)	0.0219 (3)
H01	0.1030	0.5928	−0.0117	0.026*
H02	0.0847	0.5218	0.0649	0.026*
O3	0.90874 (17)	0.49438 (16)	0.09843 (10)	0.0314 (4)
O2	0.72433 (16)	0.31331 (19)	0.09674 (9)	0.0375 (4)
C1	0.1762 (2)	0.3802 (2)	−0.00257 (13)	0.0236 (4)
H1	0.1487	0.2997	0.0331	0.028*
C4	0.2783 (2)	0.6037 (2)	0.07653 (12)	0.0231 (4)
H4	0.3077	0.6771	0.0377	0.028*
F1	0.6452 (2)	−0.1360 (2)	0.19782 (10)	0.0767 (6)
C13	0.4614 (2)	0.2654 (2)	0.35157 (14)	0.0315 (5)
H13	0.4122	0.3496	0.3677	0.038*
C3	0.3824 (2)	0.4740 (2)	0.08761 (12)	0.0228 (4)
H3	0.4781	0.5130	0.0898	0.027*
F6	0.3739 (2)	−0.0092 (2)	0.48886 (11)	0.0730 (6)
C8	0.5137 (2)	0.2724 (2)	0.28012 (13)	0.0279 (5)
C10	0.6004 (2)	0.0201 (2)	0.30462 (13)	0.0297 (5)
C9	0.5837 (2)	0.1499 (2)	0.25668 (13)	0.0288 (5)
H9	0.6205	0.1548	0.2074	0.035*

C5	0.0957 (2)	0.3592 (3)	−0.09020 (14)	0.0347 (5)
H5A	−0.0036	0.3700	−0.0917	0.052*
H5B	0.1140	0.2587	−0.1098	0.052*
H5C	0.1245	0.4353	−0.1260	0.052*
C2	0.3331 (2)	0.3809 (2)	0.00915 (13)	0.0266 (4)
H2	0.3582	0.4356	−0.0385	0.032*
C17	0.2033 (2)	0.6268 (3)	0.21336 (14)	0.0366 (5)
H17	0.1825	0.5212	0.2116	0.044*
C27	0.8636 (3)	0.4639 (3)	0.27480 (14)	0.0332 (5)
H27	0.8233	0.5513	0.2451	0.040*
C14	0.6737 (3)	−0.1147 (3)	0.27936 (15)	0.0391 (6)
F4	0.3304 (2)	0.2286 (2)	0.48227 (10)	0.0643 (5)
F3	0.6353 (2)	−0.24233 (18)	0.30903 (15)	0.0831 (7)
C12	0.4806 (3)	0.1352 (2)	0.40011 (13)	0.0324 (5)
C16	0.2570 (2)	0.6920 (2)	0.15034 (14)	0.0298 (5)
F2	0.80897 (19)	−0.1078 (2)	0.30150 (15)	0.0857 (7)
C15	0.4271 (3)	0.1259 (3)	0.47797 (16)	0.0445 (6)
F5	0.5269 (2)	0.1468 (2)	0.54538 (9)	0.0658 (5)
C23	0.9661 (3)	0.2183 (3)	0.27533 (16)	0.0433 (6)
H23	0.9960	0.1355	0.2462	0.052*
C21	0.2864 (2)	0.8458 (3)	0.15421 (17)	0.0424 (6)
H21	0.3243	0.8921	0.1114	0.051*
C6	0.3915 (3)	0.2223 (3)	0.01173 (18)	0.0434 (6)
H6A	0.3593	0.1739	−0.0422	0.065*
H6B	0.3598	0.1641	0.0546	0.065*
H6C	0.4929	0.2267	0.0244	0.065*
C11	0.5503 (3)	0.0123 (2)	0.37705 (14)	0.0326 (5)
H11	0.5637	−0.0769	0.4106	0.039*
C26	0.8799 (3)	0.4565 (3)	0.35995 (16)	0.0444 (6)
H26	0.8494	0.5392	0.3889	0.053*
C25	0.9393 (3)	0.3325 (4)	0.40412 (15)	0.0582 (8)
C7	0.4933 (2)	0.4109 (2)	0.22669 (14)	0.0334 (5)
H7A	0.4726	0.4985	0.2590	0.040*
H7B	0.5785	0.4329	0.2062	0.040*
C19	0.2081 (3)	0.8657 (4)	0.2817 (2)	0.0667 (10)
H19	0.1911	0.9255	0.3270	0.080*
C20	0.2611 (3)	0.9324 (4)	0.2195 (2)	0.0644 (10)
H20	0.2805	1.0383	0.2214	0.077*
C24	0.9823 (4)	0.2139 (3)	0.35982 (17)	0.0600 (9)
H24	1.0242	0.1270	0.3894	0.072*
C28	0.9566 (5)	0.3227 (5)	0.4972 (2)	0.0924 (14)
H28A	0.8959	0.3964	0.5157	0.139*
H28B	1.0532	0.3441	0.5236	0.139*
H28C	0.9321	0.2213	0.5124	0.139*
C18	0.1794 (3)	0.7139 (4)	0.27920 (17)	0.0538 (8)
H18	0.1430	0.6681	0.3227	0.065*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0239 (2)	0.0227 (2)	0.0254 (2)	−0.0052 (2)	0.00559 (18)	−0.0033 (2)
O1	0.0231 (7)	0.0231 (7)	0.0290 (7)	−0.0031 (5)	−0.0009 (6)	0.0062 (5)
C22	0.0259 (10)	0.0262 (9)	0.0256 (9)	−0.0048 (9)	0.0042 (8)	−0.0031 (9)
O4	0.0354 (9)	0.0273 (7)	0.0327 (8)	−0.0018 (6)	0.0101 (7)	−0.0056 (6)
N	0.0186 (8)	0.0210 (8)	0.0259 (9)	0.0023 (6)	0.0042 (7)	0.0038 (6)
O3	0.0353 (9)	0.0240 (7)	0.0373 (8)	−0.0026 (7)	0.0133 (7)	0.0008 (6)
O2	0.0285 (8)	0.0527 (11)	0.0311 (8)	−0.0114 (7)	0.0063 (6)	−0.0063 (7)
C1	0.0235 (10)	0.0154 (9)	0.0316 (11)	0.0000 (7)	0.0055 (8)	0.0001 (7)
C4	0.0216 (11)	0.0191 (9)	0.0277 (10)	−0.0023 (8)	0.0029 (8)	0.0047 (8)
F1	0.1050 (15)	0.0774 (13)	0.0503 (10)	0.0484 (12)	0.0218 (9)	−0.0036 (9)
C13	0.0348 (12)	0.0259 (10)	0.0304 (11)	0.0003 (9)	−0.0008 (9)	−0.0037 (8)
C3	0.0177 (10)	0.0237 (10)	0.0280 (10)	−0.0015 (8)	0.0070 (8)	0.0021 (8)
F6	0.1218 (17)	0.0566 (11)	0.0555 (11)	−0.0296 (11)	0.0526 (11)	−0.0109 (8)
C8	0.0255 (11)	0.0239 (10)	0.0301 (11)	−0.0032 (9)	−0.0035 (9)	0.0028 (8)
C10	0.0297 (12)	0.0291 (11)	0.0286 (11)	−0.0013 (9)	0.0023 (9)	0.0007 (8)
C9	0.0235 (11)	0.0316 (11)	0.0298 (11)	−0.0029 (9)	0.0025 (8)	0.0039 (9)
C5	0.0318 (12)	0.0248 (10)	0.0416 (12)	0.0003 (9)	−0.0054 (9)	−0.0106 (10)
C2	0.0204 (10)	0.0311 (11)	0.0291 (10)	−0.0022 (8)	0.0070 (8)	−0.0044 (8)
C17	0.0283 (12)	0.0510 (14)	0.0295 (12)	0.0033 (11)	0.0036 (9)	−0.0048 (10)
C27	0.0351 (13)	0.0293 (11)	0.0325 (12)	−0.0003 (10)	0.0012 (10)	−0.0070 (9)
C14	0.0437 (15)	0.0351 (13)	0.0396 (13)	0.0075 (10)	0.0109 (11)	0.0067 (9)
F4	0.0759 (12)	0.0733 (12)	0.0493 (10)	0.0194 (10)	0.0261 (9)	−0.0029 (8)
F3	0.1231 (19)	0.0276 (9)	0.1210 (18)	0.0115 (9)	0.0769 (15)	0.0050 (9)
C12	0.0409 (14)	0.0283 (10)	0.0260 (11)	−0.0036 (10)	0.0023 (9)	−0.0035 (8)
C16	0.0196 (11)	0.0287 (11)	0.0367 (12)	0.0033 (9)	−0.0041 (9)	−0.0058 (9)
F2	0.0429 (10)	0.0702 (13)	0.1344 (19)	0.0213 (9)	−0.0035 (11)	−0.0284 (12)
C15	0.0613 (18)	0.0405 (14)	0.0346 (13)	0.0004 (13)	0.0166 (12)	−0.0059 (10)
F5	0.0891 (14)	0.0783 (12)	0.0265 (8)	0.0045 (10)	0.0041 (8)	−0.0050 (8)
C23	0.0572 (17)	0.0369 (13)	0.0348 (13)	0.0162 (12)	0.0078 (11)	0.0002 (10)
C21	0.0230 (11)	0.0308 (11)	0.0689 (16)	−0.0007 (11)	−0.0006 (10)	−0.0115 (13)
C6	0.0268 (12)	0.0398 (14)	0.0611 (16)	0.0068 (10)	0.0037 (11)	−0.0225 (12)
C11	0.0408 (14)	0.0260 (10)	0.0292 (11)	0.0010 (9)	0.0031 (10)	0.0047 (8)
C26	0.0540 (17)	0.0433 (14)	0.0354 (14)	0.0059 (12)	0.0085 (12)	−0.0133 (10)
C25	0.076 (2)	0.0685 (19)	0.0277 (12)	0.0220 (18)	0.0049 (13)	−0.0052 (14)
C7	0.0322 (12)	0.0242 (10)	0.0364 (12)	−0.0050 (9)	−0.0095 (10)	0.0069 (9)
C19	0.0358 (15)	0.087 (3)	0.071 (2)	0.0021 (16)	−0.0027 (14)	−0.053 (2)
C20	0.0338 (16)	0.0483 (17)	0.101 (3)	0.0012 (13)	−0.0075 (17)	−0.0418 (17)
C24	0.089 (2)	0.0526 (17)	0.0346 (15)	0.0322 (17)	0.0052 (14)	0.0051 (12)
C28	0.140 (4)	0.100 (3)	0.0358 (16)	0.045 (3)	0.0138 (19)	−0.0008 (18)
C18	0.0364 (15)	0.087 (2)	0.0376 (15)	0.0083 (15)	0.0066 (12)	−0.0162 (14)

Geometric parameters (Å, °)

S—O2	1.4440 (16)	C10—C9	1.389 (3)
S—O3	1.4588 (15)	C10—C11	1.390 (3)

S—O4	1.4670 (16)	C10—C14	1.504 (3)
S—C22	1.776 (2)	C2—C6	1.521 (3)
O1—C3	1.426 (2)	C17—C16	1.391 (3)
O1—C7	1.438 (2)	C17—C18	1.395 (4)
C22—C23	1.379 (3)	C27—C26	1.384 (3)
C22—C27	1.392 (3)	C14—F2	1.310 (3)
N—C4	1.523 (3)	C14—F3	1.323 (3)
N—C1	1.524 (2)	F4—C15	1.333 (3)
C1—C5	1.509 (3)	C12—C11	1.387 (3)
C1—C2	1.520 (3)	C12—C15	1.493 (4)
C4—C16	1.502 (3)	C16—C21	1.396 (3)
C4—C3	1.530 (3)	C15—F5	1.336 (3)
F1—C14	1.331 (3)	C23—C24	1.372 (4)
C13—C8	1.386 (3)	C21—C20	1.391 (4)
C13—C12	1.399 (3)	C26—C25	1.382 (4)
C3—C2	1.529 (3)	C25—C24	1.400 (4)
F6—C15	1.338 (3)	C25—C28	1.514 (4)
C8—C9	1.388 (3)	C19—C18	1.378 (5)
C8—C7	1.504 (3)	C19—C20	1.382 (6)
O2—S—O3	113.39 (10)	C26—C27—C22	119.3 (2)
O2—S—O4	113.22 (9)	F2—C14—F3	106.9 (2)
O3—S—O4	111.47 (10)	F2—C14—F1	105.9 (2)
O2—S—C22	105.91 (9)	F3—C14—F1	103.7 (2)
O3—S—C22	106.52 (9)	F2—C14—C10	114.0 (2)
O4—S—C22	105.61 (10)	F3—C14—C10	112.9 (2)
C3—O1—C7	113.49 (15)	F1—C14—C10	112.53 (19)
C23—C22—C27	120.5 (2)	C11—C12—C13	120.7 (2)
C23—C22—S	120.18 (17)	C11—C12—C15	118.7 (2)
C27—C22—S	119.14 (17)	C13—C12—C15	120.7 (2)
C4—N—C1	108.56 (14)	C17—C16—C21	118.8 (2)
C5—C1—C2	116.03 (18)	C17—C16—C4	122.1 (2)
C5—C1—N	110.24 (16)	C21—C16—C4	119.1 (2)
C2—C1—N	103.78 (15)	F4—C15—F5	105.7 (2)
C16—C4—N	111.38 (17)	F4—C15—F6	107.6 (2)
C16—C4—C3	120.14 (17)	F5—C15—F6	104.7 (2)
N—C4—C3	104.04 (15)	F4—C15—C12	113.3 (2)
C8—C13—C12	119.7 (2)	F5—C15—C12	112.2 (2)
O1—C3—C2	109.26 (16)	F6—C15—C12	112.65 (19)
O1—C3—C4	113.40 (16)	C24—C23—C22	119.3 (2)
C2—C3—C4	102.75 (16)	C20—C21—C16	120.6 (3)
C13—C8—C9	119.96 (19)	C12—C11—C10	119.0 (2)
C13—C8—C7	120.6 (2)	C25—C26—C27	121.4 (2)
C9—C8—C7	119.5 (2)	C26—C25—C24	117.8 (2)
C9—C10—C11	120.8 (2)	C26—C25—C28	121.8 (3)
C9—C10—C14	120.7 (2)	C24—C25—C28	120.4 (3)
C11—C10—C14	118.6 (2)	O1—C7—C8	107.78 (16)
C8—C9—C10	119.9 (2)	C18—C19—C20	120.3 (3)
C1—C2—C6	111.72 (17)	C19—C20—C21	119.9 (3)
C1—C2—C3	104.13 (16)	C23—C24—C25	121.8 (2)

C6—C2—C3	115.28 (18)	C19—C18—C17	120.0 (3)
C16—C17—C18	120.4 (3)		
O2—S—C22—C23	100.4 (2)	C11—C10—C14—F1	142.6 (2)
O3—S—C22—C23	−138.6 (2)	C8—C13—C12—C11	−0.7 (3)
O4—S—C22—C23	−20.0 (2)	C8—C13—C12—C15	179.1 (2)
O2—S—C22—C27	−74.7 (2)	C18—C17—C16—C21	0.0 (3)
O3—S—C22—C27	46.3 (2)	C18—C17—C16—C4	−177.2 (2)
O4—S—C22—C27	164.94 (17)	N—C4—C16—C17	57.6 (3)
C4—N—C1—C5	−136.79 (18)	C3—C4—C16—C17	−64.3 (3)
C4—N—C1—C2	−11.91 (19)	N—C4—C16—C21	−119.5 (2)
C1—N—C4—C16	−144.34 (16)	C3—C4—C16—C21	118.6 (2)
C1—N—C4—C3	−13.53 (19)	C11—C12—C15—F4	−162.0 (2)
C7—O1—C3—C2	142.37 (18)	C13—C12—C15—F4	18.3 (3)
C7—O1—C3—C4	−103.7 (2)	C11—C12—C15—F5	78.3 (3)
C16—C4—C3—O1	41.1 (2)	C13—C12—C15—F5	−101.4 (3)
N—C4—C3—O1	−84.36 (19)	C11—C12—C15—F6	−39.6 (3)
C16—C4—C3—C2	158.87 (18)	C13—C12—C15—F6	140.7 (2)
N—C4—C3—C2	33.44 (19)	C27—C22—C23—C24	0.4 (4)
C12—C13—C8—C9	0.6 (3)	S—C22—C23—C24	−174.6 (2)
C12—C13—C8—C7	179.56 (19)	C17—C16—C21—C20	−0.6 (3)
C13—C8—C9—C10	0.4 (3)	C4—C16—C21—C20	176.7 (2)
C7—C8—C9—C10	−178.58 (19)	C13—C12—C11—C10	−0.3 (3)
C11—C10—C9—C8	−1.3 (3)	C15—C12—C11—C10	−180.0 (2)
C14—C10—C9—C8	178.9 (2)	C9—C10—C11—C12	1.3 (3)
C5—C1—C2—C6	−81.0 (2)	C14—C10—C11—C12	−178.9 (2)
N—C1—C2—C6	157.94 (18)	C22—C27—C26—C25	0.8 (4)
C5—C1—C2—C3	153.96 (17)	C27—C26—C25—C24	−0.2 (5)
N—C1—C2—C3	32.89 (19)	C27—C26—C25—C28	−179.5 (4)
O1—C3—C2—C1	79.05 (19)	C3—O1—C7—C8	−156.42 (18)
C4—C3—C2—C1	−41.64 (19)	C13—C8—C7—O1	−99.6 (2)
O1—C3—C2—C6	−43.7 (2)	C9—C8—C7—O1	79.4 (3)
C4—C3—C2—C6	−164.39 (19)	C18—C19—C20—C21	−0.3 (5)
C23—C22—C27—C26	−1.0 (4)	C16—C21—C20—C19	0.8 (4)
S—C22—C27—C26	174.09 (19)	C22—C23—C24—C25	0.2 (5)
C9—C10—C14—F2	83.0 (3)	C26—C25—C24—C23	−0.4 (5)
C11—C10—C14—F2	−96.8 (3)	C28—C25—C24—C23	179.0 (4)
C9—C10—C14—F3	−154.7 (2)	C20—C19—C18—C17	−0.3 (4)
C11—C10—C14—F3	25.5 (3)	C16—C17—C18—C19	0.5 (4)
C9—C10—C14—F1	−37.6 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H02···O3^{i}	0.90	1.95	2.801 (2)	158
N—H01···O4^{ii}	0.90	1.88	2.771 (2)	173

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