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6-Methoxymethoxy-7-trifluoromethyl-1,3-benzodioxole-5-carbaldehyde

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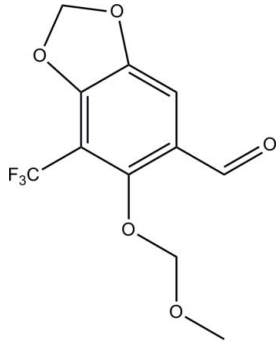
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.041; wR factor = 0.122; data-to-parameter ratio = 10.7.

The title compound, $C_{11}H_9F_3O_5$, crystallizes with three molecules in the asymmetric unit. One $-CF_3$ group is disordered by rotation, with the F atoms split over two sets of sites with occupancies which converged to 0.888 (6) and 0.112 (6). Weak π–π interactions are observed between adjacent benzene rings [the shortest centroid–centroid distance is 3.8858 (4) Å], resulting in the formation of a supramolecular chain along [100].

Related literature

For the enhancement of the biological activity of F and CF_3 containing molecules compared to their unfluorinated analogues, see: Vrábel *et al.* (2007); Wilson & Danishefsky (2010); Bravo *et al.* (1994); Jung *et al.* (2002). For the synthesis of the title compound, an intermediate for the synthesis of biologically active compounds, see: Corey *et al.* (1996); Weeratunga *et al.* (1987); Wu *et al.* (2004).



Experimental

Crystal data

$C_{11}H_9F_3O_5$
M_r = 278.18
Triclinic, $P\overline{1}$
a = 9.3608 (10) Å
b = 12.6895 (14) Å
c = 14.6765 (16) Å
α = 97.007 (2)°
β = 93.625 (2)°
γ = 102.983 (2)°
V = 1678.7 (3) Å^3
Z = 6
Mo $K\alpha$ radiation
μ = 0.16 mm^{-1}
T = 173 K
0.10 × 0.10 × 0.10 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.984, T_{max} = 0.984
8355 measured reflections
5842 independent reflections
4928 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.122
S = 1.03
5842 reflections
545 parameters
18 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2008); software used to prepare material for publication: *publCIF* (Westrip, 2010).

The data were collected at the School of Chemistry and Chemical Engineering, Shandong University. This work was supported financially by the Shandong Provincial Doctoral Foundation, China (grant No. 21310004021142).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2420).

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supplementary materials

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6-Methoxymethoxy-7-trifluoromethyl-1,3-benzodioxole-5-carbaldehyde

Yanfeng Wang, Xinhui Pan, Dexiu Liu and Hong-Xiang Lou

Comment

In recent years, fluorinated compounds have been very important in the pharmaceutical field. Incorporation of a F atom instead of an H atom can alter the biological activity of a compound. Introduction of further F atoms in a CF_3 group provides better lipophilicity, and the compounds might be pharmacologically more interesting compared to their non-fluorinated analogues (Vrábel *et al.*, 2007). Many heterocyclic compounds, which bear the trifluoromethyl group, are far superior to the parent compounds regarding their biological activity (Wilson & Danishefsky, 2010), including herbicides (Bravo *et al.*, 1994) and fungicides (Jung *et al.*, 2002). We report here the crystal structure of the title compound, which is used as an important starting material for the synthesis of a natural product derivative.

Experimental

The title compound was synthesized according to literature methods (Corey *et al.*, 1996; Wu *et al.*, 2004; Weeratunga *et al.*, 1987). The resulting product was purified by column chromatography (silica gel, 200–300 meshes, eluent petroleum ether/ethyl acetate 10:1). The solid residue was crystallized by slow evaporation of an hexane/ethyl acetate (5:1) solution at room temperature. ^{1}H-NMR (600 MHz, $CDCl_3$) δ 3.62 (s, 3H, OCH_3), 5.09 (s, 2H, CH_2), 6.16 (s, 2H, $ArOCH_2$), 7.42 (s, 1H, ArH), 10.14 (s, 1H, CHO); ^{13}C-NMR (150 MHz, $CDCl_3$) δ 58.11, 102, 103, 108, 123, 125, 145, 151, 155, 188; HRMS (EI^+) m/*z*: calcd. for $C_{11}H_9F_3O_5$ (M^+): 279.19, found: 279.19.

Refinement

All H atoms were placed geometrically and treated as riding on their parent atoms with C—H = 0.98 (methyl), 0.95 (aromatic) or 0.99 Å (methylene), and $U_{iso}(H) = x\,U_{eq}(C)$, with x = 1.5 for methyl groups and 1.2 for other H atoms. The CF_3 group is disordered by rotation, and the F atoms are split over two sets of sites with occupancies of 0.888 (6) and 0.112 (6). Restraints were applied on these atoms.

Computing details

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2008); software used to prepare material for publication: *publCIF* (Westrip, 2010).

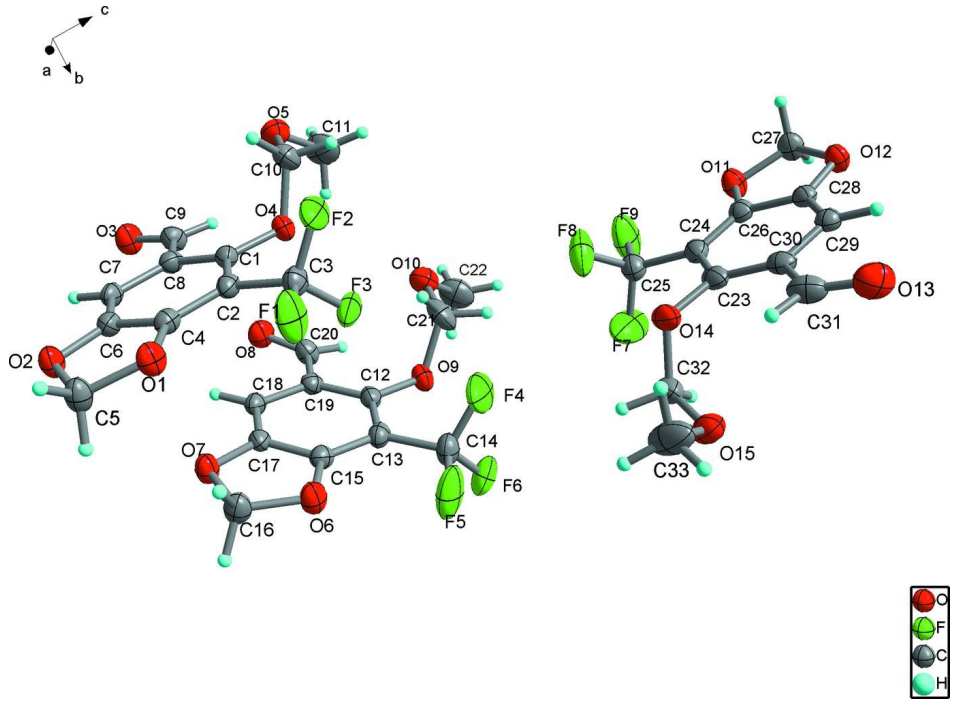

Figure 1

The asymmetric unit view of the title compound with displacement ellipsoids drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii.

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Crystal data

$C_{11}H_9F_3O_5$
$M_r = 278.18$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 9.3608$ (10) Å
$b = 12.6895$ (14) Å
$c = 14.6765$ (16) Å
$\alpha = 97.007$ (2)°
$\beta = 93.625$ (2)°
$\gamma = 102.983$ (2)°
$V = 1678.7$ (3) Å^3
$Z = 6$
$F(000) = 852$
$D_x = 1.651$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4637 reflections
$\theta = 2.8$–27.5°
$\mu = 0.16$ mm^{-1}
$T = 173$ K
Block, colourless
0.10 × 0.10 × 0.10 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.984$, $T_{max} = 0.984$
8355 measured reflections
5842 independent reflections
4928 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.7°$
$h = -10 \rightarrow 11$
$k = -15 \rightarrow 10$
$l = -17 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.122$
$S = 1.03$
5842 reflections
545 parameters
18 restraints
0 constraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0634P)^2 + 0.6455P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.4148 (2)	0.44220 (14)	0.24627 (12)	0.0258 (4)	
C2	0.5685 (2)	0.46616 (14)	0.26506 (12)	0.0259 (4)	
C3	0.6377 (2)	0.50456 (16)	0.36188 (13)	0.0322 (4)	
C4	0.6498 (2)	0.45487 (14)	0.19079 (12)	0.0255 (4)	
C5	0.8251 (2)	0.46037 (18)	0.09341 (13)	0.0340 (4)	
H5A	0.8685	0.5324	0.0751	0.041*	
H5B	0.8944	0.4126	0.0825	0.041*	
C6	0.5815 (2)	0.41995 (14)	0.10140 (12)	0.0253 (4)	
C7	0.4337 (2)	0.39694 (14)	0.08251 (12)	0.0254 (4)	
H7	0.3894	0.3732	0.0212	0.030*	
C8	0.3471 (2)	0.40933 (14)	0.15670 (12)	0.0261 (4)	
C9	0.1878 (2)	0.39213 (16)	0.13658 (13)	0.0325 (4)	
H9	0.1311	0.4092	0.1854	0.039*	
C10	0.2684 (2)	0.36310 (17)	0.36009 (14)	0.0368 (5)	
H10A	0.3165	0.3032	0.3405	0.044*	
H10B	0.2849	0.3801	0.4281	0.044*	
C11	0.0396 (3)	0.4048 (2)	0.3714 (2)	0.0561 (7)	
H11A	−0.0659	0.3744	0.3546	0.084*	
H11B	0.0701	0.4737	0.3463	0.084*	
H11C	0.0591	0.4183	0.4387	0.084*	
C12	0.4460 (2)	0.79048 (15)	0.33160 (12)	0.0260 (4)	
C13	0.6003 (2)	0.82136 (15)	0.34819 (12)	0.0278 (4)	
C14	0.6724 (2)	0.88593 (18)	0.43803 (14)	0.0377 (5)	
C15	0.6789 (2)	0.78481 (15)	0.27987 (12)	0.0261 (4)	
C16	0.8521 (2)	0.74583 (17)	0.19183 (14)	0.0350 (5)	
H16A	0.9085	0.7991	0.1557	0.042*	
H16B	0.9093	0.6907	0.2018	0.042*	
C17	0.6090 (2)	0.72025 (14)	0.19871 (12)	0.0242 (4)	
C18	0.4606 (2)	0.69090 (14)	0.18152 (12)	0.0257 (4)	
H18	0.4149	0.6471	0.1256	0.031*	
C19	0.3766 (2)	0.72763 (14)	0.24954 (12)	0.0253 (4)	
C20	0.2158 (2)	0.70242 (17)	0.23068 (13)	0.0327 (4)	
H20	0.1615	0.7387	0.2713	0.039*	
C21	0.3062 (3)	0.7483 (2)	0.45855 (15)	0.0446 (6)	
H21A	0.3602	0.6894	0.4546	0.054*	

H21B	0.3214	0.7851	0.5230	0.054*	
C22	0.0704 (3)	0.7777 (2)	0.4588 (2)	0.0585 (7)	
H22A	−0.0336	0.7405	0.4440	0.088*	
H22B	0.0947	0.8390	0.4234	0.088*	
H22C	0.0887	0.8052	0.5248	0.088*	
C23	0.5757 (2)	0.86851 (15)	0.85712 (13)	0.0308 (4)	
C24	0.4215 (2)	0.85065 (15)	0.84339 (13)	0.0306 (4)	
C25	0.3445 (3)	0.84013 (18)	0.74898 (14)	0.0396 (5)	
C26	0.3451 (2)	0.84472 (14)	0.92068 (13)	0.0281 (4)	
C27	0.1749 (2)	0.82327 (18)	1.02165 (13)	0.0353 (5)	
H27A	0.1154	0.8752	1.0431	0.042*	
H27B	0.1220	0.7488	1.0302	0.042*	
C28	0.4178 (2)	0.85820 (14)	1.00856 (12)	0.0263 (4)	
C29	0.5655 (2)	0.87629 (14)	1.02279 (13)	0.0282 (4)	
H29	0.6132	0.8862	1.0834	0.034*	
C30	0.6474 (2)	0.88013 (15)	0.94515 (13)	0.0300 (4)	
C31	0.8066 (2)	0.89239 (17)	0.96167 (17)	0.0410 (5)	
H31	0.8616	0.8853	0.9099	0.049*	
C32	0.7103 (3)	0.97212 (17)	0.75019 (14)	0.0423 (5)	
H32A	0.7049	0.9627	0.6820	0.051*	
H32B	0.6487	1.0232	0.7702	0.051*	
C33	0.9564 (3)	0.9601 (2)	0.7452 (2)	0.0651 (8)	
H33A	1.0551	0.9917	0.7766	0.098*	
H33B	0.9573	0.9680	0.6796	0.098*	
H33C	0.9271	0.8825	0.7519	0.098*	
F1	0.78370 (14)	0.52599 (14)	0.36687 (9)	0.0609 (4)	
F2	0.59564 (16)	0.43130 (10)	0.41881 (8)	0.0491 (3)	
F3	0.60334 (15)	0.59570 (9)	0.39913 (8)	0.0439 (3)	
F4	0.63938 (19)	0.83087 (13)	0.50907 (9)	0.0677 (5)	
F5	0.81807 (15)	0.91193 (15)	0.44024 (11)	0.0737 (5)	
F6	0.63210 (16)	0.97844 (11)	0.45818 (9)	0.0548 (4)	
F7	0.3831 (3)	0.93019 (15)	0.71022 (16)	0.0706 (9)	0.888 (6)
F7B	0.302 (3)	0.9299 (16)	0.7436 (13)	0.074 (6)	0.112 (6)
F8	0.3705 (3)	0.7592 (2)	0.69059 (10)	0.0583 (7)	0.888 (6)
F8B	0.431 (2)	0.827 (2)	0.6842 (10)	0.067 (5)	0.112 (6)
F9	0.1981 (2)	0.8201 (3)	0.74828 (13)	0.0635 (7)	0.888 (6)
F9B	0.231 (2)	0.7631 (17)	0.7437 (11)	0.059 (5)	0.112 (6)
O1	0.79772 (14)	0.47249 (11)	0.18950 (9)	0.0330 (3)	
O2	0.68690 (14)	0.41280 (11)	0.04150 (9)	0.0324 (3)	
O3	0.12449 (15)	0.35701 (13)	0.06032 (10)	0.0409 (4)	
O4	0.33447 (15)	0.45868 (11)	0.31963 (9)	0.0315 (3)	
O5	0.12040 (16)	0.32932 (12)	0.33439 (10)	0.0430 (4)	
O6	0.82681 (14)	0.80063 (12)	0.27905 (9)	0.0368 (3)	
O7	0.71247 (14)	0.69427 (11)	0.14359 (9)	0.0320 (3)	
O8	0.14850 (15)	0.63790 (12)	0.16616 (10)	0.0402 (4)	
O9	0.36529 (15)	0.82610 (11)	0.39864 (9)	0.0332 (3)	
O10	0.15914 (18)	0.70333 (13)	0.43583 (11)	0.0484 (4)	
O11	0.19842 (15)	0.82781 (12)	0.92587 (9)	0.0376 (3)	
O12	0.31665 (14)	0.85179 (11)	1.07222 (9)	0.0321 (3)	

O13	0.87177 (16)	0.91108 (14)	1.03801 (12)	0.0492 (4)
O14	0.65266 (17)	0.86765 (11)	0.78023 (10)	0.0403 (4)
O15	0.85359 (18)	1.01619 (12)	0.78551 (11)	0.0475 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0318 (10)	0.0228 (9)	0.0231 (9)	0.0070 (7)	0.0041 (7)	0.0030 (7)
C2	0.0323 (10)	0.0223 (9)	0.0223 (9)	0.0061 (7)	−0.0012 (7)	0.0028 (7)
C3	0.0376 (11)	0.0331 (10)	0.0251 (10)	0.0095 (9)	−0.0019 (8)	0.0012 (8)
C4	0.0261 (9)	0.0228 (9)	0.0270 (9)	0.0049 (7)	−0.0013 (7)	0.0040 (7)
C5	0.0247 (10)	0.0436 (12)	0.0313 (10)	0.0075 (9)	0.0026 (8)	−0.0035 (9)
C6	0.0287 (10)	0.0223 (9)	0.0237 (9)	0.0041 (7)	0.0028 (7)	0.0018 (7)
C7	0.0296 (10)	0.0237 (9)	0.0208 (9)	0.0040 (7)	−0.0016 (7)	0.0010 (7)
C8	0.0269 (10)	0.0233 (9)	0.0266 (9)	0.0032 (7)	0.0006 (7)	0.0037 (7)
C9	0.0303 (10)	0.0374 (11)	0.0290 (10)	0.0057 (8)	0.0030 (8)	0.0060 (8)
C10	0.0416 (12)	0.0398 (11)	0.0304 (10)	0.0076 (9)	0.0090 (9)	0.0113 (9)
C11	0.0421 (14)	0.0574 (15)	0.0715 (17)	0.0114 (12)	0.0225 (12)	0.0110 (13)
C12	0.0320 (10)	0.0260 (9)	0.0216 (9)	0.0095 (8)	0.0053 (7)	0.0037 (7)
C13	0.0333 (10)	0.0260 (9)	0.0229 (9)	0.0066 (8)	−0.0004 (8)	0.0012 (7)
C14	0.0393 (12)	0.0420 (12)	0.0280 (10)	0.0080 (9)	−0.0018 (9)	−0.0049 (9)
C15	0.0258 (9)	0.0259 (9)	0.0257 (9)	0.0040 (7)	0.0004 (7)	0.0049 (7)
C16	0.0259 (10)	0.0394 (11)	0.0363 (11)	0.0050 (8)	0.0046 (8)	−0.0034 (9)
C17	0.0291 (10)	0.0234 (9)	0.0203 (9)	0.0055 (7)	0.0047 (7)	0.0036 (7)
C18	0.0293 (10)	0.0244 (9)	0.0219 (9)	0.0039 (7)	−0.0008 (7)	0.0029 (7)
C19	0.0264 (9)	0.0266 (9)	0.0234 (9)	0.0058 (7)	0.0024 (7)	0.0061 (7)
C20	0.0291 (10)	0.0427 (11)	0.0270 (10)	0.0087 (9)	0.0045 (8)	0.0059 (9)
C21	0.0489 (14)	0.0646 (15)	0.0299 (11)	0.0241 (12)	0.0136 (10)	0.0188 (10)
C22	0.0427 (14)	0.0707 (18)	0.0671 (17)	0.0150 (13)	0.0168 (12)	0.0199 (14)
C23	0.0410 (11)	0.0205 (9)	0.0317 (10)	0.0068 (8)	0.0114 (8)	0.0032 (8)
C24	0.0410 (11)	0.0240 (9)	0.0263 (10)	0.0075 (8)	0.0019 (8)	0.0023 (7)
C25	0.0499 (14)	0.0379 (12)	0.0304 (11)	0.0103 (10)	0.0011 (10)	0.0043 (9)
C26	0.0305 (10)	0.0224 (9)	0.0310 (10)	0.0064 (7)	0.0004 (8)	0.0033 (7)
C27	0.0286 (10)	0.0443 (12)	0.0326 (10)	0.0080 (9)	0.0039 (8)	0.0044 (9)
C28	0.0310 (10)	0.0208 (9)	0.0268 (9)	0.0049 (7)	0.0036 (8)	0.0037 (7)
C29	0.0322 (10)	0.0222 (9)	0.0293 (10)	0.0044 (8)	−0.0004 (8)	0.0058 (7)
C30	0.0325 (10)	0.0214 (9)	0.0362 (11)	0.0048 (8)	0.0055 (8)	0.0060 (8)
C31	0.0351 (11)	0.0374 (12)	0.0532 (14)	0.0076 (9)	0.0125 (10)	0.0149 (10)
C32	0.0579 (14)	0.0348 (11)	0.0305 (11)	0.0005 (10)	0.0124 (10)	0.0057 (9)
C33	0.0581 (16)	0.0467 (14)	0.086 (2)	0.0018 (12)	0.0384 (15)	−0.0041 (14)
F1	0.0348 (7)	0.1043 (12)	0.0347 (7)	0.0138 (7)	−0.0097 (6)	−0.0128 (7)
F2	0.0797 (10)	0.0418 (7)	0.0255 (6)	0.0151 (7)	−0.0074 (6)	0.0086 (5)
F3	0.0665 (9)	0.0327 (6)	0.0298 (6)	0.0150 (6)	−0.0069 (6)	−0.0059 (5)
F4	0.1021 (12)	0.0654 (10)	0.0266 (7)	0.0079 (9)	−0.0151 (7)	0.0041 (6)
F5	0.0385 (8)	0.1045 (13)	0.0585 (9)	0.0084 (8)	−0.0117 (7)	−0.0416 (9)
F6	0.0660 (9)	0.0422 (7)	0.0490 (8)	0.0134 (7)	−0.0026 (7)	−0.0187 (6)
F7	0.100 (2)	0.0513 (11)	0.0553 (13)	0.0039 (11)	−0.0237 (12)	0.0275 (9)
F7B	0.090 (10)	0.087 (9)	0.060 (8)	0.041 (8)	0.001 (7)	0.027 (7)
F8	0.0898 (16)	0.0586 (14)	0.0277 (8)	0.0307 (12)	−0.0052 (8)	−0.0091 (8)
F8B	0.071 (8)	0.080 (10)	0.044 (7)	0.015 (7)	−0.002 (6)	−0.001 (6)

F9	0.0491 (11)	0.102 (2)	0.0359 (9)	0.0194 (12)	−0.0100 (7)	−0.0001 (11)
F9B	0.042 (7)	0.065 (9)	0.056 (7)	−0.011 (6)	−0.016 (6)	0.012 (7)
O1	0.0248 (7)	0.0443 (8)	0.0283 (7)	0.0074 (6)	−0.0019 (5)	0.0019 (6)
O2	0.0266 (7)	0.0425 (8)	0.0255 (7)	0.0059 (6)	0.0037 (5)	−0.0018 (6)
O3	0.0273 (7)	0.0540 (9)	0.0351 (8)	0.0020 (7)	−0.0033 (6)	−0.0017 (7)
O4	0.0358 (7)	0.0327 (7)	0.0260 (7)	0.0067 (6)	0.0094 (6)	0.0026 (5)
O5	0.0402 (9)	0.0381 (8)	0.0459 (9)	0.0005 (7)	0.0076 (7)	0.0018 (7)
O6	0.0254 (7)	0.0481 (9)	0.0310 (7)	0.0023 (6)	0.0004 (6)	−0.0042 (6)
O7	0.0250 (7)	0.0414 (8)	0.0275 (7)	0.0067 (6)	0.0041 (5)	−0.0026 (6)
O8	0.0291 (7)	0.0477 (9)	0.0380 (8)	0.0017 (6)	−0.0017 (6)	−0.0002 (7)
O9	0.0367 (8)	0.0392 (8)	0.0259 (7)	0.0136 (6)	0.0089 (6)	0.0018 (6)
O10	0.0526 (10)	0.0443 (9)	0.0478 (9)	0.0061 (7)	0.0148 (8)	0.0091 (7)
O11	0.0298 (7)	0.0524 (9)	0.0307 (7)	0.0103 (6)	−0.0004 (6)	0.0070 (6)
O12	0.0287 (7)	0.0409 (8)	0.0264 (7)	0.0072 (6)	0.0041 (5)	0.0044 (6)
O13	0.0317 (8)	0.0565 (10)	0.0582 (11)	0.0059 (7)	−0.0002 (7)	0.0138 (8)
O14	0.0552 (9)	0.0282 (7)	0.0364 (8)	0.0045 (6)	0.0207 (7)	0.0017 (6)
O15	0.0506 (10)	0.0347 (8)	0.0508 (9)	−0.0011 (7)	0.0165 (8)	−0.0044 (7)

Geometric parameters (Å, º)

C1—O4	1.373 (2)	C18—C19	1.408 (3)
C1—C8	1.395 (3)	C18—H18	0.9500
C1—C2	1.404 (3)	C19—C20	1.467 (3)
C2—C4	1.380 (3)	C20—O8	1.212 (2)
C2—C3	1.500 (2)	C20—H20	0.9500
C3—F1	1.327 (2)	C21—O10	1.369 (3)
C3—F3	1.332 (2)	C21—O9	1.443 (2)
C3—F2	1.336 (2)	C21—H21A	0.9900
C4—O1	1.354 (2)	C21—H21B	0.9900
C4—C6	1.397 (3)	C22—O10	1.416 (3)
C5—O2	1.425 (2)	C22—H22A	0.9800
C5—O1	1.445 (2)	C22—H22B	0.9800
C5—H5A	0.9900	C22—H22C	0.9800
C5—H5B	0.9900	C23—O14	1.377 (2)
C6—C7	1.352 (3)	C23—C30	1.393 (3)
C6—O2	1.373 (2)	C23—C24	1.407 (3)
C7—C8	1.412 (3)	C24—C26	1.379 (3)
C7—H7	0.9500	C24—C25	1.497 (3)
C8—C9	1.464 (3)	C25—F9B	1.266 (15)
C9—O3	1.216 (2)	C25—F8B	1.304 (16)
C9—H9	0.9500	C25—F7B	1.300 (18)
C10—O5	1.368 (2)	C25—F7	1.326 (3)
C10—O4	1.446 (2)	C25—F9	1.336 (3)
C10—H10A	0.9900	C25—F8	1.331 (3)
C10—H10B	0.9900	C26—O11	1.350 (2)
C11—O5	1.427 (3)	C26—C28	1.392 (3)
C11—H11A	0.9800	C27—O12	1.427 (2)
C11—H11B	0.9800	C27—O11	1.443 (2)
C11—H11C	0.9800	C27—H27A	0.9900
C12—O9	1.372 (2)	C27—H27B	0.9900

C12—C19	1.394 (3)	C28—C29	1.348 (3)
C12—C13	1.406 (3)	C28—O12	1.368 (2)
C13—C15	1.377 (3)	C29—C30	1.413 (3)
C13—C14	1.497 (3)	C29—H29	0.9500
C14—F6	1.318 (3)	C30—C31	1.465 (3)
C14—F5	1.326 (3)	C31—O13	1.211 (3)
C14—F4	1.339 (3)	C31—H31	0.9500
C15—O6	1.355 (2)	C32—O15	1.373 (3)
C15—C17	1.393 (3)	C32—O14	1.447 (2)
C16—O7	1.422 (2)	C32—H32A	0.9900
C16—O6	1.440 (2)	C32—H32B	0.9900
C16—H16A	0.9900	C33—O15	1.435 (3)
C16—H16B	0.9900	C33—H33A	0.9800
C17—C18	1.353 (3)	C33—H33B	0.9800
C17—O7	1.371 (2)	C33—H33C	0.9800

O4—C1—C8	121.40 (16)	O8—C20—C19	123.64 (18)
O4—C1—C2	116.80 (15)	O8—C20—H20	118.2
C8—C1—C2	121.70 (16)	C19—C20—H20	118.2
C4—C2—C1	116.89 (16)	O10—C21—O9	112.04 (17)
C4—C2—C3	122.85 (17)	O10—C21—H21A	109.2
C1—C2—C3	120.25 (16)	O9—C21—H21A	109.2
F1—C3—F3	106.24 (16)	O10—C21—H21B	109.2
F1—C3—F2	106.72 (16)	O9—C21—H21B	109.2
F3—C3—F2	105.70 (16)	H21A—C21—H21B	107.9
F1—C3—C2	112.31 (16)	O10—C22—H22A	109.5
F3—C3—C2	112.74 (16)	O10—C22—H22B	109.5
F2—C3—C2	112.61 (16)	H22A—C22—H22B	109.5
O1—C4—C2	128.95 (16)	O10—C22—H22C	109.5
O1—C4—C6	109.80 (16)	H22A—C22—H22C	109.5
C2—C4—C6	121.24 (17)	H22B—C22—H22C	109.5
O2—C5—O1	107.09 (14)	O14—C23—C30	120.96 (18)
O2—C5—H5A	110.3	O14—C23—C24	117.75 (17)
O1—C5—H5A	110.3	C30—C23—C24	121.18 (17)
O2—C5—H5B	110.3	C26—C24—C23	117.07 (17)
O1—C5—H5B	110.3	C26—C24—C25	121.70 (18)
H5A—C5—H5B	108.6	C23—C24—C25	121.22 (18)
C7—C6—O2	128.38 (16)	F9B—C25—F8B	113.5 (12)
C7—C6—C4	122.35 (17)	F9B—C25—F7B	107.7 (12)
O2—C6—C4	109.26 (16)	F8B—C25—F7B	109.0 (12)
C6—C7—C8	117.95 (16)	F7—C25—F9	105.5 (2)
C6—C7—H7	121.0	F7—C25—F8	106.5 (2)
C8—C7—H7	121.0	F9—C25—F8	105.2 (2)
C1—C8—C7	119.84 (17)	F7—C25—C24	112.52 (18)
C1—C8—C9	121.52 (17)	F9—C25—C24	113.25 (18)
C7—C8—C9	118.57 (16)	F8—C25—C24	113.20 (18)
O3—C9—C8	123.04 (18)	O11—C26—C24	128.49 (17)
O3—C9—H9	118.5	O11—C26—C28	110.13 (16)
C8—C9—H9	118.5	C24—C26—C28	121.38 (18)

O5—C10—O4	111.60 (16)	O12—C27—O11	106.96 (15)
O5—C10—H10A	109.3	O12—C27—H27A	110.3
O4—C10—H10A	109.3	O11—C27—H27A	110.3
O5—C10—H10B	109.3	O12—C27—H27B	110.3
O4—C10—H10B	109.3	O11—C27—H27B	110.3
H10A—C10—H10B	108.0	H27A—C27—H27B	108.6
O5—C11—H11A	109.5	C29—C28—O12	128.65 (17)
O5—C11—H11B	109.5	C29—C28—C26	122.17 (17)
H11A—C11—H11B	109.5	O12—C28—C26	109.17 (16)
O5—C11—H11C	109.5	C28—C29—C30	118.16 (17)
H11A—C11—H11C	109.5	C28—C29—H29	120.9
H11B—C11—H11C	109.5	C30—C29—H29	120.9
O9—C12—C19	120.80 (16)	C23—C30—C29	120.00 (18)
O9—C12—C13	117.59 (16)	C23—C30—C31	122.72 (18)
C19—C12—C13	121.61 (16)	C29—C30—C31	117.23 (18)
C15—C13—C12	116.53 (16)	O13—C31—C30	123.3 (2)
C15—C13—C14	122.72 (17)	O13—C31—H31	118.4
C12—C13—C14	120.69 (17)	C30—C31—H31	118.4
F6—C14—F5	106.30 (18)	O15—C32—O14	111.72 (18)
F6—C14—F4	105.33 (17)	O15—C32—H32A	109.3
F5—C14—F4	106.69 (18)	O14—C32—H32A	109.3
F6—C14—C13	113.57 (17)	O15—C32—H32B	109.3
F5—C14—C13	112.56 (17)	O14—C32—H32B	109.3
F4—C14—C13	111.84 (17)	H32A—C32—H32B	107.9
O6—C15—C13	128.50 (16)	O15—C33—H33A	109.5
O6—C15—C17	109.80 (16)	O15—C33—H33B	109.5
C13—C15—C17	121.68 (17)	H33A—C33—H33B	109.5
O7—C16—O6	107.78 (15)	O15—C33—H33C	109.5
O7—C16—H16A	110.2	H33A—C33—H33C	109.5
O6—C16—H16A	110.2	H33B—C33—H33C	109.5
O7—C16—H16B	110.2	C4—O1—C5	106.13 (13)
O6—C16—H16B	110.2	C6—O2—C5	106.07 (14)
H16A—C16—H16B	108.5	C1—O4—C10	116.60 (14)
C18—C17—O7	128.01 (16)	C10—O5—C11	112.88 (17)
C18—C17—C15	122.36 (17)	C15—O6—C16	106.43 (14)
O7—C17—C15	109.63 (15)	C17—O7—C16	106.33 (14)
C17—C18—C19	117.56 (16)	C12—O9—C21	116.13 (15)
C17—C18—H18	121.2	C21—O10—C22	112.81 (19)
C19—C18—H18	121.2	C26—O11—C27	106.53 (14)
C12—C19—C18	120.25 (16)	C28—O12—C27	106.64 (14)
C12—C19—C20	120.90 (17)	C23—O14—C32	116.82 (15)
C18—C19—C20	118.80 (16)	C32—O15—C33	113.45 (18)

O4—C1—C2—C4	−176.86 (15)	C26—C24—C25—F9B	−42.5 (13)
C8—C1—C2—C4	−0.5 (3)	C23—C24—C25—F9B	138.4 (12)
O4—C1—C2—C3	1.8 (2)	C26—C24—C25—F8B	−167.6 (12)
C8—C1—C2—C3	178.13 (16)	C23—C24—C25—F8B	13.2 (12)
C4—C2—C3—F1	0.1 (3)	C26—C24—C25—F7B	72.8 (13)
C1—C2—C3—F1	−178.42 (17)	C23—C24—C25—F7B	−106.4 (13)

C4—C2—C3—F3	120.10 (19)	C26—C24—C25—F7	116.7 (3)
C1—C2—C3—F3	−58.4 (2)	C23—C24—C25—F7	−62.4 (3)
C4—C2—C3—F2	−120.39 (19)	C26—C24—C25—F9	−2.8 (3)
C1—C2—C3—F2	61.1 (2)	C23—C24—C25—F9	178.0 (2)
C1—C2—C4—O1	179.94 (17)	C26—C24—C25—F8	−122.5 (2)
C3—C2—C4—O1	1.4 (3)	C23—C24—C25—F8	58.3 (3)
C1—C2—C4—C6	−0.7 (3)	C23—C24—C26—O11	−179.62 (17)
C3—C2—C4—C6	−179.32 (17)	C25—C24—C26—O11	1.2 (3)
O1—C4—C6—C7	−179.61 (16)	C23—C24—C26—C28	1.4 (3)
C2—C4—C6—C7	0.9 (3)	C25—C24—C26—C28	−177.84 (17)
O1—C4—C6—O2	1.2 (2)	O11—C26—C28—C29	179.98 (16)
C2—C4—C6—O2	−178.25 (16)	C24—C26—C28—C29	−0.8 (3)
O2—C6—C7—C8	179.14 (17)	O11—C26—C28—O12	−1.0 (2)
C4—C6—C7—C8	0.1 (3)	C24—C26—C28—O12	178.14 (16)
O4—C1—C8—C7	177.74 (16)	O12—C28—C29—C30	−179.55 (17)
C2—C1—C8—C7	1.5 (3)	C26—C28—C29—C30	−0.8 (3)
O4—C1—C8—C9	0.7 (3)	O14—C23—C30—C29	−177.40 (16)
C2—C1—C8—C9	−175.48 (17)	C24—C23—C30—C29	−1.3 (3)
C6—C7—C8—C1	−1.3 (3)	O14—C23—C30—C31	0.2 (3)
C6—C7—C8—C9	175.79 (16)	C24—C23—C30—C31	176.32 (17)
C1—C8—C9—O3	−175.27 (18)	C28—C29—C30—C23	1.8 (3)
C7—C8—C9—O3	7.7 (3)	C28—C29—C30—C31	−175.92 (17)
O9—C12—C13—C15	−179.89 (15)	C23—C30—C31—O13	174.0 (2)
C19—C12—C13—C15	−1.0 (3)	C29—C30—C31—O13	−8.3 (3)
O9—C12—C13—C14	3.1 (3)	C2—C4—O1—C5	−173.78 (18)
C19—C12—C13—C14	−178.07 (17)	C6—C4—O1—C5	6.8 (2)
C15—C13—C14—F6	126.8 (2)	O2—C5—O1—C4	−12.1 (2)
C12—C13—C14—F6	−56.4 (3)	C7—C6—O2—C5	172.08 (18)
C15—C13—C14—F5	5.9 (3)	C4—C6—O2—C5	−8.79 (19)
C12—C13—C14—F5	−177.21 (18)	O1—C5—O2—C6	12.8 (2)
C15—C13—C14—F4	−114.2 (2)	C8—C1—O4—C10	83.8 (2)
C12—C13—C14—F4	62.7 (2)	C2—C1—O4—C10	−99.78 (19)
C12—C13—C15—O6	−178.52 (17)	O5—C10—O4—C1	−103.73 (19)
C14—C13—C15—O6	−1.5 (3)	O4—C10—O5—C11	−67.4 (2)
C12—C13—C15—C17	−0.2 (3)	C13—C15—O6—C16	−179.92 (19)
C14—C13—C15—C17	176.76 (18)	C17—C15—O6—C16	1.6 (2)
O6—C15—C17—C18	179.50 (16)	O7—C16—O6—C15	−2.1 (2)
C13—C15—C17—C18	0.9 (3)	C18—C17—O7—C16	179.16 (18)
O6—C15—C17—O7	−0.6 (2)	C15—C17—O7—C16	−0.75 (19)
C13—C15—C17—O7	−179.15 (16)	O6—C16—O7—C17	1.7 (2)
O7—C17—C18—C19	179.76 (16)	C19—C12—O9—C21	81.4 (2)
C15—C17—C18—C19	−0.3 (3)	C13—C12—O9—C21	−99.8 (2)
O9—C12—C19—C18	−179.54 (15)	O10—C21—O9—C12	−102.7 (2)
C13—C12—C19—C18	1.6 (3)	O9—C21—O10—C22	−71.9 (2)
O9—C12—C19—C20	3.1 (3)	C24—C26—O11—C27	177.13 (19)
C13—C12—C19—C20	−175.78 (17)	C28—C26—O11—C27	−3.8 (2)
C17—C18—C19—C12	−0.9 (3)	O12—C27—O11—C26	7.0 (2)
C17—C18—C19—C20	176.55 (16)	C29—C28—O12—C27	−175.68 (19)
C12—C19—C20—O8	−170.82 (18)	C26—C28—O12—C27	5.4 (2)

C18—C19—C20—O8	11.7 (3)	O11—C27—O12—C28	−7.6 (2)
O14—C23—C24—C26	175.93 (16)	C30—C23—O14—C32	−89.6 (2)
C30—C23—C24—C26	−0.3 (3)	C24—C23—O14—C32	94.2 (2)
O14—C23—C24—C25	−4.9 (3)	O15—C32—O14—C23	94.5 (2)
C30—C23—C24—C25	178.90 (17)	O14—C32—O15—C33	71.4 (2)