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A new polymorph of 2,6-bis(trifluoromethyl)benzoic acid

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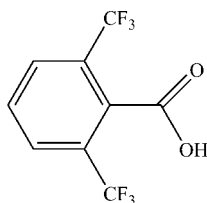
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.048; wR factor = 0.151; data-to-parameter ratio = 10.8.

The asymmetric unit of a second polymorph of the title compound, $\text{C}_9\text{H}_4\text{F}_6\text{O}_2$, contains five independent molecules, which form hydrogen-bonded $\text{O}-\text{H}\cdots\text{O}$ dimers about inversion centers. The most significant structural difference between this structure and that of the first polymorph [Tobin & Masuda (2009). *Acta Cryst.* E65, o1217] is the hydrogen-bonded, dimeric orientation of the carboxylic acid functionalities.

Related literature

For the first polymorph of the title compound, see: Tobin & Masuda (2009). For details of the synthesis, see: Dmowski & Piasecka-Maciejewska (1998). For information on dimeric *versus* catemeric crystal growth in benzoic acids, see: Moorthy *et al.* (2002).



Experimental

Crystal data

$\text{C}_9\text{H}_4\text{F}_6\text{O}_2$
 $M_r = 258.12$
Triclinic, $P\bar{1}$
 $a = 10.312$ (2) Å
 $b = 11.243$ (2) Å
 $c = 21.283$ (4) Å

$\alpha = 79.565$ (3)°
 $\beta = 88.961$ (3)°
 $\gamma = 85.125$ (3)°
 $V = 2418.0$ (9) Å³
 $Z = 10$
Mo $K\alpha$ radiation

$\mu = 0.20$ mm⁻¹
 $T = 100$ K

0.14 × 0.11 × 0.11 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (SADABS; Bruker, 2010)
 $T_{\min} = 0.973$, $T_{\max} = 0.980$

16341 measured reflections
8352 independent reflections
5092 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.046$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.151$
 $S = 0.85$
8352 reflections
771 parameters

6 restraints
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.28$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.29$ e Å⁻³

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

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O2}-\text{H2}\cdots\text{O1}^{\text{i}}$	0.84	1.87	2.701 (4)	172
$\text{O4}-\text{H4A}\cdots\text{O10}^{\text{ii}}$	0.84	1.82	2.657 (3)	174
$\text{O6}-\text{H6A}\cdots\text{O8}^{\text{i}}$	0.84	1.84	2.673 (4)	170
$\text{O7}-\text{H7}\cdots\text{O5}^{\text{i}}$	0.84	1.80	2.638 (4)	173
$\text{O9}-\text{H9}\cdots\text{O3}^{\text{iii}}$	0.84	1.81	2.644 (3)	174

Symmetry codes: (i) $-x + 1, -y + 1, -z + 1$; (ii) $x + 1, y, z$; (iii) $x - 1, y, z$.

Data collection: APEX2 (Bruker, 2010); cell refinement: SAINT (Bruker, 2010); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997) and Mercury (Macrae *et al.*, 2006); software used to prepare material for publication: SHELXTL (Sheldrick, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2403).

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Tobin, J. M. & Masuda, J. D. (2009). *Acta Cryst.* E65, o1217.

supporting information

Acta Cryst. (2011). E67, o1054 [doi:10.1107/S1600536811011731]

A new polymorph of 2,6-bis(trifluoromethyl)benzoic acid

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S1. Comment

Recent studies on benzoic acids have indicated a potential for selective crystal polymorph engineering. It is suggested that catemeric or dimeric structures of benzoic acids can be influenced by concentration and the presence of solvent (Moorthy *et al.*, 2002). Crystallization from solvent free oil led to the formation of the hydrogen bound, dimeric form of the title compound.

The molecular structure of the title compound is presented in Fig. 1. The most significant structural difference between this structure and the literature polymorph (Tobin & Masuda, 2009) is the hydrogen bound, dimeric orientation of the carboxylic acid functionalities. The five molecules in the asymmetric unit are defined by O—H \cdots O hydrogen bonds ranging from 2.638 (4)–2.701 (4) Å and angles of 170–174° (Tab. 1 & Fig. 2).

S2. Experimental

The title compound was prepared following the literature methods (Dmowski & Piasecka-Maciejewska, 1998). The compound was crystallized slowly from the resultant oil forming colorless, block-like crystals.

S3. Refinement

The H atoms were placed in geometrically idealized positions with C—H and O—H distances = 0.95 and 0.98 Å, respectively, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{O})$.

Several short contact distances are found for F(2) & F(4) to F(17), F(8) to F(23), F(9) to F(20), F(10) to F(25), F(13) to F(21) and F(14) to F(27). The short contacts are believed to arise from disorder present in the crystal. Modeling this disorder yielded low occupancy which was detrimental to the integrity of the data set upon refinement. In order to obtain satisfactory thermal parameters the use of DELU restraints were applied to fluorine atoms F(4)>F(6) relative to the adjacent C(8) atom.

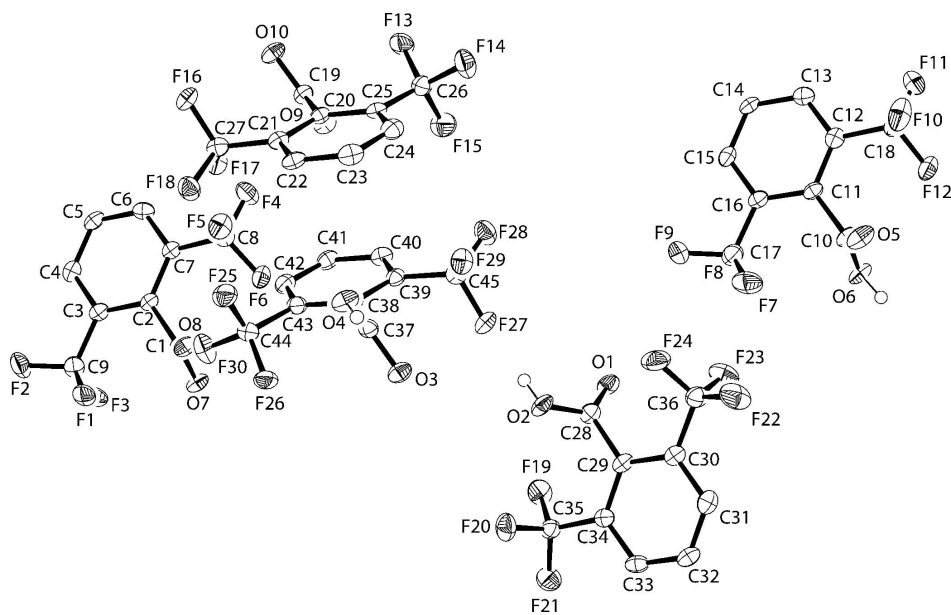


Figure 1

The molecular structure of the title compound, with atom labels and 50% probability displacement ellipsoids for non-H atoms. Aromatic hydrogen atoms are removed for clarity.

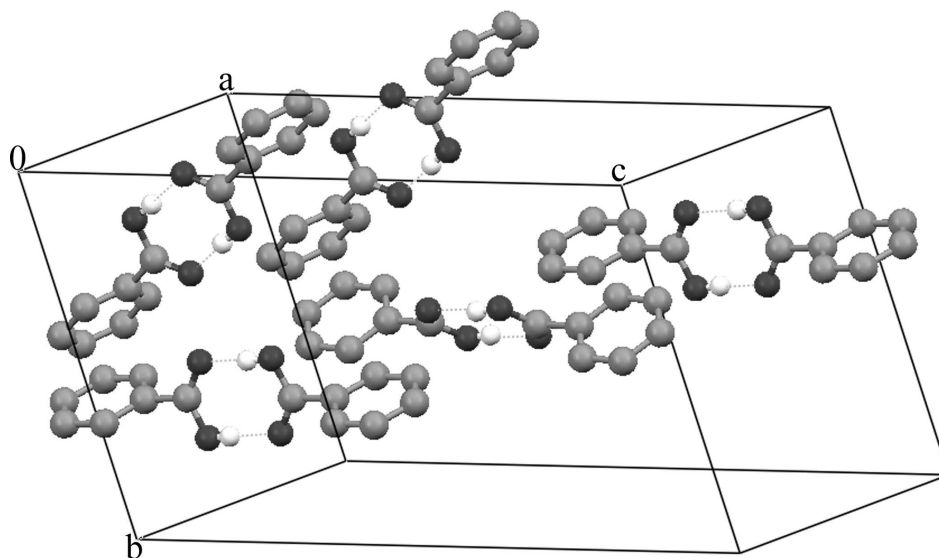


Figure 2

The hydrogen bonding interactions between molecules of the title compound lying about inversion centers. Trifluoromethyl groups and aromatic H atoms are removed for clarity.

2,6-Bis(trifluoromethyl)benzoic acid

Crystal data

$C_9H_4F_6O_2$

$M_r = 258.12$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 10.312\ (2)\ \text{\AA}$

$b = 11.243\ (2)\ \text{\AA}$

$c = 21.283\ (4)\ \text{\AA}$

$\alpha = 79.565\ (3)^\circ$

$\beta = 88.961 (3)^\circ$
 $\gamma = 85.125 (3)^\circ$
 $V = 2418.0 (9) \text{ \AA}^3$
 $Z = 10$
 $F(000) = 1280$
 $D_x = 1.773 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 4371 reflections
 $\theta = 2.2\text{--}27.4^\circ$
 $\mu = 0.20 \text{ mm}^{-1}$
 $T = 100 \text{ K}$
 Block, colourless
 $0.14 \times 0.11 \times 0.11 \text{ mm}$

Data collection

Bruker APEXII CCD
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 2010)
 $T_{\min} = 0.973$, $T_{\max} = 0.980$

16341 measured reflections
 8352 independent reflections
 5092 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.046$
 $\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 1.9^\circ$
 $h = -11 \rightarrow 12$
 $k = -13 \rightarrow 13$
 $l = -25 \rightarrow 25$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.151$
 $S = 0.85$
 8352 reflections
 771 parameters
 6 restraints
 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
 $w = 1/[\sigma^2(F_o^2) + (0.0887P)^2 + 1.2723P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.28 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.29 \text{ e \AA}^{-3}$

Special details

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

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
F1	0.2018 (2)	0.7426 (2)	-0.03174 (10)	0.0326 (5)
C1	0.1804 (4)	0.6502 (3)	0.08995 (16)	0.0210 (8)
O1	0.5747 (2)	0.5485 (2)	0.55641 (12)	0.0268 (6)
F2	0.0386 (2)	0.7786 (2)	-0.09609 (10)	0.0391 (6)
C2	0.0469 (3)	0.6267 (3)	0.06966 (16)	0.0184 (8)
O2	0.6703 (2)	0.4971 (2)	0.46844 (12)	0.0286 (6)
H2	0.5936	0.4894	0.4581	0.043*
F3	0.0525 (2)	0.88431 (19)	-0.02227 (11)	0.0412 (6)
C3	-0.0026 (3)	0.6834 (3)	0.00950 (17)	0.0207 (8)
O3	0.8142 (2)	0.2255 (2)	0.30586 (12)	0.0256 (6)

C4	-0.1242 (4)	0.6592 (3)	-0.00972 (17)	0.0224 (8)
H4	-0.1558	0.6956	-0.0510	0.027*
O4	0.7566 (2)	0.0674 (2)	0.26430 (12)	0.0263 (6)
H4A	0.8359	0.0441	0.2695	0.039*
C5	-0.1996 (4)	0.5822 (3)	0.03096 (17)	0.0227 (8)
H5	-0.2831	0.5665	0.0179	0.027*
O5	0.5766 (2)	0.2035 (2)	0.86826 (13)	0.0313 (6)
C6	-0.1525 (4)	0.5288 (3)	0.09046 (17)	0.0234 (9)
H6	-0.2049	0.4770	0.1185	0.028*
O6	0.4856 (2)	0.3878 (2)	0.87912 (12)	0.0264 (6)
H6A	0.5603	0.3966	0.8917	0.040*
C7	-0.0301 (3)	0.5491 (3)	0.11012 (16)	0.0186 (8)
F7	0.3484 (2)	0.50617 (18)	0.75965 (11)	0.0366 (6)
O7	0.1849 (2)	0.7599 (2)	0.10138 (12)	0.0250 (6)
H7	0.2624	0.7721	0.1080	0.037*
C8	0.0199 (4)	0.4852 (3)	0.17366 (17)	0.0231 (6)
F8	0.4944 (2)	0.3692 (2)	0.74177 (11)	0.0434 (6)
O8	0.2712 (2)	0.5717 (2)	0.09473 (11)	0.0241 (6)
C9	0.0723 (4)	0.7717 (3)	-0.03502 (17)	0.0269 (9)
F9	0.3243 (3)	0.4099 (2)	0.68321 (11)	0.0577 (8)
O9	0.0663 (2)	0.1576 (2)	0.31237 (12)	0.0244 (6)
H9	-0.0132	0.1803	0.3076	0.037*
F10	0.4166 (2)	0.00770 (19)	0.94653 (11)	0.0433 (7)
C10	0.4828 (4)	0.2792 (3)	0.86606 (17)	0.0233 (8)
O10	0.0096 (2)	-0.0017 (2)	0.27140 (12)	0.0266 (6)
F11	0.4018 (2)	0.17788 (18)	0.98037 (10)	0.0309 (5)
C11	0.3507 (3)	0.2508 (3)	0.84751 (16)	0.0199 (8)
F12	0.2413 (2)	0.0653 (2)	0.99315 (10)	0.0424 (6)
C12	0.2800 (4)	0.1666 (3)	0.88805 (16)	0.0218 (8)
C13	0.1607 (4)	0.1366 (3)	0.87036 (17)	0.0252 (9)
H13	0.1138	0.0803	0.8987	0.030*
F13	0.0824 (2)	-0.0491 (2)	0.41419 (10)	0.0353 (6)
C14	0.1091 (4)	0.1894 (3)	0.81057 (17)	0.0224 (8)
H14	0.0270	0.1691	0.7980	0.027*
F14	0.2542 (2)	-0.1135 (2)	0.46976 (10)	0.0400 (6)
C15	0.1785 (4)	0.2714 (3)	0.76988 (17)	0.0234 (8)
H15	0.1441	0.3064	0.7290	0.028*
F15	0.2106 (2)	0.07786 (19)	0.43794 (10)	0.0370 (6)
C16	0.2977 (4)	0.3034 (3)	0.78793 (16)	0.0217 (8)
F16	0.1837 (2)	-0.03585 (18)	0.15962 (9)	0.0278 (5)
C17	0.3656 (4)	0.3974 (4)	0.74290 (18)	0.0299 (9)
F17	0.1763 (2)	0.14663 (17)	0.17769 (9)	0.0273 (5)
C18	0.3351 (4)	0.1034 (3)	0.95232 (18)	0.0278 (9)
F18	0.3479 (2)	0.07010 (19)	0.13671 (9)	0.0310 (5)
C19	0.0905 (4)	0.0543 (3)	0.29297 (16)	0.0211 (8)
F19	0.6650 (2)	0.7594 (2)	0.45210 (10)	0.0356 (6)
C20	0.2308 (3)	0.0048 (3)	0.30006 (16)	0.0192 (8)
F20	0.8213 (2)	0.6977 (2)	0.39510 (9)	0.0365 (6)

C21	0.3090 (3)	-0.0021 (3)	0.24565 (16)	0.0195 (8)
F21	0.8352 (2)	0.85782 (18)	0.43459 (10)	0.0361 (6)
C22	0.4375 (4)	-0.0487 (3)	0.25249 (18)	0.0223 (8)
H22	0.4903	-0.0511	0.2156	0.027*
F22	0.9052 (2)	0.3168 (2)	0.66535 (12)	0.0454 (7)
C23	0.4900 (4)	-0.0917 (3)	0.31231 (18)	0.0255 (9)
H23	0.5779	-0.1248	0.3164	0.031*
F23	0.7250 (2)	0.4195 (2)	0.67639 (11)	0.0417 (6)
C24	0.4143 (4)	-0.0866 (3)	0.36632 (18)	0.0261 (9)
H24	0.4503	-0.1167	0.4075	0.031*
F24	0.7544 (2)	0.32825 (19)	0.59631 (11)	0.0419 (6)
C25	0.2858 (3)	-0.0376 (3)	0.36039 (16)	0.0202 (8)
F25	0.6092 (2)	0.14615 (19)	0.14326 (10)	0.0337 (5)
C26	0.2081 (4)	-0.0306 (3)	0.42061 (18)	0.0273 (9)
F26	0.7286 (2)	0.28146 (19)	0.16435 (10)	0.0320 (5)
C27	0.2537 (4)	0.0439 (3)	0.18000 (17)	0.0228 (8)
F27	0.6488 (2)	0.26378 (18)	0.41950 (9)	0.0277 (5)
C28	0.6694 (4)	0.5377 (3)	0.52244 (17)	0.0237 (8)
F28	0.4893 (2)	0.1538 (2)	0.44626 (9)	0.0325 (5)
C29	0.8011 (3)	0.5702 (3)	0.54062 (16)	0.0198 (8)
F29	0.6595 (2)	0.07953 (17)	0.40289 (9)	0.0281 (5)
C30	0.8664 (4)	0.5031 (3)	0.59459 (17)	0.0226 (8)
F30	0.5517 (2)	0.3352 (2)	0.11093 (10)	0.0409 (6)
C31	0.9828 (4)	0.5382 (3)	0.61349 (17)	0.0259 (9)
H31	1.0254	0.4930	0.6505	0.031*
C32	1.0379 (4)	0.6378 (3)	0.57941 (18)	0.0255 (9)
H32	1.1180	0.6610	0.5927	0.031*
C33	0.9757 (4)	0.7032 (3)	0.52601 (17)	0.0238 (9)
H33	1.0136	0.7716	0.5023	0.029*
C34	0.8579 (4)	0.6706 (3)	0.50624 (16)	0.0210 (8)
C35	0.7957 (4)	0.7459 (3)	0.44775 (17)	0.0245 (9)
C36	0.8120 (4)	0.3929 (3)	0.63253 (18)	0.0287 (9)
C37	0.7321 (4)	0.1698 (3)	0.28488 (16)	0.0205 (8)
C38	0.5913 (3)	0.2176 (3)	0.28029 (16)	0.0189 (8)
C39	0.5192 (4)	0.2240 (3)	0.33629 (16)	0.0197 (8)
C40	0.3908 (4)	0.2727 (3)	0.33246 (17)	0.0226 (8)
H40	0.3421	0.2763	0.3705	0.027*
C41	0.3330 (4)	0.3163 (3)	0.27363 (17)	0.0238 (8)
H41	0.2454	0.3504	0.2715	0.029*
C42	0.4026 (4)	0.3101 (3)	0.21823 (17)	0.0244 (9)
H42	0.3626	0.3394	0.1779	0.029*
C43	0.5319 (4)	0.2609 (3)	0.22122 (16)	0.0216 (8)
C44	0.6044 (4)	0.2552 (3)	0.16000 (17)	0.0254 (9)
C45	0.5793 (4)	0.1803 (3)	0.40084 (17)	0.0237 (8)
F4	0.1004 (2)	0.38607 (18)	0.16895 (10)	0.0319 (5)
F5	-0.0752 (2)	0.4452 (2)	0.21359 (10)	0.0356 (5)
F6	0.0871 (2)	0.55444 (18)	0.20437 (9)	0.0289 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0198 (13)	0.0469 (14)	0.0305 (12)	-0.0067 (10)	0.0055 (10)	-0.0040 (10)
C1	0.020 (2)	0.0212 (18)	0.0229 (19)	-0.0016 (16)	0.0007 (16)	-0.0061 (15)
O1	0.0138 (14)	0.0371 (15)	0.0322 (14)	-0.0036 (11)	0.0023 (12)	-0.0127 (12)
F2	0.0322 (14)	0.0588 (15)	0.0219 (11)	-0.0052 (12)	-0.0015 (10)	0.0054 (11)
C2	0.0151 (19)	0.0191 (17)	0.0228 (18)	-0.0004 (14)	0.0000 (15)	-0.0084 (15)
O2	0.0157 (14)	0.0436 (16)	0.0324 (15)	-0.0070 (13)	0.0011 (12)	-0.0205 (13)
F3	0.0440 (15)	0.0242 (11)	0.0533 (15)	-0.0063 (10)	0.0131 (12)	-0.0006 (11)
C3	0.014 (2)	0.0228 (18)	0.0271 (19)	-0.0001 (15)	0.0019 (16)	-0.0098 (16)
O3	0.0168 (14)	0.0280 (13)	0.0340 (15)	-0.0026 (11)	-0.0016 (12)	-0.0103 (12)
C4	0.021 (2)	0.0225 (18)	0.0248 (19)	0.0048 (16)	-0.0030 (16)	-0.0092 (16)
O4	0.0139 (14)	0.0247 (13)	0.0415 (15)	0.0025 (11)	-0.0002 (13)	-0.0113 (12)
C5	0.016 (2)	0.0237 (18)	0.031 (2)	-0.0006 (15)	-0.0031 (17)	-0.0123 (17)
O5	0.0164 (14)	0.0317 (14)	0.0505 (17)	0.0031 (12)	-0.0024 (13)	-0.0217 (13)
C6	0.019 (2)	0.0204 (18)	0.031 (2)	-0.0014 (15)	0.0047 (17)	-0.0053 (16)
O6	0.0175 (15)	0.0262 (13)	0.0395 (15)	-0.0010 (11)	-0.0075 (13)	-0.0158 (12)
C7	0.014 (2)	0.0190 (17)	0.0235 (18)	0.0012 (15)	0.0011 (15)	-0.0081 (15)
F7	0.0372 (14)	0.0255 (11)	0.0470 (14)	-0.0088 (10)	0.0025 (12)	-0.0033 (11)
O7	0.0173 (14)	0.0251 (13)	0.0356 (15)	-0.0029 (11)	-0.0036 (12)	-0.0128 (12)
C8	0.0206 (19)	0.0219 (16)	0.0268 (19)	-0.0002 (10)	-0.0001 (13)	-0.0054 (14)
F8	0.0318 (15)	0.0483 (14)	0.0512 (15)	-0.0117 (11)	0.0169 (12)	-0.0099 (12)
O8	0.0149 (14)	0.0250 (13)	0.0333 (14)	0.0007 (11)	-0.0050 (11)	-0.0085 (11)
C9	0.023 (2)	0.031 (2)	0.027 (2)	-0.0001 (17)	0.0004 (17)	-0.0053 (17)
F9	0.080 (2)	0.0733 (18)	0.0228 (13)	-0.0463 (16)	-0.0016 (13)	0.0012 (12)
O9	0.0163 (14)	0.0250 (13)	0.0331 (14)	0.0021 (11)	0.0002 (12)	-0.0104 (11)
F10	0.0571 (17)	0.0273 (12)	0.0453 (14)	0.0187 (12)	-0.0244 (13)	-0.0143 (11)
C10	0.018 (2)	0.0258 (19)	0.030 (2)	-0.0022 (16)	-0.0014 (17)	-0.0157 (17)
O10	0.0163 (14)	0.0281 (13)	0.0383 (15)	-0.0014 (11)	-0.0046 (12)	-0.0135 (12)
F11	0.0327 (14)	0.0293 (11)	0.0320 (12)	0.0006 (10)	-0.0128 (10)	-0.0097 (10)
C11	0.017 (2)	0.0196 (17)	0.0255 (19)	-0.0017 (15)	0.0004 (16)	-0.0102 (15)
F12	0.0482 (16)	0.0463 (14)	0.0290 (12)	-0.0138 (12)	-0.0048 (12)	0.0080 (11)
C12	0.023 (2)	0.0177 (17)	0.0257 (19)	0.0045 (15)	-0.0020 (17)	-0.0081 (15)
C13	0.025 (2)	0.0226 (19)	0.029 (2)	-0.0043 (16)	0.0023 (18)	-0.0062 (16)
F13	0.0236 (13)	0.0486 (14)	0.0326 (12)	-0.0120 (11)	0.0047 (10)	-0.0004 (11)
C14	0.017 (2)	0.0238 (18)	0.029 (2)	-0.0055 (15)	-0.0042 (16)	-0.0088 (16)
F14	0.0439 (15)	0.0475 (14)	0.0246 (12)	-0.0004 (12)	-0.0034 (11)	0.0023 (11)
C15	0.025 (2)	0.0231 (18)	0.0233 (19)	-0.0012 (16)	-0.0055 (17)	-0.0084 (16)
F15	0.0412 (15)	0.0362 (13)	0.0377 (13)	-0.0073 (11)	0.0100 (11)	-0.0167 (11)
C16	0.019 (2)	0.0232 (18)	0.0255 (19)	-0.0037 (15)	0.0017 (16)	-0.0099 (16)
F16	0.0269 (13)	0.0299 (11)	0.0304 (11)	-0.0039 (10)	-0.0054 (10)	-0.0142 (10)
C17	0.029 (2)	0.033 (2)	0.030 (2)	-0.0056 (18)	-0.0006 (18)	-0.0095 (18)
F17	0.0308 (13)	0.0216 (11)	0.0284 (11)	0.0073 (9)	-0.0059 (10)	-0.0055 (9)
C18	0.028 (2)	0.0235 (19)	0.033 (2)	0.0009 (17)	-0.0071 (19)	-0.0080 (17)
F18	0.0270 (13)	0.0392 (12)	0.0258 (11)	-0.0035 (10)	0.0032 (10)	-0.0035 (10)
C19	0.019 (2)	0.0226 (18)	0.0232 (19)	-0.0017 (16)	0.0003 (16)	-0.0074 (16)
F19	0.0186 (12)	0.0465 (14)	0.0371 (13)	0.0052 (10)	-0.0028 (10)	0.0008 (11)

C20	0.019 (2)	0.0149 (16)	0.0247 (19)	-0.0022 (14)	-0.0017 (16)	-0.0056 (15)
F20	0.0402 (15)	0.0470 (14)	0.0243 (12)	-0.0007 (11)	0.0013 (10)	-0.0129 (11)
C21	0.018 (2)	0.0140 (16)	0.0257 (19)	0.0011 (15)	-0.0031 (16)	-0.0038 (15)
F21	0.0414 (15)	0.0264 (11)	0.0381 (13)	-0.0028 (10)	-0.0032 (11)	0.0008 (10)
C22	0.019 (2)	0.0167 (17)	0.034 (2)	-0.0030 (15)	0.0003 (17)	-0.0099 (16)
F22	0.0348 (15)	0.0353 (13)	0.0569 (16)	0.0032 (11)	-0.0033 (13)	0.0139 (12)
C23	0.016 (2)	0.0206 (18)	0.039 (2)	0.0039 (15)	-0.0028 (18)	-0.0049 (17)
F23	0.0372 (15)	0.0417 (13)	0.0444 (14)	-0.0084 (11)	0.0167 (12)	-0.0025 (11)
C24	0.028 (2)	0.0179 (18)	0.032 (2)	-0.0003 (16)	-0.0050 (18)	-0.0028 (16)
F24	0.0456 (16)	0.0279 (12)	0.0536 (15)	-0.0161 (11)	-0.0043 (13)	-0.0044 (11)
C25	0.020 (2)	0.0166 (17)	0.0254 (19)	-0.0055 (15)	0.0000 (16)	-0.0054 (15)
F25	0.0370 (14)	0.0351 (12)	0.0335 (12)	-0.0105 (10)	0.0078 (11)	-0.0152 (10)
C26	0.026 (2)	0.029 (2)	0.027 (2)	-0.0062 (17)	-0.0065 (18)	0.0004 (17)
F26	0.0302 (14)	0.0381 (12)	0.0314 (12)	-0.0174 (10)	0.0097 (10)	-0.0101 (10)
C27	0.021 (2)	0.0201 (18)	0.029 (2)	-0.0013 (16)	0.0031 (17)	-0.0084 (16)
F27	0.0247 (12)	0.0306 (11)	0.0311 (11)	-0.0046 (10)	-0.0071 (10)	-0.0123 (10)
C28	0.019 (2)	0.0261 (19)	0.027 (2)	-0.0002 (16)	0.0002 (17)	-0.0076 (17)
F28	0.0272 (13)	0.0429 (13)	0.0253 (11)	-0.0023 (10)	0.0032 (10)	-0.0008 (10)
C29	0.0131 (19)	0.0220 (18)	0.0270 (19)	0.0010 (15)	0.0011 (16)	-0.0126 (16)
F29	0.0290 (13)	0.0250 (11)	0.0289 (11)	0.0071 (10)	-0.0052 (10)	-0.0048 (9)
C30	0.019 (2)	0.0233 (18)	0.027 (2)	-0.0003 (16)	0.0009 (17)	-0.0093 (16)
F30	0.0473 (16)	0.0472 (14)	0.0243 (12)	0.0005 (12)	0.0010 (11)	0.0015 (11)
C31	0.023 (2)	0.0263 (19)	0.029 (2)	0.0050 (17)	-0.0053 (17)	-0.0077 (17)
C32	0.018 (2)	0.0262 (19)	0.035 (2)	-0.0029 (16)	-0.0033 (17)	-0.0102 (17)
C33	0.019 (2)	0.0214 (18)	0.033 (2)	-0.0039 (16)	0.0060 (17)	-0.0087 (17)
C34	0.021 (2)	0.0201 (18)	0.0233 (19)	-0.0019 (15)	0.0034 (16)	-0.0076 (15)
C35	0.021 (2)	0.029 (2)	0.025 (2)	-0.0047 (16)	0.0023 (17)	-0.0075 (17)
C36	0.023 (2)	0.028 (2)	0.033 (2)	-0.0025 (18)	-0.0043 (19)	-0.0012 (18)
C37	0.019 (2)	0.0220 (18)	0.0214 (18)	-0.0027 (16)	0.0034 (16)	-0.0051 (16)
C38	0.0130 (19)	0.0169 (17)	0.0275 (19)	-0.0016 (14)	-0.0010 (16)	-0.0059 (15)
C39	0.022 (2)	0.0152 (17)	0.0226 (18)	-0.0023 (15)	-0.0023 (16)	-0.0045 (15)
C40	0.019 (2)	0.0215 (18)	0.029 (2)	-0.0015 (15)	0.0029 (17)	-0.0084 (16)
C41	0.017 (2)	0.0233 (19)	0.031 (2)	-0.0004 (16)	-0.0049 (17)	-0.0068 (16)
C42	0.026 (2)	0.0194 (18)	0.027 (2)	-0.0025 (16)	-0.0070 (17)	-0.0023 (16)
C43	0.022 (2)	0.0209 (18)	0.0232 (19)	-0.0049 (16)	0.0019 (16)	-0.0050 (15)
C44	0.026 (2)	0.025 (2)	0.026 (2)	-0.0067 (17)	0.0010 (17)	-0.0023 (17)
C45	0.018 (2)	0.0278 (19)	0.027 (2)	0.0003 (16)	0.0031 (17)	-0.0093 (17)
F4	0.0323 (13)	0.0240 (10)	0.0372 (12)	0.0096 (9)	-0.0095 (10)	-0.0040 (9)
F5	0.0306 (12)	0.0427 (13)	0.0286 (12)	-0.0050 (9)	0.0018 (9)	0.0067 (10)
F6	0.0317 (13)	0.0310 (11)	0.0250 (11)	-0.0016 (9)	-0.0067 (9)	-0.0076 (9)

Geometric parameters (Å, °)

F1—C9	1.347 (4)	C16—C17	1.503 (5)
C1—O8	1.223 (4)	F16—C27	1.332 (4)
C1—O7	1.305 (4)	F17—C27	1.340 (4)
C1—C2	1.509 (5)	F18—C27	1.341 (4)
O1—C28	1.215 (4)	C19—C20	1.505 (5)

F2—C9	1.339 (4)	F19—C35	1.347 (4)
C2—C7	1.398 (5)	C20—C25	1.399 (5)
C2—C3	1.407 (5)	C20—C21	1.409 (5)
O2—C28	1.310 (4)	F20—C35	1.343 (4)
O2—H2	0.8400	C21—C22	1.384 (5)
F3—C9	1.341 (4)	C21—C27	1.504 (5)
C3—C4	1.389 (5)	F21—C35	1.336 (4)
C3—C9	1.498 (5)	C22—C23	1.380 (5)
O3—C37	1.231 (4)	C22—H22	0.9500
C4—C5	1.386 (5)	F22—C36	1.346 (4)
C4—H4	0.9500	C23—C24	1.384 (5)
O4—C37	1.308 (4)	C23—H23	0.9500
O4—H4A	0.8400	F23—C36	1.338 (4)
C5—C6	1.377 (5)	C24—C25	1.390 (5)
C5—H5	0.9500	C24—H24	0.9500
O5—C10	1.229 (4)	F24—C36	1.330 (5)
C6—C7	1.387 (5)	C25—C26	1.510 (5)
C6—H6	0.9500	F25—C44	1.335 (4)
O6—C10	1.303 (4)	F26—C44	1.347 (4)
O6—H6A	0.8400	F27—C45	1.345 (4)
C7—C8	1.490 (5)	C28—C29	1.511 (5)
F7—C17	1.332 (4)	F28—C45	1.340 (4)
O7—H7	0.8400	C29—C34	1.396 (5)
C8—F5	1.338 (4)	C29—C30	1.404 (5)
C8—F6	1.345 (4)	F29—C45	1.340 (4)
C8—F4	1.350 (4)	C30—C31	1.383 (5)
F8—C17	1.340 (5)	C30—C36	1.497 (5)
F9—C17	1.327 (4)	F30—C44	1.339 (4)
O9—C19	1.305 (4)	C31—C32	1.379 (5)
O9—H9	0.8400	C31—H31	0.9500
F10—C18	1.330 (4)	C32—C33	1.373 (5)
C10—C11	1.500 (5)	C32—H32	0.9500
O10—C19	1.229 (4)	C33—C34	1.392 (5)
F11—C18	1.351 (4)	C33—H33	0.9500
C11—C16	1.398 (5)	C34—C35	1.494 (5)
C11—C12	1.404 (5)	C37—C38	1.503 (5)
F12—C18	1.334 (4)	C38—C43	1.396 (5)
C12—C13	1.379 (5)	C38—C39	1.403 (5)
C12—C18	1.519 (5)	C39—C40	1.387 (5)
C13—C14	1.397 (5)	C39—C45	1.499 (5)
C13—H13	0.9500	C40—C41	1.385 (5)
F13—C26	1.344 (4)	C40—H40	0.9500
C14—C15	1.383 (5)	C41—C42	1.379 (5)
C14—H14	0.9500	C41—H41	0.9500
F14—C26	1.334 (4)	C42—C43	1.397 (5)
C15—C16	1.390 (5)	C42—H42	0.9500
C15—H15	0.9500	C43—C44	1.499 (5)
F15—C26	1.338 (4)		

O8—C1—O7	126.0 (3)	C24—C23—H23	120.0
O8—C1—C2	121.5 (3)	C23—C24—C25	120.1 (4)
O7—C1—C2	112.5 (3)	C23—C24—H24	120.0
C7—C2—C3	118.8 (3)	C25—C24—H24	120.0
C7—C2—C1	120.9 (3)	C24—C25—C20	120.6 (3)
C3—C2—C1	120.3 (3)	C24—C25—C26	118.3 (3)
C28—O2—H2	109.5	C20—C25—C26	121.1 (3)
C4—C3—C2	120.2 (3)	F14—C26—F15	106.9 (3)
C4—C3—C9	118.3 (3)	F14—C26—F13	106.6 (3)
C2—C3—C9	121.5 (3)	F15—C26—F13	107.0 (3)
C5—C4—C3	120.4 (3)	F14—C26—C25	111.8 (3)
C5—C4—H4	119.8	F15—C26—C25	111.8 (3)
C3—C4—H4	119.8	F13—C26—C25	112.4 (3)
C37—O4—H4A	109.5	F16—C27—F17	107.1 (3)
C6—C5—C4	119.5 (4)	F16—C27—F18	107.1 (3)
C6—C5—H5	120.2	F17—C27—F18	106.1 (3)
C4—C5—H5	120.2	F16—C27—C21	112.7 (3)
C5—C6—C7	121.2 (3)	F17—C27—C21	111.9 (3)
C5—C6—H6	119.4	F18—C27—C21	111.5 (3)
C7—C6—H6	119.4	O1—C28—O2	125.4 (4)
C10—O6—H6A	109.5	O1—C28—C29	121.2 (3)
C6—C7—C2	119.9 (3)	O2—C28—C29	113.4 (3)
C6—C7—C8	119.6 (3)	C34—C29—C30	118.4 (3)
C2—C7—C8	120.4 (3)	C34—C29—C28	121.1 (3)
C1—O7—H7	109.5	C30—C29—C28	120.4 (3)
F5—C8—F6	106.1 (3)	C31—C30—C29	120.1 (3)
F5—C8—F4	105.6 (3)	C31—C30—C36	118.9 (3)
F6—C8—F4	106.2 (3)	C29—C30—C36	120.9 (3)
F5—C8—C7	112.7 (3)	C32—C31—C30	121.0 (4)
F6—C8—C7	113.6 (3)	C32—C31—H31	119.5
F4—C8—C7	112.0 (3)	C30—C31—H31	119.5
F2—C9—F3	106.6 (3)	C33—C32—C31	119.3 (4)
F2—C9—F1	106.6 (3)	C33—C32—H32	120.3
F3—C9—F1	106.5 (3)	C31—C32—H32	120.3
F2—C9—C3	112.1 (3)	C32—C33—C34	120.8 (3)
F3—C9—C3	111.8 (3)	C32—C33—H33	119.6
F1—C9—C3	112.7 (3)	C34—C33—H33	119.6
C19—O9—H9	109.5	C33—C34—C29	120.2 (3)
O5—C10—O6	125.4 (3)	C33—C34—C35	118.0 (3)
O5—C10—C11	120.7 (3)	C29—C34—C35	121.8 (3)
O6—C10—C11	113.9 (3)	F21—C35—F20	105.8 (3)
C16—C11—C12	118.3 (3)	F21—C35—F19	106.2 (3)
C16—C11—C10	120.6 (3)	F20—C35—F19	105.7 (3)
C12—C11—C10	121.0 (3)	F21—C35—C34	113.0 (3)
C13—C12—C11	121.4 (3)	F20—C35—C34	112.7 (3)
C13—C12—C18	118.1 (3)	F19—C35—C34	112.9 (3)
C11—C12—C18	120.5 (4)	F24—C36—F23	107.2 (3)

C12—C13—C14	119.8 (3)	F24—C36—F22	106.1 (3)
C12—C13—H13	120.1	F23—C36—F22	105.4 (3)
C14—C13—H13	120.1	F24—C36—C30	112.9 (3)
C15—C14—C13	119.4 (4)	F23—C36—C30	112.9 (3)
C15—C14—H14	120.3	F22—C36—C30	111.8 (3)
C13—C14—H14	120.3	O3—C37—O4	124.8 (3)
C14—C15—C16	121.0 (3)	O3—C37—C38	121.1 (3)
C14—C15—H15	119.5	O4—C37—C38	114.0 (3)
C16—C15—H15	119.5	C43—C38—C39	119.0 (3)
C15—C16—C11	120.1 (3)	C43—C38—C37	121.3 (3)
C15—C16—C17	118.6 (3)	C39—C38—C37	119.7 (3)
C11—C16—C17	121.3 (3)	C40—C39—C38	120.0 (3)
F9—C17—F7	107.2 (3)	C40—C39—C45	119.0 (3)
F9—C17—F8	106.7 (3)	C38—C39—C45	121.0 (3)
F7—C17—F8	106.5 (3)	C41—C40—C39	120.5 (3)
F9—C17—C16	112.5 (3)	C41—C40—H40	119.7
F7—C17—C16	111.9 (3)	C39—C40—H40	119.7
F8—C17—C16	111.7 (3)	C42—C41—C40	120.0 (3)
F10—C18—F12	107.9 (3)	C42—C41—H41	120.0
F10—C18—F11	107.1 (3)	C40—C41—H41	120.0
F12—C18—F11	106.6 (3)	C41—C42—C43	120.2 (3)
F10—C18—C12	111.5 (3)	C41—C42—H42	119.9
F12—C18—C12	111.7 (3)	C43—C42—H42	119.9
F11—C18—C12	111.9 (3)	C38—C43—C42	120.2 (3)
O10—C19—O9	125.4 (3)	C38—C43—C44	121.0 (3)
O10—C19—C20	120.9 (3)	C42—C43—C44	118.7 (3)
O9—C19—C20	113.7 (3)	F25—C44—F30	106.9 (3)
C25—C20—C21	118.5 (3)	F25—C44—F26	106.5 (3)
C25—C20—C19	121.1 (3)	F30—C44—F26	106.4 (3)
C21—C20—C19	120.4 (3)	F25—C44—C43	113.2 (3)
C22—C21—C20	120.1 (3)	F30—C44—C43	112.1 (3)
C22—C21—C27	119.8 (3)	F26—C44—C43	111.2 (3)
C20—C21—C27	120.0 (3)	F29—C45—F28	106.4 (3)
C23—C22—C21	120.7 (3)	F29—C45—F27	106.9 (3)
C23—C22—H22	119.6	F28—C45—F27	106.2 (3)
C21—C22—H22	119.6	F29—C45—C39	112.6 (3)
C22—C23—C24	120.0 (3)	F28—C45—C39	111.9 (3)
C22—C23—H23	120.0	F27—C45—C39	112.4 (3)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O2—H2...O1 ⁱ	0.84	1.87	2.701 (4)	172
O4—H4A...O10 ⁱⁱ	0.84	1.82	2.657 (3)	174
O6—H6A...O8 ⁱ	0.84	1.84	2.673 (4)	170

O7—H7...O5 ⁱ	0.84	1.80	2.638 (4)	173
O9—H9...O3 ⁱⁱⁱ	0.84	1.81	2.644 (3)	174

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $x+1, y, z$; (iii) $x-1, y, z$.