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2,2,3,3-Tetrafluorobutane-1,4-diol

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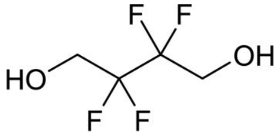
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.002 Å; R factor = 0.031; wR factor = 0.085; data-to-parameter ratio = 13.5.

In the title compound, $C_4H_6F_4O_2$, a partially fluorinated aliphatic diol, cooperative O—H···O hydrogen bonds form $R_2^2(14)$ rings, which are connected into infinite layers parallel to the (100) plane by $C(7)$ chains. A C—H···F link is also seen.

Related literature

For crystal structures containing 2,2,3,3-tetrafluorobutane-1,4-dioxy units, see: Elias *et al.* (1994); Beşli *et al.* (2004, 2005, 2006). For details on graph-set analysis of hydrogen-bond networks, see: Bernstein *et al.* (1995); Etter *et al.* (1990).



Experimental

Crystal data

$C_4H_6F_4O_2$
M_r = 162.09
Monoclinic, $P2_1/c$
a = 5.4392 (2) Å
b = 8.6935 (3) Å
c = 12.4123 (4) Å
β = 99.768 (2)°

V = 578.42 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 200 (2) K
0.18 × 0.08 × 0.06 mm

Data collection

Nonius KappaCCD area-detector diffractometer

Absorption correction: none
2531 measured reflections
1313 independent reflections
1133 reflections with $I > 2\sigma(I)$

$R_{\rm int}$ = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.085
S = 1.04
1313 reflections
97 parameters

H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\rm max}$ = 0.38 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.25 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.80 (2)	2.001 (19)	2.7972 (14)	171.9 (18)
O2—H2···O1^{ii}	0.845 (17)	1.940 (17)	2.7608 (14)	163.6 (17)
C1—H1*B*···F3^{i}	0.99	2.44	3.2343 (15)	137

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

Data collection: *COLLECT* (Hooft, 2004); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); 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); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2164).

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supporting information

Acta Cryst. (2009). E**65**, o33 [doi:10.1107/S1600536808040555]

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S1. Comment

The asymmetric unit of the title compound contains one complete molecule, which is shown in Figure 1.

The molecular packing is dominated by two O—H···O hydrogen bonds. According to graph set theory [Bernstein *et al.* (1995), Etter *et al.* (1990)] the descriptors $C(7)$ and $R_2^2(14)$ can be assigned. Together with one C—H···F hydrogen bond [motif $C(5)$] the first-level (unitary) graph set $N_1 = C(5)C(7)R_2^2(14)$ is obtained.

Figure 2 shows a cutout of the layers parallel to the (100) plane which are generated by the O—H···O hydrogen bond framework. The C—H···F bonds which are located within these layers are omitted for clarity.

Due to packing effects and the specific hydrogen bonding interactions the O1—C1—C2—C3—C4—O2 chain adopts a somewhat unusual conformation. The substituents at the C2—C3 fragment are staggered with the CH_2OH moieties being gauche to each other. For Newman projections see Fig. 3.

S2. Experimental

The title compound was obtained from Acros Organics. A single-crystal suitable for X-ray diffraction was isolated from the supplied material.

S3. Refinement

All H atoms were found in difference maps. C-bonded H atoms were positioned geometrically (C—H = 0.99 Å) and treated as riding on their parent atoms [$U_{iso}(H) = 1.2U_{eq}(C)$]. Coordinates of O-bonded H atoms and O—H distances were refined freely [$U_{iso}(H) = 1.5U_{eq}(O)$].

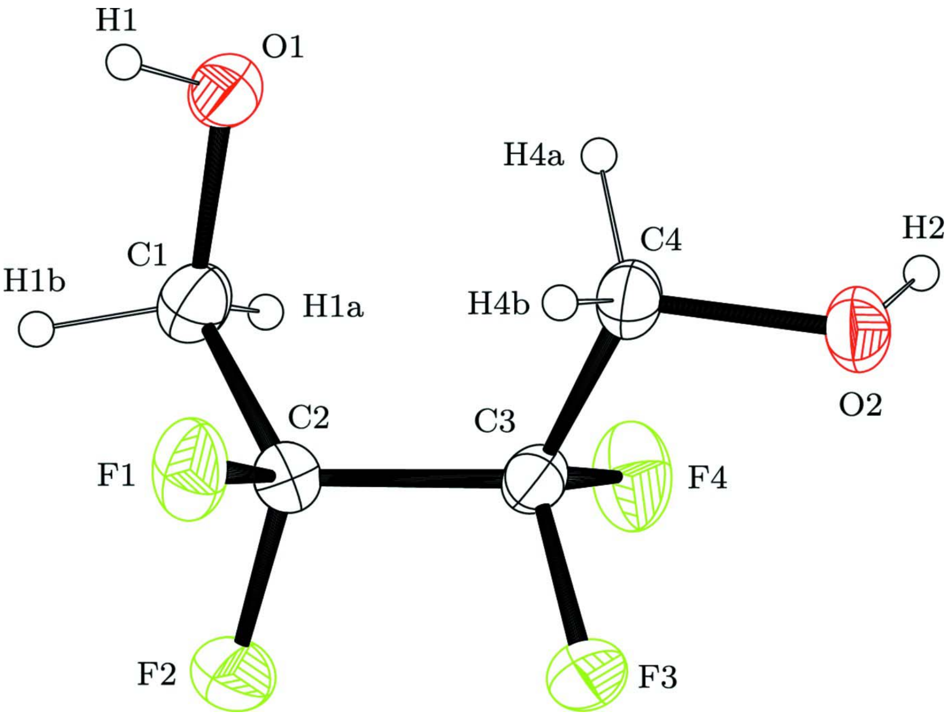

Figure 1

The molecular structure of the title compound, with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level) for non-H atoms.

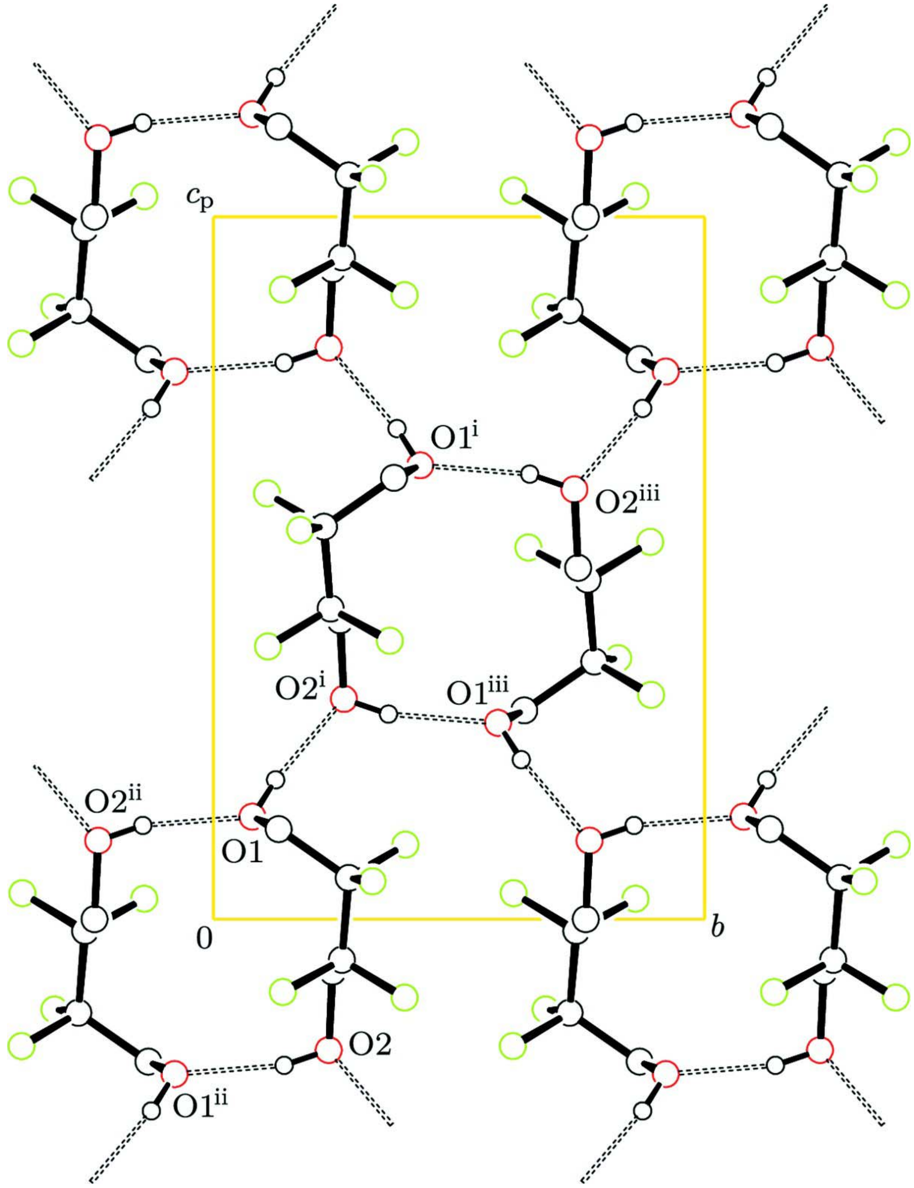

Figure 2

Hydrogen bonding in (I). [Symmetry codes: (i) x, 1/2-y, 1/2+z; (ii) 1-x, -y, -z; (iii) 1-x, 1/2+y, 1/2-z.]

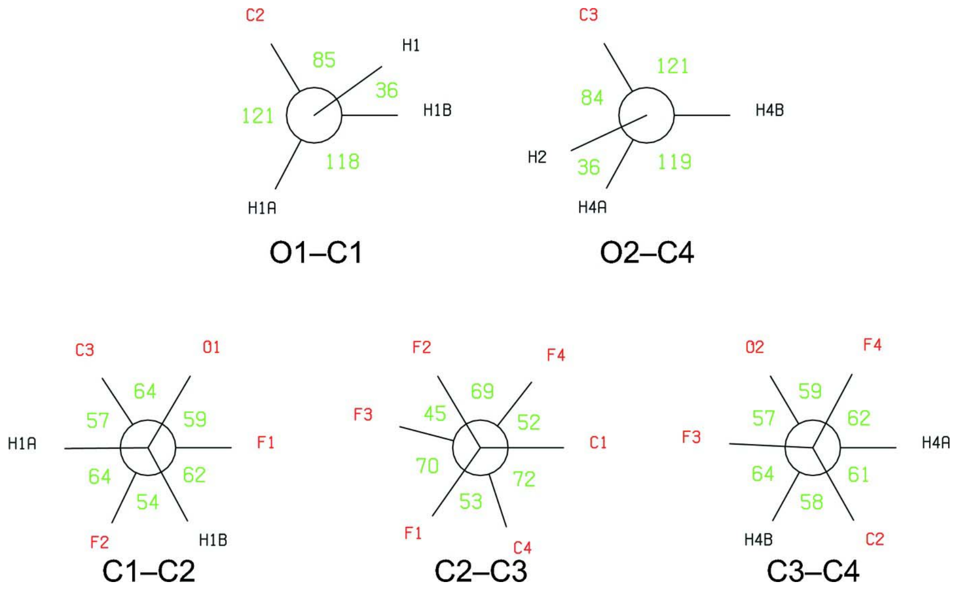

Figure 3

Newman projections along the bonds of the O1—C1—C2—C3—C4—O2 chain.

2,2,3,3-tetrafluorobutane-1,4-diol

Crystal data

$C_4H_6F_4O_2$
$M_r = 162.09$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 5.4392$ (2) Å
$b = 8.6935$ (3) Å
$c = 12.4123$ (4) Å
$\beta = 99.768$ (2)°
$V = 578.42$ (3) Å^3
$Z = 4$

$F(000) = 328$
$D_x = 1.861$ (1) Mg m^{-3}
Melting point = 355.3–356.3 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7007 reflections
$\theta = 3.1–27.5°$
$\mu = 0.22$ mm^{-1}
$T = 200$ K
Block, colourless
0.18 × 0.08 × 0.06 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Radiation source: rotating anode
MONTEL, graded multilayered X-ray optics monochromator
Detector resolution: 9 pixels mm^{-1}
φ and ω scans
2531 measured reflections

1313 independent reflections
1133 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.3°$
$h = -7 \rightarrow 7$
$k = -11 \rightarrow 11$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.085$
$S = 1.04$
1313 reflections
97 parameters
0 restraints

Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0398P)^2 + 0.2156P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.38$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-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 $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on all data will be even larger.

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

	x	y	z	U_{iso}*/U_{eq}
F1	0.70269 (17)	0.39078 (9)	0.15846 (6)	0.0318 (2)
F2	1.07701 (15)	0.32375 (11)	0.13418 (7)	0.0343 (2)
F3	0.81808 (17)	0.38903 (11)	−0.05076 (6)	0.0349 (2)
F4	0.86586 (15)	0.14104 (11)	−0.03922 (6)	0.0328 (2)
O1	0.58505 (19)	0.07917 (11)	0.18952 (8)	0.0256 (2)
H1	0.528 (4)	0.125 (2)	0.2357 (16)	0.038*
O2	0.41094 (18)	0.23464 (12)	−0.15572 (7)	0.0253 (2)
H2	0.431 (3)	0.143 (2)	−0.1751 (15)	0.038*
C1	0.8308 (3)	0.13254 (16)	0.19011 (10)	0.0246 (3)
H1A	0.9293	0.0505	0.1621	0.030*
H1B	0.9099	0.1556	0.2662	0.030*
C2	0.8355 (2)	0.27521 (15)	0.12090 (10)	0.0216 (3)
C3	0.7415 (2)	0.26145 (14)	−0.00258 (10)	0.0202 (3)
C4	0.4637 (2)	0.24221 (16)	−0.04003 (10)	0.0227 (3)
H4A	0.4061	0.1468	−0.0086	0.027*
H4B	0.3737	0.3302	−0.0142	0.027*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0491 (5)	0.0227 (4)	0.0240 (4)	0.0031 (4)	0.0075 (4)	−0.0057 (3)
F2	0.0285 (4)	0.0452 (5)	0.0273 (4)	−0.0150 (4)	−0.0012 (3)	0.0044 (4)
F3	0.0445 (5)	0.0363 (5)	0.0227 (4)	−0.0187 (4)	0.0026 (3)	0.0077 (3)
F4	0.0305 (5)	0.0407 (5)	0.0275 (4)	0.0102 (4)	0.0059 (3)	−0.0101 (4)
O1	0.0327 (5)	0.0239 (5)	0.0217 (5)	−0.0053 (4)	0.0091 (4)	−0.0022 (4)
O2	0.0325 (5)	0.0252 (5)	0.0169 (4)	−0.0018 (4)	0.0000 (4)	−0.0003 (4)
C1	0.0278 (7)	0.0261 (7)	0.0203 (6)	0.0001 (5)	0.0051 (5)	0.0028 (5)
C2	0.0226 (6)	0.0231 (6)	0.0189 (6)	−0.0036 (5)	0.0035 (5)	−0.0021 (5)
C3	0.0252 (6)	0.0193 (6)	0.0171 (6)	−0.0024 (5)	0.0068 (5)	−0.0004 (5)
C4	0.0238 (6)	0.0274 (7)	0.0165 (6)	−0.0005 (5)	0.0024 (5)	0.0001 (5)

Geometric parameters (Å, º)

F1—C2	1.3651 (15)	C1—C2	1.5114 (18)
F2—C2	1.3626 (15)	C1—H1A	0.9900
F3—C3	1.3587 (14)	C1—H1B	0.9900
F4—C3	1.3656 (14)	C2—C3	1.5360 (17)
O1—C1	1.4135 (17)	C3—C4	1.5128 (17)
O1—H1	0.80 (2)	C4—H4A	0.9900
O2—C4	1.4173 (15)	C4—H4B	0.9900
O2—H2	0.84 (2)		
C1—O1—H1	108.0 (14)	C1—C2—C3	117.92 (11)
C4—O2—H2	108.6 (13)	F3—C3—F4	105.84 (9)
O1—C1—C2	111.96 (11)	F3—C3—C4	108.66 (10)
O1—C1—H1A	109.2	F4—C3—C4	109.78 (10)
C2—C1—H1A	109.2	F3—C3—C2	107.49 (10)
O1—C1—H1B	109.2	F4—C3—C2	106.97 (10)
C2—C1—H1B	109.2	C4—C3—C2	117.48 (10)
H1A—C1—H1B	107.9	O2—C4—C3	109.65 (10)
F2—C2—F1	106.61 (10)	O2—C4—H4A	109.7
F2—C2—C1	107.13 (10)	C3—C4—H4A	109.7
F1—C2—C1	110.41 (10)	O2—C4—H4B	109.7
F2—C2—C3	107.21 (9)	C3—C4—H4B	109.7
F1—C2—C3	106.98 (10)	H4A—C4—H4B	108.2
O1—C1—C2—F2	175.17 (10)	C1—C2—C3—F4	−52.09 (14)
O1—C1—C2—F1	59.45 (14)	F2—C2—C3—C4	−167.32 (11)
O1—C1—C2—C3	−63.91 (14)	F1—C2—C3—C4	−53.27 (14)
F2—C2—C3—F3	−44.50 (13)	C1—C2—C3—C4	71.80 (15)
F1—C2—C3—F3	69.55 (12)	F3—C3—C4—O2	56.60 (13)
C1—C2—C3—F3	−165.38 (11)	F4—C3—C4—O2	−58.71 (13)
F2—C2—C3—F4	68.79 (12)	C2—C3—C4—O2	178.82 (11)
F1—C2—C3—F4	−177.16 (9)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.80 (2)	2.001 (19)	2.7972 (14)	171.9 (18)
O2—H2···O1^{ii}	0.845 (17)	1.940 (17)	2.7608 (14)	163.6 (17)
C1—H1*B*···F3^{i}	0.99	2.44	3.2343 (15)	137

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