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1-(3,5-Difluorophenyl)-4,4,4-trifluorobutane-1,3-dione

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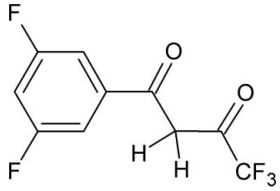
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.006 Å; R factor = 0.074; wR factor = 0.239; data-to-parameter ratio = 10.4.

In the title compound, $C_{10}H_5F_5O_2$, the C=O bonds are *syn* to one another. In the crystal, molecules are linked into $C(9)$ chains parallel to [101] through weak C—H···O interactions, with the O atom adjacent to the $-CF_3$ group acting as the acceptor.

Related literature

For biological-activity studies of compounds with trifluoromethyl substituents, see: Manoj Kumar *et al.* (2013).



Experimental

Crystal data

$C_{10}H_5F_5O_2$
M_r = 252.14
Monoclinic, $C2/c$
a = 12.393 (4) Å
b = 13.433 (5) Å
c = 12.877 (5) Å
β = 112.49 (2)°
V = 1980.7 (13) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.18 mm^{-1}
T = 294 K
0.24 × 0.20 × 0.16 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.959, T_{max} = 0.972
5651 measured reflections
1604 independent reflections
1246 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.239
S = 1.08
1604 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O2^{i}	0.93	2.53	3.462 (5)	177

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT-Plus* (Bruker, 2009); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97*.

The authors acknowledge the IOE X-ray diffractometer Facility, University of Mysore, Mysore, for the data collection. KEM and SS acknowledge Tumkur University, Tumkur, for providing laboratory facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB7151).

References

Bruker (2009). *APEX2*, *SAINT-Plus* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2013). E**69**, o1705 [doi:10.1107/S1600536813028535]

1-(3,5-Difluorophenyl)-4,4,4-trifluorobutane-1,3-dione

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S1. Comment

As part of our ongoing studies of the biological activities of compounds with a trifluoromethyl substituent (Manoj Kumar *et al.*, 2013), we now describe the structure of the title compound, (I).

The two C=O bonds are *syn* to one another (Fig.1), the O1—C7—C8—C9 and O2—C9—C8—C7 torsion angles being 0.4 (5) and -0.3 (6)°, respectively. In the crystal, the molecules are linked into C(9) chains parallel to [101] through a weak C3—H3···O2 interaction (Fig.2).

S2. Experimental

3,5-Difluoroacetophenone (1 mmol) and sodium hydride (1.5 mmol) were taken in dry THF (20 ml), and the solution was stirred for 30 min at 0°C. To this solution trifluoroethylacetate (1.2 mmol) was added and the reaction mixture was stirred for 10 h at room temperature under nitrogen atmosphere. The reaction was monitored by TLC. The crude mass was purified by column chromatography using petroleum ether and ethyl acetate as an eluent (7:3), to obtain a yellow solid. Pale yellow prisms were obtained by recrystallisation fron dichloromethane/methanol (9:1) solution.

S3. Refinement

The H atoms were positioned with idealized geometry using a riding model with C—H = 0.93–0.97 Å. All H atoms were refined with isotropic displacement parameters (set to 1.2 times of the U_{eq} of the parent atom).

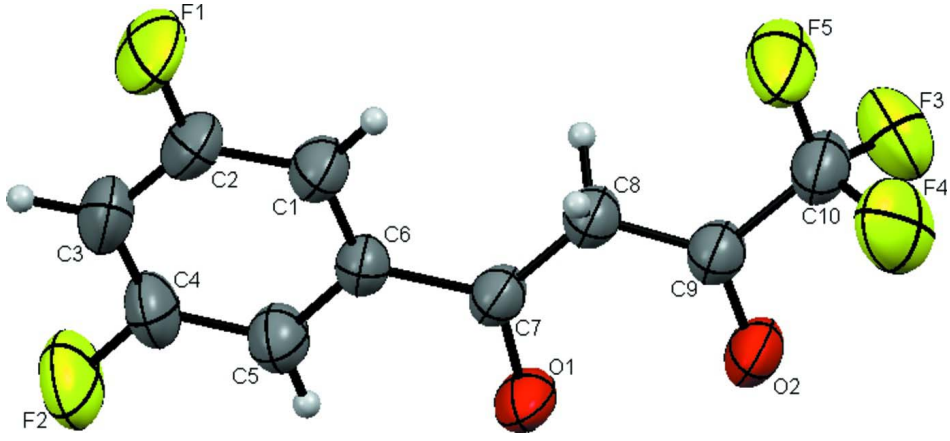

Figure 1

Molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level.

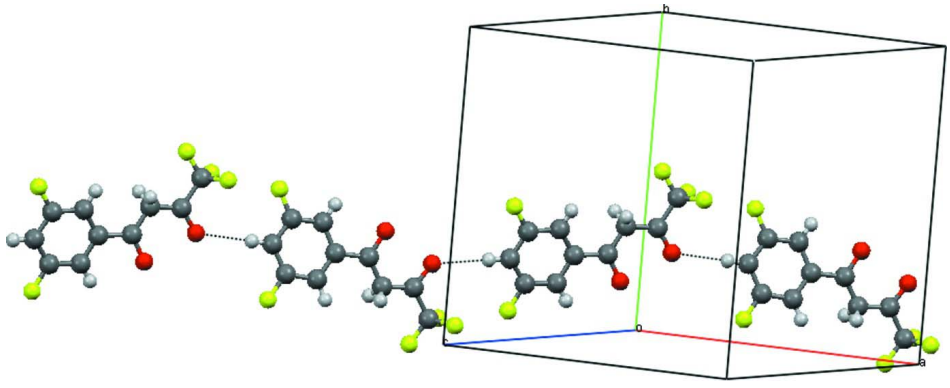

Figure 2

Molecular packing foming C(9) chains parallel to [101] with hydrogen bonding shown as dashed lines.

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Crystal data

$C_{10}H_5F_5O_2$
$M_r = 252.14$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 12.393$ (4) Å
$b = 13.433$ (5) Å
$c = 12.877$ (5) Å
$\beta = 112.49$ (2)°
$V = 1980.7$ (13) Å^3
$Z = 8$
$F(000) = 1008$

Prism
$D_x = 1.691$ Mg m^{-3}
Melting point: 393 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1023 reflections
$\theta = 0.0$–$24.6°$
$\mu = 0.18$ mm^{-1}
$T = 294$ K
Prism, colourless
0.24 × 0.20 × 0.16 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min} = 0.959$, $T_{max} = 0.972$

5651 measured reflections
1604 independent reflections
1246 reflections with $I > 2\sigma(I)$
$R_{int} = 0.040$
$\theta_{max} = 24.6°$, $\theta_{min} = 2.3°$
$h = -14 \rightarrow 14$
$k = -15 \rightarrow 7$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.074$
$wR(F^2) = 0.239$
$S = 1.08$
1604 reflections
154 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.1433P)^2 + 2.2069P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.012$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.47$ 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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.0373 (3)	0.3441 (3)	0.4970 (3)	0.0558 (9)
H1	0.0691	0.4058	0.4923	0.067*
C2	−0.0308 (3)	0.3323 (3)	0.5594 (3)	0.0624 (10)
C3	−0.0787 (4)	0.2426 (3)	0.5705 (3)	0.0684 (11)
H3	−0.1238	0.2360	0.6135	0.082*
C4	−0.0565 (4)	0.1636 (3)	0.5147 (4)	0.0649 (10)
C5	0.0106 (3)	0.1700 (3)	0.4506 (3)	0.0574 (9)
H5	0.0242	0.1143	0.4144	0.069*
C6	0.0574 (3)	0.2624 (2)	0.4416 (3)	0.0474 (8)
C7	0.1298 (3)	0.2699 (2)	0.3734 (3)	0.0476 (8)
C8	0.1640 (3)	0.3603 (3)	0.3419 (3)	0.0511 (8)
H8A	0.0937	0.3983	0.3019	0.061*
H8B	0.2077	0.3967	0.4101	0.061*
C9	0.2304 (3)	0.3596 (3)	0.2760 (3)	0.0515 (9)
C10	0.2656 (3)	0.4562 (3)	0.2375 (3)	0.0624 (10)
F1	−0.0500 (3)	0.4121 (2)	0.6129 (3)	0.1000 (11)
F2	−0.1027 (3)	0.0740 (2)	0.5224 (3)	0.1010 (11)
F3	0.2245 (3)	0.4627 (2)	0.1272 (2)	0.0913 (9)
F4	0.3807 (2)	0.4629 (2)	0.2695 (3)	0.0989 (10)
F5	0.2306 (3)	0.5370 (2)	0.2748 (3)	0.1110 (13)
O1	0.1598 (2)	0.18650 (19)	0.3425 (2)	0.0671 (8)
O2	0.2645 (2)	0.2827 (2)	0.2411 (2)	0.0666 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.065 (2)	0.051 (2)	0.066 (2)	0.0018 (15)	0.0410 (17)	−0.0022 (16)
C2	0.072 (2)	0.063 (2)	0.070 (2)	0.0110 (18)	0.0473 (19)	−0.0002 (17)
C3	0.070 (2)	0.083 (3)	0.073 (2)	0.007 (2)	0.050 (2)	0.012 (2)
C4	0.072 (2)	0.059 (2)	0.081 (2)	−0.0095 (18)	0.048 (2)	0.0108 (19)
C5	0.064 (2)	0.050 (2)	0.069 (2)	−0.0058 (15)	0.0380 (17)	−0.0011 (15)
C6	0.0508 (18)	0.0460 (18)	0.0561 (18)	0.0013 (13)	0.0323 (15)	0.0034 (14)
C7	0.0473 (16)	0.0457 (18)	0.0588 (19)	0.0002 (13)	0.0306 (15)	−0.0036 (14)
C8	0.0564 (18)	0.0448 (19)	0.065 (2)	0.0001 (14)	0.0373 (16)	−0.0009 (15)
C9	0.0535 (18)	0.048 (2)	0.063 (2)	−0.0011 (14)	0.0335 (16)	−0.0016 (15)
C10	0.069 (2)	0.058 (2)	0.079 (3)	−0.0047 (17)	0.049 (2)	−0.0035 (18)

F1	0.146 (3)	0.0803 (18)	0.121 (2)	0.0129 (16)	0.105 (2)	−0.0090 (15)
F2	0.125 (2)	0.0773 (18)	0.138 (3)	−0.0305 (16)	0.092 (2)	0.0011 (16)
F3	0.112 (2)	0.0859 (19)	0.0818 (18)	−0.0116 (15)	0.0440 (15)	0.0191 (13)
F4	0.0676 (15)	0.096 (2)	0.139 (3)	−0.0226 (13)	0.0460 (15)	0.0177 (17)
F5	0.169 (3)	0.0515 (16)	0.173 (3)	−0.0089 (16)	0.133 (3)	−0.0034 (16)
O1	0.0897 (19)	0.0399 (14)	0.102 (2)	0.0042 (12)	0.0704 (16)	−0.0031 (12)
O2	0.0793 (17)	0.0555 (16)	0.0924 (19)	0.0030 (12)	0.0633 (15)	−0.0029 (13)

Geometric parameters (Å, º)

C1—C2	1.379 (5)	C6—C7	1.479 (5)
C1—C6	1.383 (5)	C7—O1	1.290 (4)
C1—H1	0.9300	C7—C8	1.396 (5)
C2—F1	1.344 (5)	C8—C9	1.389 (5)
C2—C3	1.374 (6)	C8—H8A	0.9700
C3—C4	1.367 (6)	C8—H8B	0.9700
C3—H3	0.9300	C9—O2	1.262 (4)
C4—F2	1.353 (5)	C9—C10	1.512 (5)
C4—C5	1.381 (5)	C10—F3	1.316 (5)
C5—C6	1.393 (5)	C10—F5	1.327 (5)
C5—H5	0.9300	C10—F4	1.328 (4)
C2—C1—C6	118.8 (3)	O1—C7—C8	120.7 (3)
C2—C1—H1	120.6	O1—C7—C6	115.8 (3)
C6—C1—H1	120.6	C8—C7—C6	123.5 (3)
F1—C2—C3	118.5 (3)	C9—C8—C7	119.2 (3)
F1—C2—C1	118.4 (4)	C9—C8—H8A	107.5
C3—C2—C1	123.1 (4)	C7—C8—H8A	107.5
C4—C3—C2	116.4 (3)	C9—C8—H8B	107.5
C4—C3—H3	121.8	C7—C8—H8B	107.5
C2—C3—H3	121.8	H8A—C8—H8B	107.0
F2—C4—C3	118.5 (3)	O2—C9—C8	125.5 (3)
F2—C4—C5	117.9 (4)	O2—C9—C10	114.0 (3)
C3—C4—C5	123.7 (4)	C8—C9—C10	120.5 (3)
C4—C5—C6	118.0 (4)	F3—C10—F5	106.9 (4)
C4—C5—H5	121.0	F3—C10—F4	104.9 (3)
C6—C5—H5	121.0	F5—C10—F4	107.1 (3)
C1—C6—C5	120.0 (3)	F3—C10—C9	111.8 (3)
C1—C6—C7	121.5 (3)	F5—C10—C9	114.1 (3)
C5—C6—C7	118.4 (3)	F4—C10—C9	111.5 (3)
C6—C1—C2—F1	−179.9 (3)	C5—C6—C7—O1	−11.0 (5)
C6—C1—C2—C3	0.8 (6)	C1—C6—C7—C8	−12.7 (5)
F1—C2—C3—C4	−179.9 (4)	C5—C6—C7—C8	168.3 (3)
C1—C2—C3—C4	−0.6 (6)	O1—C7—C8—C9	0.4 (5)
C2—C3—C4—F2	−179.5 (4)	C6—C7—C8—C9	−178.9 (3)
C2—C3—C4—C5	0.4 (6)	C7—C8—C9—O2	−0.3 (6)
F2—C4—C5—C6	179.4 (3)	C7—C8—C9—C10	178.1 (3)

C3—C4—C5—C6	−0.5 (6)	O2—C9—C10—F3	59.0 (4)
C2—C1—C6—C5	−0.9 (5)	C8—C9—C10—F3	−119.5 (4)
C2—C1—C6—C7	−179.9 (3)	O2—C9—C10—F5	−179.5 (3)
C4—C5—C6—C1	0.7 (5)	C8—C9—C10—F5	1.9 (5)
C4—C5—C6—C7	179.8 (3)	O2—C9—C10—F4	−58.1 (4)
C1—C6—C7—O1	168.0 (3)	C8—C9—C10—F4	123.4 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O2^{i}	0.93	2.53	3.462 (5)	177

Symmetry code: (i) $x-1/2, -y+1/2, z+1/2$.