Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3,3,4,4-Tetrafluoro-2,3,4,5-tetrahydro-1,6-benzodioxocine-8-carbaldehyde

Zhuo Zeng,* Jun-Wen Zhong, Hui Wang, Jin Wang and Wan-Wan Cao

School of Chemistry and Environment, South China Normal University, Guangzhou 510006, People's Republic of China
Correspondence e-mail: zhuosioc@yahoo.com.cn

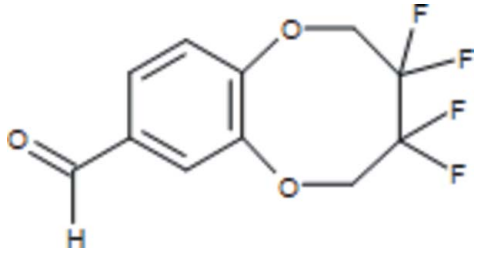
Received 13 March 2010; accepted 16 April 2010

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.047; wR factor = 0.146; data-to-parameter ratio = 12.2.

In the title compound, $C_{11}H_8F_4O_3$, the eight-membered dialkoxy ring adopts a highly puckered conformation. In the crystal, molecules are linked by weak C—H···O interactions.

Related literature

For the applications of fluorinated mcarocyles, see: Babudri *et al.* (2007).



Experimental

Crystal data

$C_{11}H_8F_4O_3$
M_r = 264.17
Monoclinic, $P2_1/n$
a = 9.142 (5) Å
b = 11.4935 (14) Å
c = 10.928 (10) Å
β = 104.109 (15)°
V = 1113.6 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.16 mm^{-1}
T = 298 K
0.35 × 0.24 × 0.11 mm

Data collection

Bruker SMART CCD diffractometer
5552 measured reflections
2000 independent reflections
972 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.146
S = 0.97
2000 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O3^{i}	0.93	2.52	3.193 (5)	130
C8—H8B···O3^{ii}	0.97	2.43	3.343 (6)	157

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

We are grateful to the Doctoral Program Foundation of the Natural Science Foundation of Guangdong Province, China (No. 5100430) and South China Normal University Grants (524002, 523467) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5362).

References

Babudri, F., Farinola, G. M., Naso, F. & Ragni, R. (2007). *Chem. Commun.* pp. 1003–1022
Bruker (2000). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Acta Cryst. (2010). E**66**, o1137 [**doi:10.1107/S1600536810014133**]

3,3,4,4-Tetrafluoro-2,3,4,5-tetrahydro-1,6-benzodioxocine-8-carbaldehyde

Z. Zeng, J.-W. Zhong, H. Wang, J. Wang and W.-W. Cao

Comment

Fluorinated mcarocyles play an important role in the pharmaceutical, agrochemical and advanced materials fields (Babudri *et al.*, 2007). As part of our studies in this area, we now report the synthesis and structure of the title compound, (I).

The structure of this compound is shown in Fig. 1. The benzene ring is attached to a highly puckered eight-membered dialkoxy ring. The torsion angle at the fusion bond (O1—C8—C9—O2) is 3.27°. The C—C bond distances of the aromatic ring vary from 1.373 (3) to 1.407 (3) A, the latter being the C2—C7 fusion bond. The ring angles of the benzene ring also vary from 118.9 (8) to 120.8 (9)° indicating a slight distortion in this ring.

Experimental

A mixture of 3,4-dihydroxy-benzaldehyde (0.345 g, 2.5 mmol), potassium carbonate (3.453 g, 25 mmol) was refluxed in acetonitrile (20 ml) at 373 K for 45 min, then a solution of methanesulfonic acid, trifluoro-, 2,2,3,3-tetrafluoro-1,4-butanediylester in acetonitrile (5 ml) was added, the mixture was heated under reflux for 12 h. After cooling to room temperature, the inorganic salts was removed by filtration . The filtrate was concentrated under reduced pressure, The residue was purified by flash chromatography on silica gel to afford the title compound as a white solid, yield 467 mg (70.8%). Colourless blocks of (I) were grown by by slow evaporation from dichloromethane at room temperature.

Refinement

H-atoms were placed in calculated positions with C—H = 0.93–0.97 Å and refined as riding atoms.

Figures

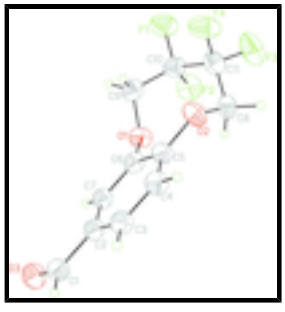


Fig. 1. View of (I) showing displacement ellipsoids drawn at the 50% probability level.

3,3,4,4-Tetrafluoro-2,3,4,5-tetrahydro-1,6-benzodioxocine-8-carbaldehyde

Crystal data

$C_{11}H_8F_4O_3$	$F(000) = 536.0$
$M_r = 264.17$	$D_x = 1.576$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 733 reflections
$a = 9.142$ (5) Å	$\theta = 3.2–20.1°$
$b = 11.4935$ (14) Å	$\mu = 0.16$ mm^{-1}
$c = 10.928$ (10) Å	$T = 298$ K
$\beta = 104.109$ (15)°	Block, colourless
$V = 1113.6$ (12) Å^3	0.35 × 0.24 × 0.11 mm
$Z = 4$	

Data collection

Bruker SMART CCD diffractometer	972 reflections with $I > 2\sigma(I)$
Radiation source: sealed tube	$R_{int} = 0.044$
graphite	$\theta_{max} = 25.2°$, $\theta_{min} = 2.6°$
ω scans	$h = -6\rightarrow10$
5552 measured reflections	$k = -13\rightarrow13$
2000 independent reflections	$l = -13\rightarrow11$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.047$	H-atom parameters constrained
$wR(F^2) = 0.146$	$w = 1/[\sigma^2(F_o^2) + (0.049P)^2 + 0.4913P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.97$	$(\Delta/\sigma)_{max} < 0.001$
2000 reflections	$\Delta\rho_{max} = 0.14$ e Å^{-3}
164 parameters	$\Delta\rho_{min} = -0.15$ e Å^{-3}
0 restraints	Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0047 (12)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds

in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
F2	0.5814 (2)	0.90137 (19)	0.9443 (2)	0.0901 (8)
F4	0.9709 (3)	0.8853 (2)	1.0613 (2)	0.1040 (9)
F1	0.7668 (3)	0.9585 (2)	0.8674 (2)	0.1008 (9)
F3	0.7960 (3)	0.9756 (2)	1.1293 (3)	0.1194 (10)
C4	0.8950 (4)	0.4731 (3)	1.1272 (3)	0.0599 (10)
H4	0.9789	0.4745	1.1953	0.072*
C7	0.6454 (3)	0.4699 (3)	0.9227 (3)	0.0545 (9)
H7	0.5614	0.4689	0.8547	0.065*
C6	0.7048 (3)	0.5746 (3)	0.9715 (3)	0.0535 (9)
C5	0.8301 (3)	0.5770 (3)	1.0754 (3)	0.0552 (9)
C2	0.7096 (3)	0.3650 (3)	0.9738 (3)	0.0549 (9)
C3	0.8354 (3)	0.3678 (3)	1.0779 (3)	0.0590 (10)
H3	0.8785	0.2986	1.1136	0.071*
C10	0.7221 (4)	0.8729 (4)	0.9373 (4)	0.0685 (11)
C8	0.8155 (4)	0.7738 (3)	1.1564 (3)	0.0669 (11)
H8A	0.8542	0.7957	1.2441	0.080*
H8B	0.7108	0.7509	1.1445	0.080*
C1	0.6488 (4)	0.2520 (4)	0.9223 (4)	0.0664 (11)
H1	0.6927	0.1853	0.9636	0.080*
C11	0.8265 (4)	0.8762 (4)	1.0718 (4)	0.0741 (11)
C9	0.7179 (4)	0.7596 (3)	0.8678 (3)	0.0657 (10)
H9A	0.6716	0.7710	0.7788	0.079*
H9B	0.8197	0.7310	0.8761	0.079*
O2	0.9011 (2)	0.6785 (2)	1.1261 (2)	0.0670 (7)
O1	0.6327 (2)	0.6767 (2)	0.9195 (2)	0.0622 (7)
O3	0.5453 (3)	0.2397 (2)	0.8295 (3)	0.0825 (9)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F2	0.0728 (15)	0.0924 (18)	0.0987 (18)	0.0255 (13)	0.0085 (12)	0.0057 (14)
F4	0.0709 (15)	0.112 (2)	0.116 (2)	−0.0292 (14)	−0.0040 (13)	0.0202 (16)
F1	0.1117 (19)	0.0856 (18)	0.0951 (19)	−0.0162 (14)	0.0061 (14)	0.0252 (14)
F3	0.157 (2)	0.0757 (18)	0.103 (2)	0.0154 (16)	−0.0126 (17)	−0.0179 (16)
C4	0.049 (2)	0.073 (3)	0.046 (2)	−0.0005 (19)	−0.0088 (15)	0.0038 (19)
C7	0.0456 (19)	0.070 (3)	0.0410 (19)	−0.0046 (18)	−0.0020 (15)	0.0014 (18)
C6	0.0420 (19)	0.067 (3)	0.046 (2)	0.0014 (17)	0.0012 (16)	0.0051 (18)

C5	0.0449 (19)	0.065 (3)	0.049 (2)	−0.0042 (17)	−0.0011 (16)	−0.0010 (18)
C2	0.049 (2)	0.065 (2)	0.047 (2)	−0.0067 (17)	0.0062 (16)	−0.0003 (18)
C3	0.056 (2)	0.065 (3)	0.049 (2)	0.0014 (18)	0.0010 (17)	0.0049 (18)
C10	0.065 (2)	0.070 (3)	0.068 (3)	−0.003 (2)	0.010 (2)	0.013 (2)
C8	0.063 (2)	0.079 (3)	0.052 (2)	−0.001 (2)	0.0015 (17)	−0.009 (2)
C1	0.064 (2)	0.071 (3)	0.063 (2)	−0.010 (2)	0.012 (2)	−0.003 (2)
C11	0.070 (3)	0.071 (3)	0.074 (3)	0.000 (2)	0.004 (2)	−0.009 (2)
C9	0.058 (2)	0.084 (3)	0.049 (2)	0.000 (2)	0.0007 (16)	0.011 (2)
O2	0.0481 (13)	0.0678 (17)	0.0733 (18)	−0.0011 (12)	−0.0077 (12)	−0.0091 (13)
O1	0.0472 (13)	0.0663 (17)	0.0647 (16)	0.0009 (12)	−0.0025 (11)	0.0115 (13)
O3	0.0749 (17)	0.096 (2)	0.0663 (18)	−0.0205 (16)	−0.0032 (14)	−0.0142 (15)

Geometric parameters (Å, °)

F2—C10	1.347 (4)	C2—C1	1.469 (5)
F4—C11	1.357 (4)	C3—H3	0.9300
F1—C10	1.368 (4)	C10—C9	1.504 (5)
F3—C11	1.365 (4)	C10—C11	1.545 (6)
C4—C3	1.381 (4)	C8—O2	1.432 (4)
C4—C5	1.392 (4)	C8—C11	1.515 (5)
C4—H4	0.9300	C8—H8A	0.9700
C7—C6	1.374 (4)	C8—H8B	0.9700
C7—C2	1.397 (4)	C1—O3	1.215 (4)
C7—H7	0.9300	C1—H1	0.9300
C6—O1	1.398 (4)	C9—O1	1.430 (4)
C6—C5	1.402 (4)	C9—H9A	0.9700
C5—O2	1.383 (4)	C9—H9B	0.9700
C2—C3	1.407 (4)		
C3—C4—C5	120.3 (3)	O2—C8—C11	109.4 (3)
C3—C4—H4	119.8	O2—C8—H8A	109.8
C5—C4—H4	119.8	C11—C8—H8A	109.8
C6—C7—C2	120.9 (3)	O2—C8—H8B	109.8
C6—C7—H7	119.6	C11—C8—H8B	109.8
C2—C7—H7	119.6	H8A—C8—H8B	108.3
C7—C6—O1	118.4 (3)	O3—C1—C2	124.6 (4)
C7—C6—C5	119.9 (3)	O3—C1—H1	117.7
O1—C6—C5	121.6 (3)	C2—C1—H1	117.7
O2—C5—C4	116.7 (3)	F4—C11—F3	106.6 (3)
O2—C5—C6	123.5 (3)	F4—C11—C8	108.8 (3)
C4—C5—C6	119.7 (3)	F3—C11—C8	108.5 (4)
C7—C2—C3	119.0 (3)	F4—C11—C10	108.1 (4)
C7—C2—C1	121.8 (3)	F3—C11—C10	108.1 (3)
C3—C2—C1	119.2 (3)	C8—C11—C10	116.4 (3)
C4—C3—C2	120.1 (3)	O1—C9—C10	109.0 (3)
C4—C3—H3	119.9	O1—C9—H9A	109.9
C2—C3—H3	119.9	C10—C9—H9A	109.9
F2—C10—F1	106.1 (3)	O1—C9—H9B	109.9
F2—C10—C9	109.4 (3)	C10—C9—H9B	109.9
F1—C10—C9	108.4 (3)	H9A—C9—H9B	108.3

F2—C10—C11	108.5 (4)	C5—O2—C8	120.5 (2)
F1—C10—C11	108.3 (3)	C6—O1—C9	118.1 (2)
C9—C10—C11	115.8 (3)		
C2—C7—C6—O1	−177.7 (3)	F2—C10—C11—F4	−160.5 (3)
C2—C7—C6—C5	−1.1 (5)	F1—C10—C11—F4	−45.8 (4)
C3—C4—C5—O2	−176.8 (3)	C9—C10—C11—F4	76.1 (4)
C3—C4—C5—C6	−0.9 (5)	F2—C10—C11—F3	−45.4 (5)
C7—C6—C5—O2	176.6 (3)	F1—C10—C11—F3	69.2 (4)
O1—C6—C5—O2	−6.8 (5)	C9—C10—C11—F3	−168.8 (3)
C7—C6—C5—C4	1.0 (5)	F2—C10—C11—C8	76.9 (4)
O1—C6—C5—C4	177.6 (3)	F1—C10—C11—C8	−168.5 (3)
C6—C7—C2—C3	0.9 (5)	C9—C10—C11—C8	−46.5 (5)
C6—C7—C2—C1	−179.4 (3)	F2—C10—C9—O1	−48.7 (4)
C5—C4—C3—C2	0.8 (5)	F1—C10—C9—O1	−163.9 (2)
C7—C2—C3—C4	−0.8 (5)	C11—C10—C9—O1	74.2 (4)
C1—C2—C3—C4	179.5 (3)	C4—C5—O2—C8	−134.3 (3)
C7—C2—C1—O3	3.8 (6)	C6—C5—O2—C8	49.9 (5)
C3—C2—C1—O3	−176.6 (4)	C11—C8—O2—C5	−114.5 (3)
O2—C8—C11—F4	−41.8 (4)	C7—C6—O1—C9	−121.7 (3)
O2—C8—C11—F3	−157.5 (3)	C5—C6—O1—C9	61.7 (4)
O2—C8—C11—C10	80.4 (4)	C10—C9—O1—C6	−120.0 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O3^{i}	0.93	2.52	3.193 (5)	130
C8—H8B···O3^{ii}	0.97	2.43	3.343 (6)	157

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

Fig. 1