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2,2-Bis(pentafluorophenyloxy)-1,3-benzodioxole

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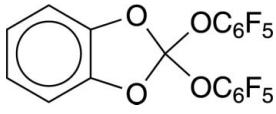
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.003 Å; R factor = 0.051; wR factor = 0.133; data-to-parameter ratio = 13.6.

The title compound, $C_{19}H_4F_{10}O_4$, an orthocarbonate of a vicincal aromatic diol and two monofunctional aromatic alcohols, was prepared for the purpose of collecting NMR data on orthocarbonates and for comparison with similar silicon compounds. The molecule does not exhibit any crystallographic symmetry. The fused ring system is almost perfectly planar. The angle between the least-squares planes through the pentafluorophenyl groups is 81.26 (6)°.

Related literature

For related literature, see: Gross *et al.* (1964); Komatsu *et al.* (1992); Mues & Buysch (1990).



Experimental

Crystal data

$C_{19}H_4F_{10}O_4$
M_r = 486.22
Monoclinic, $P2_1/c$
a = 15.8249 (15) Å
b = 6.3560 (7) Å
c = 18.0213 (19) Å
β = 103.552 (9)°
V = 1762.2 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.20 mm^{-1}
T = 200 (3) K
0.30 × 0.25 × 0.10 mm

Data collection

Oxford XCalibur3 diffractometer
Absorption correction: none
9833 measured reflections
4059 independent reflections
2950 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.133
S = 1.12
4059 reflections
299 parameters
Only H-atom displacement parameters refined
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2005); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2005); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2082).

References

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supplementary materials

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2,2-Bis(pentafluorophenyloxy)-1,3-benzodioxole

R. Betz, P. Klüfers and M. M. Reichvilser

Comment

The title compound, $C_{19}H_4F_{10}O_4$, an orthocarbonate of a vicincal aromatic diol and two monofunctional aromatic alcohols, was prepared for the purpose of collecting NMR data on orthocarbonates and for comparison with similar silicon compounds. The molecular structure is shown in Fig. 1. The molecule does not exhibit any crystallographic symmetry.

The molecular packing is shown in Fig. 2.

Experimental

2,2-Dichlorobenzo[1,3]dioxole was prepared according to Komatsu *et al.* (1992).

To a colorless solution of pentafluorophenol (1.84 g, 10.0 mmol) and pyridine (0.91 ml, 10.0 mmol) in dichloromethane, 2,2-dichlorobenzo[1,3]dioxole (0.96 g, 5.00 mmol) was added dropwise within 20 min at room temperature. The yellow reaction mixture was stirred over night and subsequently washed twice with water (5 ml). The organic phase was dried over Na_2SO_4. Removal of the solvent under reduced pressure afforded colorless crystals suitable for X-ray diffraction.

Refinement

All H atoms were located in a difference map but placed at idealized positions (C—H: 0.95 Å) and refined as riding on their parent atoms. One common isotropic displacement parameter for all H atoms was refined to U_{iso}(H) = 0.045 (3) Å^2.

Figures

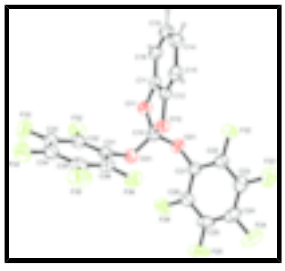


Fig. 1. The molecular structure of (I), with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level) for non-H atoms.

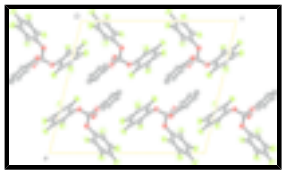


Fig. 2. The packing of (I), viewed along [0 1 0]. H atoms omitted for clarity.

2,2-Bis(pentafluorophenyloxy)-1,3-benzodioxole

Crystal data

$C_{19}H_4F_{10}O_4$	$Z = 4$
$M_r = 486.22$	$F_{000} = 960$
Monoclinic, $P2_1/c$	$D_x = 1.833$ Mg m^{-3}
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 15.8249$ (15) Å	$\theta = 3.9$–27.5°
$b = 6.3560$ (7) Å	$\mu = 0.20$ mm^{-1}
$c = 18.0213$ (19) Å	$T = 200$ (3) K
$\beta = 103.552$ (9)°	Platelet, colourless
$V = 1762.2$ (3) Å^3	0.30 × 0.25 × 0.10 mm

Data collection

Oxford XCalibur3 diffractometer	2950 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.039$
Monochromator: graphite	$\theta_{max} = 27.5^\circ$
$T = 200$(3) K	$\theta_{min} = 4.0^\circ$
ω scans	$h = -20\rightarrow19$
Absorption correction: none	$k = -6\rightarrow8$
9833 measured reflections	$l = -19\rightarrow23$
4059 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.051$	Only H-atom displacement parameters refined
$wR(F^2) = 0.133$	$w = 1/[\sigma^2(F_o^2) + (0.0621P)^2 + 0.0854P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.12$	$(\Delta/\sigma)_{max} < 0.001$
4059 reflections	$\Delta\rho_{max} = 0.25$ e Å^{-3}
299 parameters	$\Delta\rho_{min} = -0.25$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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}
F22	0.41079 (9)	0.4138 (2)	0.40677 (7)	0.0534 (4)
F23	0.36927 (10)	0.6908 (2)	0.50644 (8)	0.0583 (4)
F24	0.24267 (10)	0.5935 (2)	0.57956 (7)	0.0621 (4)
F25	0.16335 (9)	0.2136 (3)	0.55760 (8)	0.0645 (4)
F26	0.20458 (9)	−0.0631 (2)	0.45812 (8)	0.0537 (4)
F32	0.17016 (9)	−0.3524 (2)	0.18969 (8)	0.0537 (4)
F33	0.03388 (11)	−0.3142 (2)	0.06818 (8)	0.0669 (4)
F34	−0.06615 (9)	0.0375 (3)	0.05095 (8)	0.0692 (5)
F35	−0.02976 (9)	0.3499 (3)	0.15615 (9)	0.0669 (4)
F36	0.10328 (9)	0.3085 (2)	0.27928 (8)	0.0536 (4)
O11	0.33597 (9)	−0.0712 (2)	0.26222 (8)	0.0363 (3)
O12	0.27790 (8)	0.2504 (2)	0.27465 (7)	0.0345 (3)
O21	0.33313 (9)	0.0340 (2)	0.37940 (7)	0.0382 (4)
O31	0.20663 (9)	−0.0460 (2)	0.30080 (8)	0.0396 (4)
C10	0.28688 (13)	0.0447 (3)	0.30304 (11)	0.0344 (4)
C11	0.37378 (12)	0.0758 (3)	0.22285 (10)	0.0309 (4)
C12	0.33733 (12)	0.2693 (3)	0.22866 (10)	0.0302 (4)
C13	0.35747 (13)	0.4440 (3)	0.19270 (11)	0.0350 (5)
H13	0.3308	0.5763	0.1964	0.045 (3)*
C14	0.41999 (14)	0.4165 (3)	0.14986 (11)	0.0376 (5)
H14	0.4363	0.5332	0.1234	0.045 (3)*
C15	0.45836 (13)	0.2231 (3)	0.14539 (11)	0.0389 (5)
H15	0.5012	0.2105	0.1164	0.045 (3)*
C16	0.43601 (13)	0.0461 (3)	0.18213 (11)	0.0356 (5)
H16	0.4623	−0.0871	0.1792	0.045 (3)*
C21	0.30653 (13)	0.1741 (3)	0.42810 (11)	0.0340 (4)
C22	0.34749 (14)	0.3668 (4)	0.44189 (11)	0.0389 (5)
C23	0.32660 (15)	0.5076 (3)	0.49263 (12)	0.0415 (5)
C24	0.26363 (14)	0.4560 (4)	0.53031 (11)	0.0430 (5)
C25	0.22338 (13)	0.2641 (4)	0.51871 (11)	0.0429 (5)
C26	0.24407 (13)	0.1229 (3)	0.46800 (11)	0.0375 (5)
C31	0.14188 (13)	−0.0184 (3)	0.23495 (11)	0.0361 (5)
C32	0.12212 (14)	−0.1776 (3)	0.18163 (12)	0.0385 (5)
C33	0.05235 (15)	−0.1581 (4)	0.11929 (12)	0.0446 (5)
C34	0.00212 (14)	0.0198 (4)	0.11078 (12)	0.0469 (6)
C35	0.02027 (13)	0.1788 (4)	0.16381 (13)	0.0440 (5)
C36	0.08919 (14)	0.1574 (3)	0.22622 (12)	0.0387 (5)

Atomic displacement parameters (Å^2)

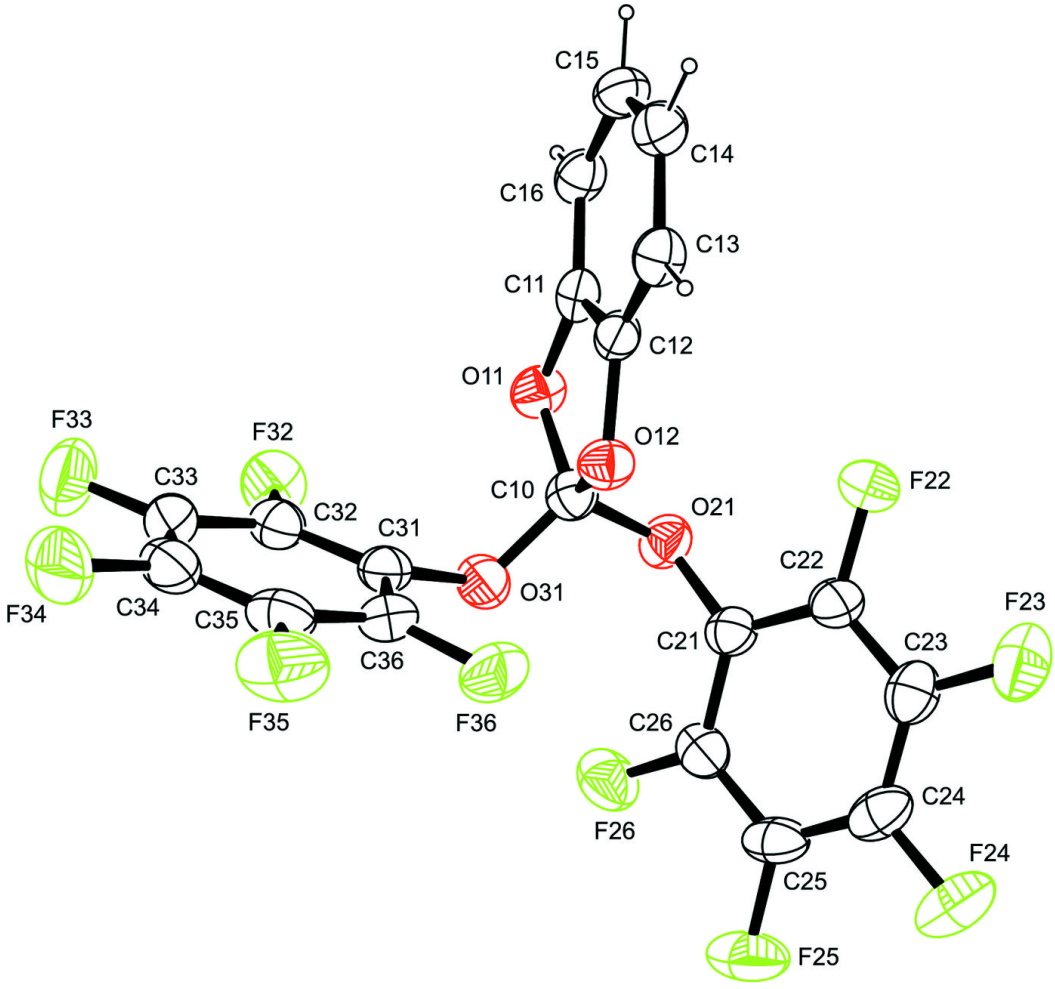
	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F22	0.0534 (8)	0.0631 (9)	0.0501 (8)	−0.0205 (7)	0.0252 (6)	−0.0057 (6)
F23	0.0757 (10)	0.0471 (8)	0.0478 (8)	−0.0076 (7)	0.0054 (7)	−0.0079 (6)
F24	0.0733 (10)	0.0760 (10)	0.0378 (7)	0.0270 (8)	0.0146 (7)	−0.0054 (7)
F25	0.0525 (8)	0.0986 (12)	0.0504 (8)	0.0076 (8)	0.0282 (7)	0.0167 (8)
F26	0.0499 (8)	0.0589 (9)	0.0522 (8)	−0.0133 (7)	0.0119 (6)	0.0103 (6)
F32	0.0575 (8)	0.0385 (7)	0.0650 (9)	0.0036 (7)	0.0142 (7)	−0.0011 (6)
F33	0.0786 (10)	0.0677 (10)	0.0499 (9)	−0.0208 (8)	0.0057 (7)	−0.0175 (7)
F34	0.0436 (8)	0.1070 (13)	0.0490 (8)	−0.0041 (9)	−0.0053 (6)	0.0108 (8)
F35	0.0519 (8)	0.0794 (10)	0.0721 (10)	0.0301 (8)	0.0201 (7)	0.0133 (8)
F36	0.0542 (8)	0.0583 (8)	0.0525 (8)	0.0079 (7)	0.0213 (6)	−0.0149 (6)
O11	0.0443 (8)	0.0282 (7)	0.0383 (8)	0.0023 (6)	0.0132 (6)	−0.0004 (6)
O12	0.0360 (7)	0.0324 (7)	0.0371 (8)	0.0036 (6)	0.0124 (6)	−0.0003 (6)
O21	0.0382 (8)	0.0435 (8)	0.0315 (8)	0.0035 (7)	0.0052 (6)	−0.0016 (6)
O31	0.0367 (7)	0.0467 (9)	0.0344 (8)	−0.0048 (7)	0.0061 (6)	0.0047 (6)
C10	0.0340 (10)	0.0361 (11)	0.0330 (10)	0.0019 (9)	0.0077 (8)	0.0003 (8)
C11	0.0316 (10)	0.0291 (10)	0.0295 (10)	−0.0001 (8)	0.0022 (7)	−0.0043 (7)
C12	0.0286 (9)	0.0336 (10)	0.0275 (10)	0.0009 (8)	0.0046 (7)	−0.0054 (7)
C13	0.0365 (10)	0.0310 (10)	0.0357 (10)	0.0031 (9)	0.0049 (8)	−0.0023 (8)
C14	0.0414 (11)	0.0377 (11)	0.0340 (11)	−0.0031 (10)	0.0095 (8)	−0.0006 (8)
C15	0.0362 (11)	0.0478 (12)	0.0345 (11)	0.0029 (10)	0.0118 (8)	−0.0046 (9)
C16	0.0351 (10)	0.0361 (11)	0.0350 (11)	0.0060 (9)	0.0066 (8)	−0.0062 (8)
C21	0.0322 (10)	0.0411 (11)	0.0282 (10)	0.0019 (9)	0.0060 (8)	0.0020 (8)
C22	0.0395 (11)	0.0493 (12)	0.0290 (10)	−0.0045 (10)	0.0101 (8)	0.0015 (9)
C23	0.0479 (12)	0.0402 (12)	0.0328 (11)	0.0009 (11)	0.0022 (9)	0.0008 (9)
C24	0.0470 (12)	0.0544 (14)	0.0270 (10)	0.0149 (11)	0.0073 (9)	0.0011 (9)
C25	0.0359 (11)	0.0656 (15)	0.0295 (11)	0.0086 (11)	0.0123 (8)	0.0125 (10)
C26	0.0344 (10)	0.0437 (12)	0.0324 (11)	−0.0035 (10)	0.0035 (8)	0.0093 (9)
C31	0.0311 (10)	0.0476 (12)	0.0308 (10)	−0.0016 (10)	0.0100 (8)	0.0028 (9)
C32	0.0377 (11)	0.0403 (12)	0.0398 (12)	−0.0033 (10)	0.0135 (9)	0.0011 (9)
C33	0.0429 (12)	0.0562 (14)	0.0353 (12)	−0.0120 (11)	0.0104 (9)	−0.0034 (10)
C34	0.0318 (10)	0.0709 (16)	0.0372 (12)	−0.0043 (11)	0.0067 (9)	0.0091 (11)
C35	0.0319 (10)	0.0552 (14)	0.0482 (13)	0.0096 (11)	0.0159 (9)	0.0081 (11)
C36	0.0391 (11)	0.0450 (12)	0.0361 (11)	0.0010 (10)	0.0168 (9)	−0.0033 (9)

Geometric parameters (Å, °)

F22—C22	1.338 (2)	C12—C13	1.361 (3)
F23—C23	1.340 (3)	C13—C14	1.401 (3)
F24—C24	1.341 (2)	C13—H13	0.9500
F25—C25	1.345 (2)	C14—C15	1.382 (3)
F26—C26	1.330 (2)	C14—H14	0.9500
F32—C32	1.334 (2)	C15—C16	1.392 (3)
F33—C33	1.339 (3)	C15—H15	0.9500
F34—C34	1.340 (3)	C16—H16	0.9500
F35—C35	1.333 (2)	C21—C22	1.381 (3)

F36—C36	1.337 (2)	C21—C26	1.390 (3)
O11—C11	1.390 (2)	C22—C23	1.374 (3)
O11—C10	1.399 (2)	C23—C24	1.371 (3)
O12—C12	1.397 (2)	C24—C25	1.369 (3)
O12—C10	1.399 (2)	C25—C26	1.374 (3)
O21—C21	1.383 (2)	C31—C32	1.380 (3)
O21—C10	1.401 (2)	C31—C36	1.381 (3)
O31—C31	1.385 (2)	C32—C33	1.384 (3)
O31—C10	1.387 (2)	C33—C34	1.370 (3)
C11—C16	1.372 (3)	C34—C35	1.374 (3)
C11—C12	1.373 (3)	C35—C36	1.378 (3)
C11—O11—C10	105.85 (14)	F22—C22—C21	118.99 (19)
C12—O12—C10	105.80 (14)	C23—C22—C21	121.41 (19)
C21—O21—C10	115.16 (15)	F23—C23—C24	120.6 (2)
C31—O31—C10	117.43 (15)	F23—C23—C22	120.0 (2)
O31—C10—O11	112.53 (16)	C24—C23—C22	119.4 (2)
O31—C10—O12	111.37 (16)	F24—C24—C25	120.2 (2)
O11—C10—O12	108.74 (15)	F24—C24—C23	119.6 (2)
O31—C10—O21	105.81 (15)	C25—C24—C23	120.2 (2)
O11—C10—O21	105.33 (15)	F25—C25—C24	119.5 (2)
O12—C10—O21	112.94 (16)	F25—C25—C26	119.9 (2)
C16—C11—C12	122.06 (18)	C24—C25—C26	120.56 (19)
C16—C11—O11	129.01 (17)	F26—C26—C25	119.47 (19)
C12—C11—O11	108.91 (16)	F26—C26—C21	120.50 (19)
C13—C12—C11	123.04 (18)	C25—C26—C21	120.0 (2)
C13—C12—O12	128.17 (17)	C32—C31—C36	118.80 (19)
C11—C12—O12	108.78 (16)	C32—C31—O31	120.32 (19)
C12—C13—C14	115.80 (18)	C36—C31—O31	120.39 (18)
C12—C13—H13	122.1	F32—C32—C31	120.28 (18)
C14—C13—H13	122.1	F32—C32—C33	119.27 (19)
C15—C14—C13	121.31 (19)	C31—C32—C33	120.5 (2)
C15—C14—H14	119.3	F33—C33—C34	120.5 (2)
C13—C14—H14	119.3	F33—C33—C32	119.7 (2)
C14—C15—C16	121.79 (18)	C34—C33—C32	119.8 (2)
C14—C15—H15	119.1	F34—C34—C33	119.8 (2)
C16—C15—H15	119.1	F34—C34—C35	119.7 (2)
C11—C16—C15	115.97 (18)	C33—C34—C35	120.5 (2)
C11—C16—H16	122.0	F35—C35—C34	120.38 (19)
C15—C16—H16	122.0	F35—C35—C36	120.2 (2)
C22—C21—O21	119.04 (17)	C34—C35—C36	119.4 (2)
C22—C21—C26	118.37 (19)	F36—C36—C35	118.72 (19)
O21—C21—C26	122.43 (19)	F36—C36—C31	120.32 (19)
F22—C22—C23	119.6 (2)	C35—C36—C31	120.94 (19)

Fig. 1


C15
C14
C16
C13
C11
C12
O11
O12
F33
F32
C33
C32
C10
O21
F22
C31
C22
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C26
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Fig. 2