Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-(Ylomethyl)cyclopentyl 1′,2′-phenylene orthocarbonate

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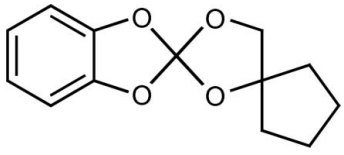
Received 3 October 2007; accepted 9 October 2007

Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.003 Å; R factor = 0.036; wR factor = 0.093; data-to-parameter ratio = 8.8.

The dispiro title compound (systematic name: 1,4,6,13-tetraoxa-2,3-benzodispiro[4.1.4.2]tridecane), $C_{13}H_{14}O_4$, is an asymmetric orthocarbonic acid ester of an aromatic and an aliphatic vicinal diol. C—O bond lengths at the orthoester centre show a typical difference of about 0.06 Å, as has been observed for related spiro esters with an aliphatic component that does not impose steric strain in the vicinity of the orthocarbonic acid centre. The C—O bond-length differences are also observed in density functional theory (DFT) calculations, thus ruling out a decisive influence of intermolecular forces in the crystal structure. The crystal structure is a polar arrangement of the ester molecules established by van der Waals interactions and, atypically for this class of compounds, by a relatively short C—H···O hydrogen bond.

Related literature

For the synthesis of the title compound, see Komatsu *et al.* (1992). For related compounds, see Betz *et al.* (2007*a*,*b*,*c*).



Experimental

Crystal data

$C_{13}H_{14}O_4$
M_r = 234.24
Orthorhombic, $Pna2_1$
a = 21.3198 (5) Å
b = 6.1626 (2) Å
c = 8.6023 (3) Å
V = 1130.22 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 200 (2) K
0.20 × 0.16 × 0.12 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
2416 measured reflections
1363 independent reflections
1217 reflections with $I > 2\sigma(I)$
R_{int} = 0.013

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.093
S = 1.04
1363 reflections
155 parameters
1 restraint
Only H-atom displacement parameters refined
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Table 1
Selected bond lengths (Å).

O11—C10	1.376 (3)	O21—C10	1.425 (2)
O12—C10	1.364 (2)	O22—C10	1.425 (3)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C11—H112···O12^{i}	0.99	2.37	3.229 (3)	144

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

Data collection: *COLLECT* (Nonius, 2004); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); 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*.

The authors thank Moritz Reichvilser for professional support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RN2032).

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  doi:10.1107/S1600536807049434

supplementary materials

Acta Cryst. (2007). E**63**, o4300 [**doi:10.1107/S1600536807049434**]

1-(Ylomethyl)cyclopentyl 1',2'-phenylene orthocarbonate

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Comment

The title compound was prepared in order to compare its NMR-spectroscopic data with those of similar silicon compounds. The aliphatic backbone models the binding capabilities of the 1,2-dihydroxy part of a ketofuranose.

In the molecule, a central C atom is chelated by bidentate substituents derived from dihydroxybenzene and 1-(hydroxymethyl)-cyclopentane-1-ol (Fig. 1). C—O bond lengths show significant differences spanning from about 1.36 to 1.43 Å. The significant differences observed in the C—O bond lengths is a molecular property, reproduced by DFT calculations at the B3LYP/6–31+G(d,p) level of theory within 2 pm and is therefore not induced by the environment in the solid. The five-membered chelate ring stemming from the aliphatic diol adopts a *twist* conformation on C11—C12. The cyclopentane ring of the aliphatic diol's backbone is present in an envelope conformation.

In the crystal structure, the aromatic moieties are arranged skew to each other (Fig. 2). Most unusual for this class of compounds is a weak C—H···O bond with a D···A distance of 3.23 Å (typically, the onset of D···A distances is close to 3.6 Å both for related compounds as well as for other interactions in the title ester).

Experimental

The title compound was prepared in adoption of a published procedure (Komatsu *et al.*, 1992) upon reaction of 1-(hydroxymethyl)-cyclopentane-1-ol with 2,2-dichlorobenzo[1.3]dioxol in dichloromethane in the presence of pyridine. Crystals suitable for X-ray analysis were obtained after recrystallization from boiling ethyl acetate.

Refinement

All H atoms were located in a difference map and refined as riding on their parent atoms. One common isotropic displacement parameter for all H atoms was refined.

Due to the absence of significant anomalous scattering the absolute structure parameter, which is 1.1 with an estimated standard deviation of 1.3 for the unmerged data set, is meaningless. Thus, Friedel opposites (1049 pairs) have been merged. The assigned polarity of the structure is thus arbitrary.

Figures

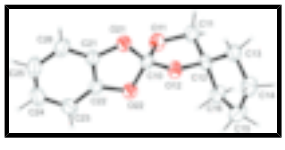

Fig. 1. The molecular structure of (I), with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level) for non-H atoms.

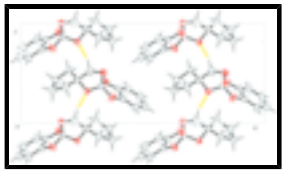

Fig. 2. The packing of (I), viewed along [0 1 0]. The tabulated C—H···O hydrogen bonds are drawn as yellow bars.

1,4,6,13-Tetraoxa-2,3-benzodispiro[4.1.4.2]tridecanecyclopentanespiro-4'-(1,3-dioxolane)-2'-spiro-2''-1,3-benzodioxole]

Crystal data

$C_{13}H_{14}O_4$	F_{000} = 496
M_r = 234.24	D_x = 1.377 Mg m^{-3}
Orthorhombic, *Pna*2$_1$	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: P 2c -2n	Cell parameters from 15992 reflections
a = 21.3198 (5) Å	θ = 3.1–27.5º
b = 6.1626 (2) Å	μ = 0.10 mm^{-1}
c = 8.6023 (3) Å	*T* = 200 (2) K
V = 1130.22 (6) Å^3	Block, colourless
Z = 4	0.20 × 0.16 × 0.12 mm

Data collection

Nonius KappaCCD diffractometer	1217 reflections with $I > 2\sigma(I)$
Radiation source: rotating anode	R_{int} = 0.013
Monochromator: MONTEL, graded multilayered X-ray optics	θ_{max} = 27.5º
T = 200(2) K	θ_{min} = 3.4º
CCD; rotation images; thick slices scans	*h* = −26→26
Absorption correction: none	*k* = −7→8
2416 measured reflections	*l* = −11→11
1363 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: difference Fourier map
Least-squares matrix: full	Only H-atom displacement parameters refined
$R[F^2 > 2\sigma(F^2)]$ = 0.036	$w = 1/[\sigma^2(F_o^2) + (0.0566P)^2 + 0.1517P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.093	$(\Delta/\sigma)_{max} < 0.001$
S = 1.04	$\Delta\rho_{max}$ = 0.14 e Å^{-3}
1363 reflections	$\Delta\rho_{min}$ = −0.17 e Å^{-3}
155 parameters	Extinction correction: none
1 restraint	

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O11	0.16273 (7)	0.2541 (3)	1.0057 (2)	0.0420 (4)
O12	0.23016 (6)	0.3539 (2)	0.81906 (19)	0.0356 (4)
O21	0.12779 (7)	0.3139 (2)	0.75454 (19)	0.0391 (4)
O22	0.15616 (7)	0.5972 (2)	0.9099 (2)	0.0399 (4)
C10	0.17048 (9)	0.3771 (3)	0.8736 (2)	0.0327 (4)
C11	0.21757 (10)	0.1210 (4)	1.0232 (3)	0.0456 (6)
H111	0.2113	−0.0233	0.9751	0.066 (3)*
H112	0.2283	0.1014	1.1343	0.066 (3)*
C12	0.26835 (9)	0.2476 (3)	0.9390 (3)	0.0335 (4)
C13	0.31944 (10)	0.1162 (4)	0.8626 (3)	0.0452 (6)
H131	0.3041	0.0507	0.7646	0.066 (3)*
H132	0.3338	−0.0014	0.9325	0.066 (3)*
C14	0.37283 (11)	0.2748 (4)	0.8298 (4)	0.0497 (6)
H141	0.3730	0.3172	0.7188	0.066 (3)*
H142	0.4138	0.2079	0.8554	0.066 (3)*
C15	0.36089 (11)	0.4726 (4)	0.9329 (4)	0.0504 (6)
H151	0.3977	0.5016	0.9998	0.066 (3)*
H152	0.3527	0.6026	0.8684	0.066 (3)*
C16	0.30353 (10)	0.4186 (4)	1.0323 (3)	0.0403 (5)
H161	0.3163	0.3607	1.1348	0.066 (3)*
H162	0.2772	0.5490	1.0484	0.066 (3)*
C21	0.08294 (9)	0.4745 (3)	0.7445 (3)	0.0350 (4)
C22	0.09975 (9)	0.6449 (3)	0.8379 (3)	0.0353 (5)
C23	0.06480 (10)	0.8315 (4)	0.8493 (3)	0.0466 (6)
H23	0.0769	0.9494	0.9138	0.066 (3)*
C24	0.01031 (11)	0.8357 (4)	0.7596 (4)	0.0569 (7)
H24	−0.0156	0.9610	0.7628	0.066 (3)*
C25	−0.00716 (11)	0.6639 (5)	0.6665 (4)	0.0579 (8)
H25	−0.0450	0.6728	0.6085	0.066 (3)*
C26	0.02957 (10)	0.4775 (5)	0.6558 (3)	0.0504 (6)
H26	0.0182	0.3592	0.5908	0.066 (3)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O11	0.0355 (7)	0.0514 (9)	0.0391 (8)	0.0008 (7)	0.0033 (7)	0.0136 (7)
O12	0.0297 (7)	0.0467 (8)	0.0305 (7)	0.0021 (5)	−0.0001 (6)	0.0073 (7)
O21	0.0375 (8)	0.0378 (7)	0.0419 (9)	0.0026 (6)	−0.0104 (7)	−0.0061 (7)
O22	0.0369 (7)	0.0363 (7)	0.0464 (9)	0.0017 (6)	−0.0093 (7)	−0.0062 (7)
C10	0.0306 (9)	0.0367 (10)	0.0308 (10)	−0.0005 (7)	−0.0032 (8)	0.0022 (8)
C11	0.0379 (11)	0.0467 (12)	0.0523 (15)	0.0006 (9)	−0.0029 (11)	0.0169 (12)
C12	0.0337 (9)	0.0352 (9)	0.0317 (10)	0.0040 (8)	−0.0046 (9)	0.0035 (8)

C13	0.0414 (12)	0.0419 (11)	0.0524 (15)	0.0088 (9)	−0.0043 (11)	−0.0063 (11)
C14	0.0422 (12)	0.0576 (14)	0.0494 (14)	0.0060 (10)	0.0048 (11)	−0.0045 (13)
C15	0.0395 (12)	0.0557 (14)	0.0560 (16)	−0.0084 (10)	0.0042 (12)	−0.0127 (13)
C16	0.0376 (10)	0.0499 (12)	0.0335 (11)	0.0004 (9)	−0.0025 (9)	−0.0070 (10)
C21	0.0286 (9)	0.0381 (10)	0.0383 (11)	0.0002 (7)	−0.0017 (9)	0.0052 (9)
C22	0.0278 (9)	0.0384 (10)	0.0398 (12)	−0.0006 (7)	0.0012 (9)	0.0039 (9)
C23	0.0402 (11)	0.0363 (10)	0.0632 (17)	0.0033 (8)	0.0099 (12)	0.0081 (11)
C24	0.0371 (12)	0.0540 (14)	0.0794 (19)	0.0133 (10)	0.0094 (13)	0.0188 (16)
C25	0.0327 (12)	0.0750 (18)	0.0661 (19)	0.0068 (11)	−0.0066 (12)	0.0179 (16)
C26	0.0368 (11)	0.0613 (15)	0.0532 (17)	−0.0052 (10)	−0.0110 (11)	0.0047 (13)

Geometric parameters (Å, °)

O11—C10	1.376 (3)	C14—H141	0.9900
O11—C11	1.436 (3)	C14—H142	0.9900
O12—C10	1.364 (2)	C15—C16	1.529 (3)
O12—C12	1.469 (3)	C15—H151	0.9900
O21—C21	1.379 (2)	C15—H152	0.9900
O21—C10	1.425 (2)	C16—H161	0.9900
O22—C22	1.384 (3)	C16—H162	0.9900
O22—C10	1.425 (3)	C21—C22	1.370 (3)
C11—C12	1.519 (3)	C21—C26	1.370 (3)
C11—H111	0.9900	C22—C23	1.374 (3)
C11—H112	0.9900	C23—C24	1.395 (4)
C12—C13	1.508 (3)	C23—H23	0.9500
C12—C16	1.522 (3)	C24—C25	1.379 (4)
C13—C14	1.527 (3)	C24—H24	0.9500
C13—H131	0.9900	C25—C26	1.393 (4)
C13—H132	0.9900	C25—H25	0.9500
C14—C15	1.529 (4)	C26—H26	0.9500
C10—O11—C11	107.68 (17)	C15—C14—H142	110.5
C10—O12—C12	108.82 (16)	H141—C14—H142	108.7
C21—O21—C10	106.95 (16)	C16—C15—C14	106.5 (2)
C22—O22—C10	106.85 (15)	C16—C15—H151	110.4
O12—C10—O11	109.76 (16)	C14—C15—H151	110.4
O12—C10—O21	108.67 (17)	C16—C15—H152	110.4
O11—C10—O21	111.48 (16)	C14—C15—H152	110.4
O12—C10—O22	112.06 (16)	H151—C15—H152	108.6
O11—C10—O22	108.52 (18)	C12—C16—C15	104.49 (19)
O21—C10—O22	106.34 (15)	C12—C16—H161	110.9
O11—C11—C12	103.69 (17)	C15—C16—H161	110.9
O11—C11—H111	111.0	C12—C16—H162	110.9
C12—C11—H111	111.0	C15—C16—H162	110.9
O11—C11—H112	111.0	H161—C16—H162	108.9
C12—C11—H112	111.0	C22—C21—C26	122.2 (2)
H111—C11—H112	109.0	C22—C21—O21	109.40 (17)
O12—C12—C13	109.48 (19)	C26—C21—O21	128.4 (2)
O12—C12—C11	99.74 (15)	C21—C22—C23	122.8 (2)
C13—C12—C11	116.55 (19)	C21—C22—O22	109.09 (17)

O12—C12—C16	109.56 (18)	C23—C22—O22	128.1 (2)
C13—C12—C16	104.22 (17)	C22—C23—C24	115.3 (2)
C11—C12—C16	117.1 (2)	C22—C23—H23	122.3
C12—C13—C14	105.98 (19)	C24—C23—H23	122.3
C12—C13—H131	110.5	C25—C24—C23	122.1 (2)
C14—C13—H131	110.5	C25—C24—H24	118.9
C12—C13—H132	110.5	C23—C24—H24	118.9
C14—C13—H132	110.5	C24—C25—C26	121.3 (2)
H131—C13—H132	108.7	C24—C25—H25	119.3
C13—C14—C15	106.2 (2)	C26—C25—H25	119.3
C13—C14—H141	110.5	C21—C26—C25	116.2 (3)
C15—C14—H141	110.5	C21—C26—H26	121.9
C13—C14—H142	110.5	C25—C26—H26	121.9
C12—O12—C10—O11	−12.8 (2)	C12—C13—C14—C15	18.0 (3)
C12—O12—C10—O21	−134.99 (17)	C13—C14—C15—C16	4.0 (3)
C12—O12—C10—O22	107.79 (18)	O12—C12—C16—C15	−81.8 (2)
C11—O11—C10—O12	−8.8 (2)	C13—C12—C16—C15	35.3 (2)
C11—O11—C10—O21	111.7 (2)	C11—C12—C16—C15	165.65 (19)
C11—O11—C10—O22	−131.51 (18)	C14—C15—C16—C12	−24.2 (3)
C21—O21—C10—O12	−132.10 (17)	C10—O21—C21—C22	7.0 (2)
C21—O21—C10—O11	106.80 (19)	C10—O21—C21—C26	−174.2 (2)
C21—O21—C10—O22	−11.3 (2)	C26—C21—C22—C23	−0.3 (4)
C22—O22—C10—O12	129.99 (18)	O21—C21—C22—C23	178.5 (2)
C22—O22—C10—O11	−108.65 (18)	C26—C21—C22—O22	−178.7 (2)
C22—O22—C10—O21	11.4 (2)	O21—C21—C22—O22	0.2 (2)
C10—O11—C11—C12	25.6 (2)	C10—O22—C22—C21	−7.3 (2)
C10—O12—C12—C13	150.00 (18)	C10—O22—C22—C23	174.5 (2)
C10—O12—C12—C11	27.2 (2)	C21—C22—C23—C24	0.4 (4)
C10—O12—C12—C16	−96.29 (19)	O22—C22—C23—C24	178.4 (2)
O11—C11—C12—O12	−31.1 (2)	C22—C23—C24—C25	0.2 (4)
O11—C11—C12—C13	−148.8 (2)	C23—C24—C25—C26	−0.9 (5)
O11—C11—C12—C16	86.9 (2)	C22—C21—C26—C25	−0.4 (4)
O12—C12—C13—C14	84.1 (2)	O21—C21—C26—C25	−179.0 (2)
C11—C12—C13—C14	−163.8 (2)	C24—C25—C26—C21	1.0 (4)
C16—C12—C13—C14	−33.1 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H112···O12^{i}	0.99	2.37	3.229 (3)	144

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

Fig. 1

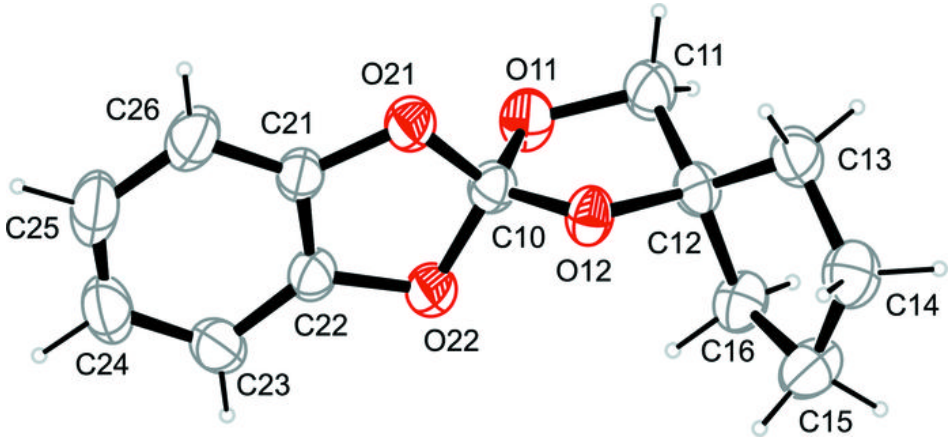
C11
O11
O21
C26
C21
C13
C25
C10
O12
C12
C14
C22
O22
C16
C24
C23
C15

Fig. 2