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5,7,13,14-Tetraoxatrispiro-[2.2.2.2^9.2^6.2^3]pentadecane

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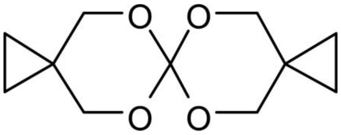
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.003 Å; R factor = 0.041; wR factor = 0.098; data-to-parameter ratio = 10.5.

The title compound, $C_{11}H_{16}O_4$, a diol ester of the nonexistent orthocarbonic acid $C(OH)_4$, was prepared for the purpose of collecting NMR data on orthocarbonates and for comparison with similar silicon compounds. The molecule shows chemical but not crystallographic C_2 symmetry. The 1,3-dioxane rings adopt chair conformations. The central C atom is in a distorted tetrahedral environment of four O atoms.

Related literature

For related literature, see: Bromley *et al.* (1998); Mues & Buysch (1990); Narasimhamurthy *et al.* (1990).



Experimental

Crystal data

$C_{11}H_{16}O_4$
M_r = 212.24
Orthorhombic, $P2_12_12_1$
a = 6.2980 (8) Å
b = 7.1520 (8) Å
c = 23.508 (3) Å
V = 1058.9 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 200 (3) K
0.26 × 0.19 × 0.10 mm

Data collection

Oxford Diffraction Xcalibur3 diffractometer
Absorption correction: none
6256 measured reflections
1442 independent reflections
1237 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.098
S = 1.08
1442 reflections
137 parameters
Only H-atom displacement parameters refined
$\Delta\rho_{\max}$ = 0.16 e Å^{-3}
$\Delta\rho_{\min}$ = −0.17 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: *SIR97* (Altomare *et al.*, 1999); 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 Sandra Albrecht and Dr Peter Mayer for professional support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2444).

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supplementary materials

Acta Cryst. (2007). E**63**, o3558 [**doi:10.1107/S1600536807034915**]

5,7,13,14-Tetraoxatrispiro[2.2.2.2^9.2^6.2^3]pentadecane

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

Comment

The title compound, $C_{11}H_{16}O_4$, a diol ester of the nonexistent orthocarbonic acid $C(OH)_4$, was prepared for the purpose of collecting NMR data on orthocarbonates and for comparison with similar silicon compounds. It was obtained as the product of the reaction between dichlorodiphenoxymethane and 1,1-bis(hydroxymethyl)cyclopropane.

The molecular structure (Fig. 1) shows C_2 symmetry. The 1,3-dioxane rings adopt chair conformations. The central carbon atom is in a distorted tetrahedral environment of four oxygen atoms. O11 and O21 are equatorial substituents, O13 and O23 are axial substituents, of their opposing six-membered rings. Due to the anomeric effect, the axial C10–O bonds are slightly, but significantly, longer than the corresponding equatorial bonds. The endocyclic O–C–O angles are significantly larger than the exocyclic ones.

The molecular packing is shown in Fig. 2.

Experimental

The title compound was prepared according to a literature procedure (Mues & Buysch, 1990) by the reaction of dichlorodiphenoxymethane, $(PhO)_2CCl_2$, with 1,1-bis(hydroxymethyl)cyclopropane. The crude product was recrystallized from boiling ethyl acetate.

Spectroscopic data: ^{1}H NMR (400 MHz, $CDCl_3$, 22 °C) δ/p.p.m.: 3.79 (s, 8 H, OCH_2), 0.55 (s, 8 H, CH_2). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$, 24 °C) δ/p.p.m.: 114.8 (CO_4), 69.1 (OCH_2), 16.2 (C_q), 8.9 (CH_2). HRMS *m*/*z* calculated for $C_{11}H_{16}O_4$ (M^+): 212.1043; found: 212.1043.

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 to $U_{iso}(H) = 0.0420\ (17)$.

Due to the absence of significant anomalous scattering the absolute structure parameter (Flack, 1983), which is −3.1 with an estimated standard deviation of 1.2 for the unmerged data set, is meaningless. Thus, Friedel opposites (995 pairs) have been merged and the absolute configuration has been arbitrarily chosen.

Figures

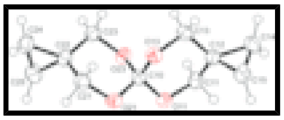

Fig. 1. The molecular structure of (I), with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level) for non-H atoms.

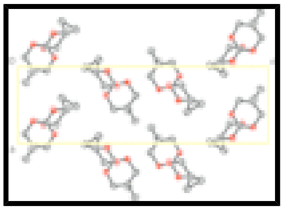

Fig. 2. The packing of (I), viewed along [$\bar{1}$ 0 0]. H atoms omitted for clarity.

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Crystal data

$C_{11}H_{16}O_4$

$M_r = 212.24$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 6.2980$ (8) Å

$b = 7.1520$ (8) Å

$c = 23.508$ (3) Å

$V = 1058.9$ (2) Å^3

$Z = 4$

$F_{000} = 456$

$D_x = 1.331$ (1) Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

$\theta = 4.4$–27.5°

$\mu = 0.10$ mm^{-1}

$T = 200$ (3) K

Block, colourless

0.26 × 0.19 × 0.10 mm

Data collection

Oxford XCalibur3
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 200(3)$ K

ω scans

Absorption correction: none

6256 measured reflections

1442 independent reflections

1237 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.044$

$\theta_{\max} = 27.6°$

$\theta_{\min} = 4.4°$

$h = -8 \rightarrow 5$

$k = -8 \rightarrow 9$

$l = -28 \rightarrow 30$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.041$

$wR(F^2) = 0.098$

Only H-atom displacement parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0525P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.16$ e Å^{-3}

$S = 1.08$	$\Delta\rho_{\min} = -0.17$ e Å^{-3}
1442 reflections	Extinction correction: none
137 parameters	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Hydrogen site location: difference Fourier map	

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}
O11	0.6614 (3)	0.7090 (2)	0.06259 (6)	0.0344 (4)
O13	0.6407 (3)	0.8773 (2)	0.14710 (6)	0.0336 (4)
O21	0.6058 (3)	0.5650 (2)	0.14343 (6)	0.0352 (4)
O23	0.3442 (2)	0.7528 (2)	0.10339 (6)	0.0318 (4)
C10	0.5614 (4)	0.7292 (3)	0.11423 (9)	0.0296 (5)
C11	0.6546 (4)	0.8783 (3)	0.02814 (9)	0.0348 (5)
H111	0.5061	0.9062	0.0172	0.0420 (17)*
H112	0.7385	0.8602	−0.0070	0.0420 (17)*
C12	0.7440 (4)	1.0372 (3)	0.06173 (10)	0.0317 (5)
C13	0.6326 (4)	1.0554 (3)	0.11755 (9)	0.0345 (5)
H131	0.7021	1.1532	0.1408	0.0420 (17)*
H132	0.4830	1.0925	0.1113	0.0420 (17)*
C14	0.8256 (4)	1.2072 (3)	0.03185 (11)	0.0405 (6)
H141	0.8046	1.3298	0.0505	0.0420 (17)*
H142	0.8179	1.2095	−0.0102	0.0420 (17)*
C15	0.9787 (4)	1.0718 (3)	0.05907 (11)	0.0388 (6)
H151	1.0645	0.9912	0.0337	0.0420 (17)*
H152	1.0512	1.1116	0.0944	0.0420 (17)*
C21	0.4940 (4)	0.5515 (4)	0.19660 (10)	0.0364 (5)
H211	0.5467	0.6485	0.2231	0.0420 (17)*
H212	0.5206	0.4276	0.2140	0.0420 (17)*
C22	0.2604 (4)	0.5770 (3)	0.18734 (10)	0.0321 (5)
C23	0.2164 (4)	0.7525 (3)	0.15418 (9)	0.0347 (5)
H231	0.0641	0.7583	0.1439	0.0420 (17)*
H232	0.2507	0.8634	0.1777	0.0420 (17)*
C24	0.1061 (4)	0.5146 (4)	0.23182 (9)	0.0421 (6)

H241	−0.0228	0.5914	0.2378	0.0420 (17)*
H242	0.1639	0.4555	0.2666	0.0420 (17)*
C25	0.1253 (4)	0.4088 (3)	0.17680 (10)	0.0412 (6)
H251	0.1948	0.2847	0.1778	0.0420 (17)*
H252	0.0081	0.4205	0.1490	0.0420 (17)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O11	0.0381 (9)	0.0323 (8)	0.0328 (8)	0.0006 (7)	0.0067 (8)	−0.0034 (6)
O13	0.0376 (9)	0.0336 (8)	0.0296 (7)	−0.0023 (7)	−0.0019 (8)	−0.0025 (6)
O21	0.0300 (8)	0.0349 (8)	0.0408 (8)	0.0072 (7)	0.0012 (8)	0.0047 (7)
O23	0.0246 (7)	0.0404 (8)	0.0303 (7)	0.0003 (7)	−0.0017 (7)	0.0030 (7)
C10	0.0285 (11)	0.0304 (11)	0.0299 (10)	0.0008 (9)	0.0012 (9)	0.0001 (9)
C11	0.0364 (13)	0.0385 (12)	0.0294 (11)	−0.0017 (11)	0.0032 (11)	0.0028 (9)
C12	0.0291 (11)	0.0323 (11)	0.0336 (11)	0.0020 (10)	0.0000 (10)	0.0035 (9)
C13	0.0379 (13)	0.0283 (11)	0.0374 (11)	−0.0007 (11)	0.0011 (11)	−0.0040 (9)
C14	0.0390 (13)	0.0346 (12)	0.0478 (13)	0.0018 (11)	−0.0009 (12)	0.0080 (10)
C15	0.0297 (12)	0.0367 (13)	0.0499 (14)	−0.0004 (10)	−0.0015 (12)	0.0004 (11)
C21	0.0365 (13)	0.0378 (13)	0.0350 (12)	0.0011 (11)	−0.0026 (11)	0.0073 (10)
C22	0.0310 (11)	0.0355 (13)	0.0299 (11)	−0.0011 (10)	0.0003 (10)	0.0011 (9)
C23	0.0297 (11)	0.0392 (13)	0.0352 (11)	0.0048 (10)	0.0036 (10)	0.0016 (10)
C24	0.0409 (15)	0.0496 (14)	0.0359 (12)	−0.0013 (11)	0.0071 (12)	0.0060 (11)
C25	0.0412 (15)	0.0391 (13)	0.0432 (13)	−0.0065 (12)	−0.0047 (12)	−0.0001 (10)

Geometric parameters (Å, °)

O11—C10	1.375 (2)	C14—H141	0.9900
O11—C11	1.457 (2)	C14—H142	0.9900
O13—C10	1.403 (2)	C15—H151	0.9900
O13—C13	1.452 (2)	C15—H152	0.9900
O21—C10	1.389 (2)	C21—C22	1.498 (3)
O21—C21	1.438 (3)	C21—H211	0.9900
O23—C10	1.401 (3)	C21—H212	0.9900
O23—C23	1.440 (2)	C22—C25	1.494 (3)
C11—C12	1.494 (3)	C22—C24	1.495 (3)
C11—H111	0.9900	C22—C23	1.504 (3)
C11—H112	0.9900	C23—H231	0.9900
C12—C13	1.494 (3)	C23—H232	0.9900
C12—C14	1.495 (3)	C24—C25	1.503 (3)
C12—C15	1.500 (3)	C24—H241	0.9900
C13—H131	0.9900	C24—H242	0.9900
C13—H132	0.9900	C25—H251	0.9900
C14—C15	1.509 (3)	C25—H252	0.9900
C10—O11—C11	112.96 (16)	C12—C15—H151	117.8
C10—O13—C13	112.74 (15)	C14—C15—H151	117.8
C10—O21—C21	112.81 (16)	C12—C15—H152	117.8
C10—O23—C23	113.24 (16)	C14—C15—H152	117.8

O11—C10—O21	104.80 (16)	H151—C15—H152	114.9
O11—C10—O23	107.41 (17)	O21—C21—C22	110.28 (19)
O21—C10—O23	112.86 (17)	O21—C21—H211	109.6
O11—C10—O13	113.70 (17)	C22—C21—H211	109.6
O21—C10—O13	107.14 (15)	O21—C21—H212	109.6
O23—C10—O13	110.89 (17)	C22—C21—H212	109.6
O11—C11—C12	109.09 (17)	H211—C21—H212	108.1
O11—C11—H111	109.9	C25—C22—C24	60.39 (16)
C12—C11—H111	109.9	C25—C22—C21	119.0 (2)
O11—C11—H112	109.9	C24—C22—C21	120.0 (2)
C12—C11—H112	109.9	C25—C22—C23	118.8 (2)
H111—C11—H112	108.3	C24—C22—C23	119.5 (2)
C13—C12—C11	110.71 (19)	C21—C22—C23	111.0 (2)
C13—C12—C14	120.2 (2)	O23—C23—C22	109.15 (18)
C11—C12—C14	120.0 (2)	O23—C23—H231	109.9
C13—C12—C15	119.0 (2)	C22—C23—H231	109.9
C11—C12—C15	118.4 (2)	O23—C23—H232	109.9
C14—C12—C15	60.49 (16)	C22—C23—H232	109.9
O13—C13—C12	109.11 (17)	H231—C23—H232	108.3
O13—C13—H131	109.9	C22—C24—C25	59.77 (15)
C12—C13—H131	109.9	C22—C24—H241	117.8
O13—C13—H132	109.9	C25—C24—H241	117.8
C12—C13—H132	109.9	C22—C24—H242	117.8
H131—C13—H132	108.3	C25—C24—H242	117.8
C12—C14—C15	59.92 (15)	H241—C24—H242	114.9
C12—C14—H141	117.8	C22—C25—C24	59.84 (16)
C15—C14—H141	117.8	C22—C25—H251	117.8
C12—C14—H142	117.8	C24—C25—H251	117.8
C15—C14—H142	117.8	C22—C25—H252	117.8
H141—C14—H142	114.9	C24—C25—H252	117.8
C12—C15—C14	59.60 (15)	H251—C25—H252	114.9

C11—O11—C10—O21	−172.98 (17)	C14—C12—C13—O13	−158.8 (2)
C11—O11—C10—O23	66.8 (2)	C15—C12—C13—O13	−88.0 (2)
C11—O11—C10—O13	−56.3 (2)	C13—C12—C14—C15	108.4 (3)
C21—O21—C10—O11	−173.57 (17)	C11—C12—C14—C15	−107.7 (3)
C21—O21—C10—O23	−57.0 (2)	C13—C12—C15—C14	−110.3 (2)
C21—O21—C10—O13	65.3 (2)	C11—C12—C15—C14	110.3 (2)
C23—O23—C10—O11	172.79 (16)	C10—O21—C21—C22	54.1 (3)
C23—O23—C10—O21	57.8 (2)	O21—C21—C22—C25	91.4 (2)
C23—O23—C10—O13	−62.4 (2)	O21—C21—C22—C24	162.05 (19)
C13—O13—C10—O11	56.2 (2)	O21—C21—C22—C23	−51.9 (3)
C13—O13—C10—O21	171.48 (18)	C10—O23—C23—C22	−54.5 (2)
C13—O13—C10—O23	−65.0 (2)	C25—C22—C23—O23	−91.6 (2)
C10—O11—C11—C12	54.8 (2)	C24—C22—C23—O23	−161.94 (19)
O11—C11—C12—C13	−54.2 (3)	C21—C22—C23—O23	51.8 (3)
O11—C11—C12—C14	158.8 (2)	C21—C22—C24—C25	−108.4 (3)
O11—C11—C12—C15	88.3 (2)	C23—C22—C24—C25	108.3 (2)
C10—O13—C13—C12	−54.3 (3)	C21—C22—C25—C24	110.0 (2)
C11—C12—C13—O13	54.2 (3)	C23—C22—C25—C24	−109.5 (2)

Fig. 1

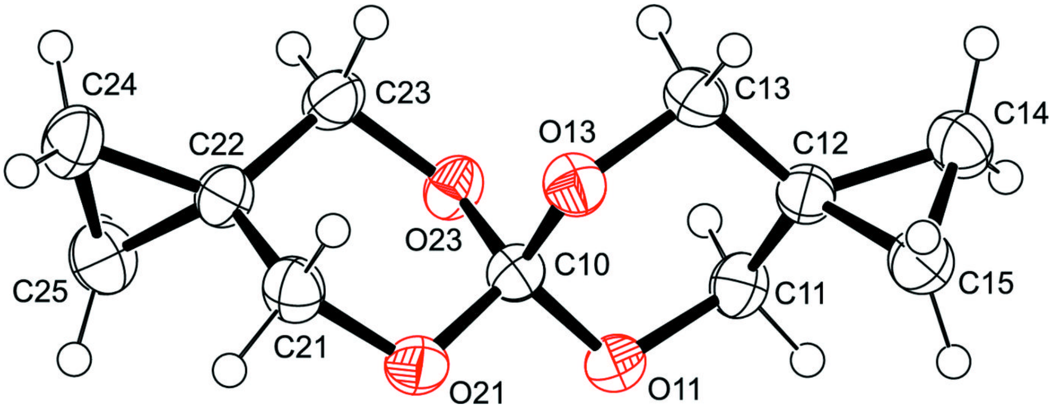

Fig. 2