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Norbornane-2,7-diyl 1′,2′-phenylene orthocarbonate

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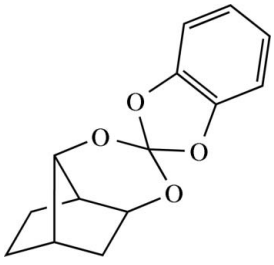
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.002 Å; R factor = 0.044; wR factor = 0.117; data-to-parameter ratio = 15.7.

In the title compound, $C_{14}H_{14}O_4$, which was synthesized in order to compare its NMR spectroscopic data with those of similar silicon compounds, the incorporation of chelating hydroxyl groups into the parent rigid bicyclic framework ensures that the resulting six-membered chelate ring has a boat conformation.

Related literature

For synthesis, see Mues & Buysch (1990).



Experimental

Crystal data

$C_{14}H_{14}O_4$
M_r = 246.26
Monoclinic, $P2_1/n$
a = 6.0509 (2) Å
b = 18.1250 (6) Å
c = 10.3711 (3) Å
β = 95.689 (2)°
V = 1131.82 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 200 (2) K
0.16 × 0.15 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
5009 measured reflections
2573 independent reflections
1779 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.117
S = 1.03
2573 reflections
164 parameters
Only H-atom displacement parameters refined
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Selected bond lengths (Å).

O12—C10	1.364 (2)	O21—C10	1.413 (2)
O17—C10	1.369 (2)	O22—C10	1.451 (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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2315).

References

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Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

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supplementary materials

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Norbornane-2,7-diyl 1',2'-phenylene orthocarbonate

R. Betz and P. Klüfers

Comment

The title compound, $C_{14}H_{14}O_4$, was prepared for the purpose of collecting NMR data on orthocarbonates and for comparison with similar silicon compounds. The incorporation of the chelating hydroxyl groups into the rigid bicyclic framework ensures the six-membered chelate ring to be present in a boat conformation. The markedly large range of the C—O distances at the spiro center is in agreement with a DFT calculation on a single molecule, i. e., it is not indicative of special packing forces in the crystalline state.

The molecular structure (Fig. 1) shows a norbornane-2,7-dioxy and a a benzene-1,2-dioxy fragment attached to a carbon atom.

The molecular packing is shown in Figure 2.

Experimental

The title compound was prepared according to standard procedures (Mues & Buysch, 1990) upon reaction of 2,2-dichlorobenzo[1.3]dioxole with 2,7-norbornanediol in the presence of pyridine in dichloromethane. Crystals suitable for X-ray analysis were directly obtained from the recrystallized reaction product.

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) = 0.0463 (14).

Figures

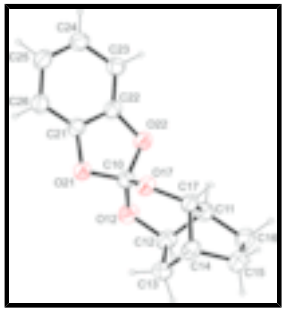


Fig. 1. The molecular of (I). Anisotropic displacement ellipsoids are drawn at the 50%-probability level for non-H atoms.

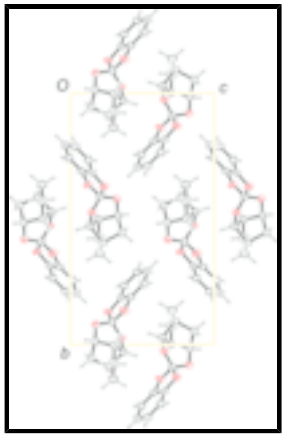


Fig. 2. The packing of (I) viewed along [1 0 0].

Norbornane-2,7-diyl 1',2'-phenylene orthocarbonate

Crystal data

$C_{14}H_{14}O_4$	F_{000} = 520
M_r = 246.26	D_x = 1.445 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 11723 reflections
a = 6.0509 (2) Å	θ = 3.1–27.5º
b = 18.1250 (6) Å	μ = 0.11 mm^{-1}
c = 10.3711 (3) Å	T = 200 (2) K
β = 95.689 (2)º	Block, colourless
V = 1131.82 (6) Å^3	0.16 × 0.15 × 0.10 mm
Z = 4	

Data collection

Nonius KappaCCD diffractometer	1779 reflections with $I > 2\sigma(I)$
Radiation source: rotating anode	R_{int} = 0.028
Monochromator: MONTEL, graded multilayered X-ray optics	θ_{max} = 27.5º
T = 200(2) K	θ_{min} = 3.6º
CCD; rotation images; thick slices scans	h = −7→7
Absorption correction: none	k = −23→23
5009 measured reflections	l = −13→13
2573 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.044	Only H-atom displacement parameters refined

$wR(F^2) = 0.117$	$w = 1/[\sigma^2(F_o^2) + (0.0525P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} < 0.001$
2573 reflections	$\Delta\rho_{max} = 0.19$ e Å^{-3}
164 parameters	$\Delta\rho_{min} = -0.20$ 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 $F^2 > 2\text{sigma}(F^2)$ 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}
O12	0.83506 (19)	0.08057 (6)	0.82385 (11)	0.0373 (3)
O17	0.97730 (18)	0.02608 (6)	0.64946 (11)	0.0327 (3)
O21	1.13089 (19)	0.12882 (6)	0.73797 (11)	0.0377 (3)
O22	0.78699 (19)	0.13656 (6)	0.62400 (11)	0.0382 (3)
C10	0.9290 (3)	0.09008 (9)	0.71081 (16)	0.0320 (4)
C11	0.5901 (3)	−0.00278 (9)	0.69471 (17)	0.0350 (4)
H11	0.5102	0.0399	0.6503	0.0463 (14)*
C12	0.7030 (3)	0.01341 (9)	0.82865 (16)	0.0358 (4)
H12	0.5917	0.0181	0.8934	0.0463 (14)*
C13	0.8536 (3)	−0.05454 (9)	0.85779 (16)	0.0393 (4)
H131	1.0068	−0.0395	0.8894	0.0463 (14)*
H132	0.7944	−0.0870	0.9230	0.0463 (14)*
C14	0.8472 (3)	−0.09294 (9)	0.72612 (17)	0.0359 (4)
H14	0.9839	−0.1216	0.7118	0.0463 (14)*
C15	0.6297 (3)	−0.13617 (10)	0.70228 (19)	0.0415 (4)
H151	0.6115	−0.1711	0.7739	0.0463 (14)*
H152	0.6220	−0.1637	0.6195	0.0463 (14)*
C16	0.4545 (3)	−0.07430 (10)	0.69715 (19)	0.0427 (4)
H161	0.3488	−0.0787	0.6183	0.0463 (14)*
H162	0.3706	−0.0759	0.7743	0.0463 (14)*
C17	0.7989 (3)	−0.02808 (9)	0.63485 (16)	0.0324 (4)
H17	0.7652	−0.0443	0.5428	0.0463 (14)*
C21	1.1277 (3)	0.18638 (8)	0.64979 (15)	0.0316 (4)
C22	0.9223 (3)	0.19053 (8)	0.58122 (15)	0.0317 (4)
C23	0.8708 (3)	0.24313 (9)	0.48849 (16)	0.0370 (4)

H23	0.7282	0.2457	0.4413	0.0463 (14)*
C24	1.0393 (3)	0.29288 (9)	0.46697 (16)	0.0389 (4)
H24	1.0114	0.3303	0.4034	0.0463 (14)*
C25	1.2457 (3)	0.28872 (9)	0.53632 (17)	0.0391 (4)
H25	1.3565	0.3235	0.5194	0.0463 (14)*
C26	1.2961 (3)	0.23468 (9)	0.63083 (16)	0.0365 (4)
H26	1.4378	0.2316	0.6789	0.0463 (14)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O12	0.0485 (7)	0.0298 (6)	0.0347 (6)	−0.0033 (5)	0.0092 (5)	−0.0051 (5)
O17	0.0337 (6)	0.0282 (6)	0.0374 (6)	−0.0027 (5)	0.0089 (5)	−0.0021 (5)
O21	0.0360 (7)	0.0316 (6)	0.0436 (7)	−0.0057 (5)	−0.0059 (5)	0.0068 (5)
O22	0.0330 (6)	0.0328 (6)	0.0476 (7)	−0.0042 (5)	−0.0030 (5)	0.0084 (5)
C10	0.0333 (9)	0.0272 (8)	0.0346 (8)	−0.0008 (7)	−0.0005 (7)	0.0016 (7)
C11	0.0299 (9)	0.0331 (9)	0.0419 (9)	0.0010 (7)	0.0025 (7)	0.0050 (7)
C12	0.0425 (10)	0.0318 (9)	0.0354 (9)	−0.0015 (7)	0.0143 (8)	−0.0020 (7)
C13	0.0448 (11)	0.0357 (9)	0.0364 (9)	−0.0001 (8)	−0.0009 (8)	0.0059 (7)
C14	0.0353 (9)	0.0290 (8)	0.0443 (9)	0.0015 (7)	0.0075 (8)	−0.0007 (7)
C15	0.0427 (10)	0.0348 (9)	0.0475 (10)	−0.0067 (8)	0.0066 (8)	−0.0010 (8)
C16	0.0337 (10)	0.0438 (10)	0.0506 (11)	−0.0060 (8)	0.0043 (8)	0.0033 (8)
C17	0.0349 (9)	0.0306 (8)	0.0323 (8)	−0.0070 (7)	0.0066 (7)	−0.0031 (7)
C21	0.0376 (9)	0.0241 (8)	0.0328 (8)	−0.0006 (7)	0.0024 (7)	−0.0002 (6)
C22	0.0351 (9)	0.0246 (8)	0.0356 (8)	−0.0035 (7)	0.0042 (7)	−0.0013 (6)
C23	0.0407 (10)	0.0318 (9)	0.0369 (9)	−0.0008 (7)	−0.0038 (8)	0.0012 (7)
C24	0.0515 (11)	0.0298 (9)	0.0350 (9)	−0.0030 (8)	0.0019 (8)	0.0036 (7)
C25	0.0450 (11)	0.0323 (9)	0.0407 (9)	−0.0102 (8)	0.0070 (8)	−0.0003 (7)
C26	0.0354 (9)	0.0335 (9)	0.0397 (9)	−0.0040 (7)	−0.0002 (7)	−0.0025 (7)

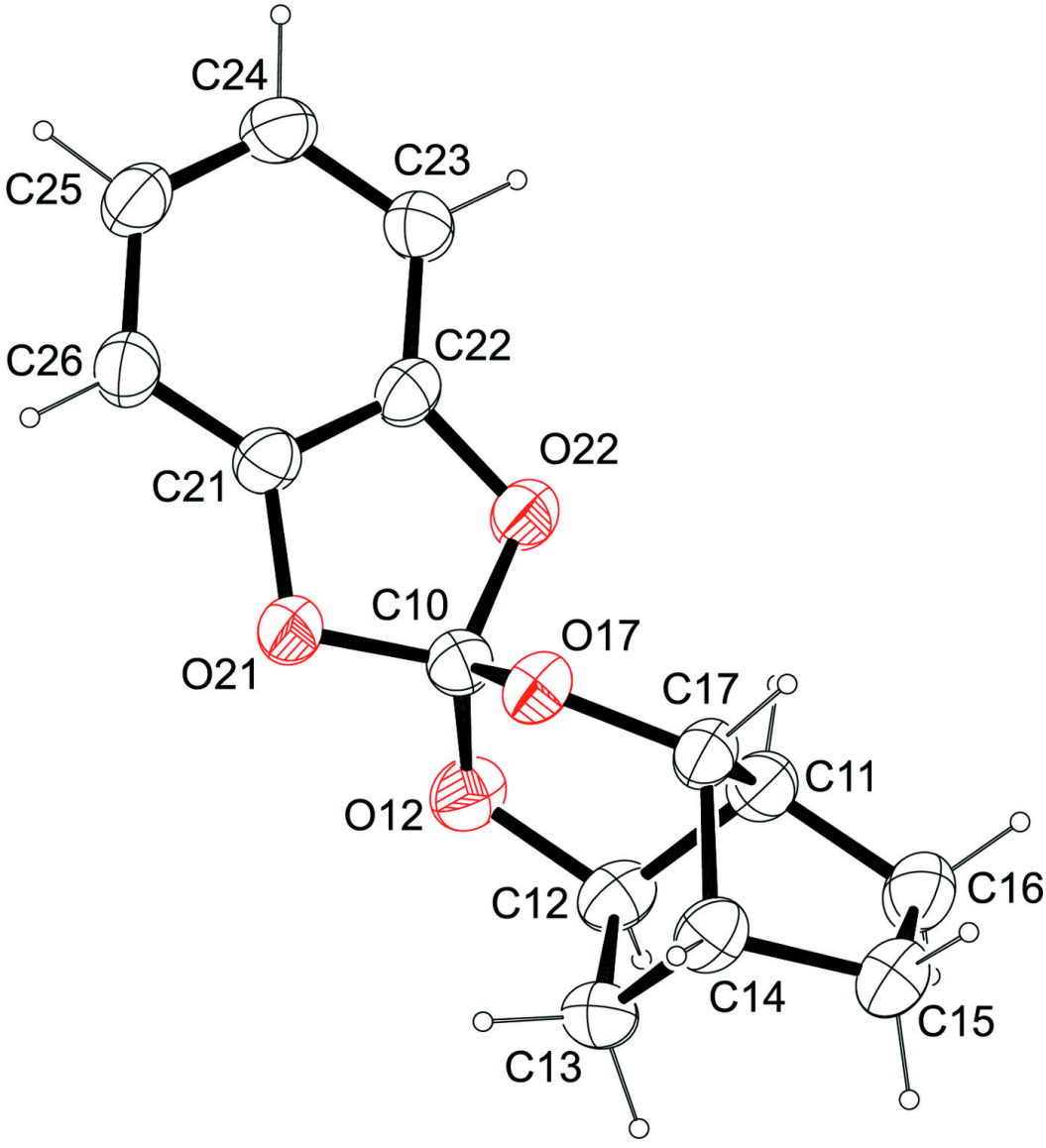
Geometric parameters (Å, °)

O12—C10	1.364 (2)	C14—C15	1.530 (2)
O12—C12	1.4598 (19)	C14—H14	1.0000
O17—C10	1.3685 (19)	C15—C16	1.541 (2)
O17—C17	1.4557 (18)	C15—H151	0.9900
O21—C21	1.3862 (19)	C15—H152	0.9900
O21—C10	1.4129 (19)	C16—H161	0.9900
O22—C22	1.3770 (18)	C16—H162	0.9900
O22—C10	1.4510 (19)	C17—H17	1.0000
C11—C12	1.515 (3)	C21—C22	1.371 (2)
C11—C17	1.532 (2)	C21—C26	1.372 (2)
C11—C16	1.536 (2)	C22—C23	1.368 (2)
C11—H11	1.0000	C23—C24	1.395 (2)
C12—C13	1.544 (2)	C23—H23	0.9500
C12—H12	1.0000	C24—C25	1.380 (3)
C13—C14	1.530 (2)	C24—H24	0.9500
C13—H131	0.9900	C25—C26	1.398 (2)
C13—H132	0.9900	C25—H25	0.9500

C14—C17	1.519 (2)	C26—H26	0.9500
C10—O12—C12	114.27 (12)	C14—C15—H151	111.3
C10—O17—C17	115.45 (12)	C16—C15—H151	111.3
C21—O21—C10	106.68 (12)	C14—C15—H152	111.3
C22—O22—C10	106.13 (12)	C16—C15—H152	111.3
O12—C10—O17	114.77 (13)	H151—C15—H152	109.2
O12—C10—O21	108.50 (13)	C11—C16—C15	104.34 (13)
O17—C10—O21	107.08 (13)	C11—C16—H161	110.9
O12—C10—O22	109.58 (13)	C15—C16—H161	110.9
O17—C10—O22	110.14 (13)	C11—C16—H162	110.9
O21—C10—O22	106.40 (12)	C15—C16—H162	110.9
C12—C11—C17	96.58 (14)	H161—C16—H162	108.9
C12—C11—C16	109.98 (14)	O17—C17—C14	111.23 (13)
C17—C11—C16	102.57 (13)	O17—C17—C11	112.88 (13)
C12—C11—H11	115.2	C14—C17—C11	95.42 (12)
C17—C11—H11	115.2	O17—C17—H17	112.1
C16—C11—H11	115.2	C14—C17—H17	112.1
O12—C12—C11	109.20 (13)	C11—C17—H17	112.1
O12—C12—C13	110.93 (14)	C22—C21—C26	122.57 (15)
C11—C12—C13	103.22 (13)	C22—C21—O21	109.65 (13)
O12—C12—H12	111.1	C26—C21—O21	127.77 (15)
C11—C12—H12	111.1	C23—C22—C21	122.30 (15)
C13—C12—H12	111.1	C23—C22—O22	128.34 (15)
C14—C13—C12	103.05 (13)	C21—C22—O22	109.34 (14)
C14—C13—H131	111.2	C22—C23—C24	116.35 (16)
C12—C13—H131	111.2	C22—C23—H23	121.8
C14—C13—H132	111.2	C24—C23—H23	121.8
C12—C13—H132	111.2	C25—C24—C23	121.26 (15)
H131—C13—H132	109.1	C25—C24—H24	119.4
C17—C14—C13	100.98 (13)	C23—C24—H24	119.4
C17—C14—C15	100.74 (14)	C24—C25—C26	121.81 (16)
C13—C14—C15	108.81 (14)	C24—C25—H25	119.1
C17—C14—H14	114.9	C26—C25—H25	119.1
C13—C14—H14	114.9	C21—C26—C25	115.72 (16)
C15—C14—H14	114.9	C21—C26—H26	122.1
C14—C15—C16	102.17 (13)	C25—C26—H26	122.1
C12—O12—C10—O17	−29.50 (19)	C14—C15—C16—C11	9.50 (18)
C12—O12—C10—O21	−149.20 (12)	C10—O17—C17—C14	−107.09 (15)
C12—O12—C10—O22	95.01 (15)	C10—O17—C17—C11	−1.20 (18)
C17—O17—C10—O12	48.45 (18)	C13—C14—C17—O17	62.57 (16)
C17—O17—C10—O21	168.94 (11)	C15—C14—C17—O17	174.35 (12)
C17—O17—C10—O22	−75.77 (15)	C13—C14—C17—C11	−54.55 (15)
C21—O21—C10—O12	−130.62 (13)	C15—C14—C17—C11	57.24 (14)
C21—O21—C10—O17	104.97 (13)	C12—C11—C17—O17	−54.39 (16)
C21—O21—C10—O22	−12.80 (15)	C16—C11—C17—O17	−166.60 (13)
C22—O22—C10—O12	130.32 (13)	C12—C11—C17—C14	61.39 (14)
C22—O22—C10—O17	−102.52 (14)	C16—C11—C17—C14	−50.83 (15)
C22—O22—C10—O21	13.21 (15)	C10—O21—C21—C22	7.81 (17)

C10—O12—C12—C11	−34.36 (18)	C10—O21—C21—C26	−173.44 (16)
C10—O12—C12—C13	78.75 (16)	C26—C21—C22—C23	0.2 (3)
C17—C11—C12—O12	72.75 (14)	O21—C21—C22—C23	179.04 (14)
C16—C11—C12—O12	178.71 (12)	C26—C21—C22—O22	−178.16 (14)
C17—C11—C12—C13	−45.31 (15)	O21—C21—C22—O22	0.67 (17)
C16—C11—C12—C13	60.65 (17)	C10—O22—C22—C23	173.13 (16)
O12—C12—C13—C14	−104.89 (14)	C10—O22—C22—C21	−8.63 (16)
C11—C12—C13—C14	11.96 (17)	C21—C22—C23—C24	0.0 (2)
C12—C13—C14—C17	26.56 (16)	O22—C22—C23—C24	178.00 (15)
C12—C13—C14—C15	−78.90 (16)	C22—C23—C24—C25	−0.1 (2)
C17—C14—C15—C16	−42.05 (16)	C23—C24—C25—C26	0.1 (3)
C13—C14—C15—C16	63.58 (17)	C22—C21—C26—C25	−0.2 (2)
C12—C11—C16—C15	−75.92 (17)	O21—C21—C26—C25	−178.81 (15)
C17—C11—C16—C15	25.97 (18)	C24—C25—C26—C21	0.0 (2)

Fig. 1


C24
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C12
C14
C15
C13

Fig. 2