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3,9-Bis(2-chlorophenyl)-2,4,8,10-tetraoxaspiro[5.5]undecane

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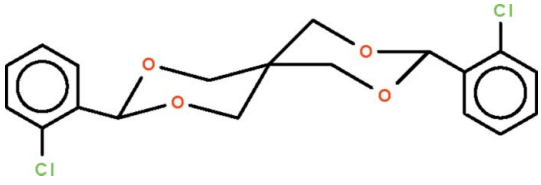
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.032; wR factor = 0.092; data-to-parameter ratio = 16.5.

The complete molecule of the title compound, $C_{19}H_{18}Cl_2O_4$, is generated by a crystallographic twofold axis that passes through the spiro C atom. The 1,3-dioxane ring adopts a chair conformation and the phenyl substituent occupies an equatorial site.

Related literature

For the crystal structure of 3,9-diphenyl-2,4,8,10-tetraoxaspiro[5.5]undecane, see: Wang *et al.* (2006).



Experimental

Crystal data

$C_{19}H_{18}Cl_2O_4$ M_r = 381.23
Monoclinic, $C2/c$
a = 10.7116 (5) Å
b = 9.4693 (5) Å
c = 17.7080 (9) Å
β = 106.745 (1)°
V = 1719.98 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.40 mm^{-1}
T = 173 K
0.46 × 0.42 × 0.22 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.838, T_{max} = 0.917
6932 measured reflections
1883 independent reflections
1707 reflections with $I > 2\sigma(I)$
R_{int} = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.092
S = 1.00
1883 reflections
114 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5174).

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Westrip, S. P. (2010). *publCIF*. In preparation.

supporting information

Acta Cryst. (2010). E**66**, o426 [https://doi.org/10.1107/S1600536810002400]

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S1. Experimental

Pentaerythritol (2 g, 0.014 mol), 2-chlorobenzaldehyde(4.6 g, 0.033 mol), toluene (12 ml) and a catalytic amount (0.2 g) of *p*-toluenesulfonic acid were heated for 4 hours. The mixture was cooled and then filtered. The organic phase was washed with water and 5% sodium bicarbonate (20 ml). The solvent was evaporated and the product recrystallized from ethyl acetate to afford colourless crystals (yield 70%); m.p. 418.5–419 K.

S2. Refinement

H-atoms were placed in calculated positions (C—H 0.95–0.99 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2*U*(C).

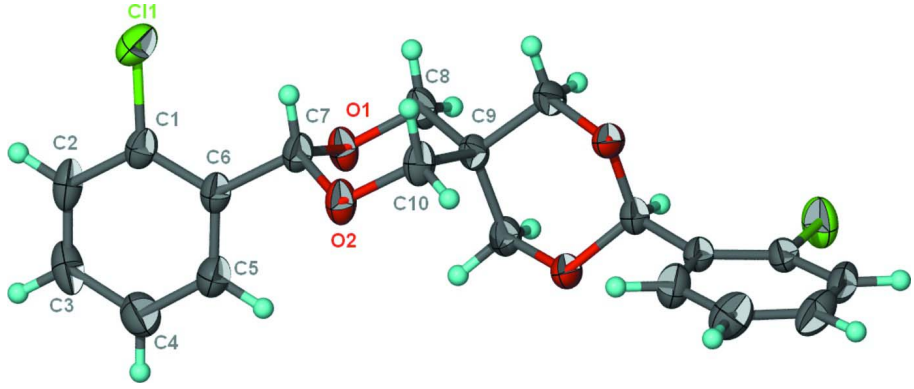

Figure 1

Anisotropic displacement ellipsoid plot of $C_{19}H_{18}Cl_2O_4$ at the 70% probability level; hydrogen atoms are shown as spheres of arbitrary radius.

3,9-Bis(2-chlorophenyl)-2,4,8,10-tetraoxaspiro[5.5]undecane

Crystal data

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M_r = 381.23
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
a = 10.7116 (5) Å
b = 9.4693 (5) Å
c = 17.7080 (9) Å
β = 106.745 (1)°
V = 1719.98 (15) Å^3
Z = 4
F(000) = 792
D_x = 1.472 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 5105 reflections
θ = 2.4–27.1°
μ = 0.40 mm^{-1}
T = 173 K
Block, yellow
0.46 × 0.42 × 0.22 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.838, T_{max} = 0.917
6932 measured reflections
1883 independent reflections
1707 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
θ_{max} = 27.1°, θ_{min} = 2.9°
h = −13→13
k = −12→11
l = −22→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.092
S = 1.00
1883 reflections
114 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0542P)^2 + 1.3419P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
Cl1	−0.05287 (3)	0.53900 (4)	0.14620 (2)	0.04156 (14)
O1	0.30941 (9)	0.51739 (10)	0.13408 (5)	0.0283 (2)
O2	0.31431 (8)	0.34130 (10)	0.22630 (5)	0.0298 (2)
C1	−0.00068 (12)	0.40843 (14)	0.09327 (7)	0.0261 (3)
C2	−0.09394 (12)	0.34711 (16)	0.03040 (8)	0.0329 (3)
H2	−0.1815	0.3799	0.0160	0.040*
C3	−0.05826 (13)	0.23835 (18)	−0.01081 (8)	0.0381 (3)
H3	−0.1214	0.1960	−0.0539	0.046*
C4	0.07000 (15)	0.19038 (18)	0.01047 (9)	0.0398 (3)
H4	0.0941	0.1134	−0.0168	0.048*
C5	0.16221 (13)	0.25578 (16)	0.07170 (8)	0.0342 (3)
H5	0.2501	0.2242	0.0852	0.041*
C6	0.12931 (12)	0.36636 (14)	0.11379 (7)	0.0259 (3)
C7	0.23515 (12)	0.44177 (14)	0.17524 (8)	0.0265 (3)
H7	0.1960	0.5084	0.2059	0.032*
C8	0.40974 (12)	0.59806 (14)	0.18815 (8)	0.0303 (3)
H8A	0.3692	0.6690	0.2148	0.036*
H8B	0.4614	0.6491	0.1586	0.036*
C9	0.5000	0.50311 (19)	0.2500	0.0240 (3)
C10	0.41483 (12)	0.41154 (15)	0.28641 (7)	0.0291 (3)
H10A	0.4699	0.3402	0.3216	0.035*
H10B	0.3743	0.4715	0.3187	0.035*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0294 (2)	0.0446 (2)	0.0492 (2)	0.01101 (14)	0.00891 (16)	−0.00178 (15)
O1	0.0231 (4)	0.0307 (5)	0.0247 (4)	−0.0053 (4)	−0.0034 (4)	0.0058 (4)
O2	0.0220 (4)	0.0323 (5)	0.0290 (5)	−0.0030 (4)	−0.0024 (4)	0.0091 (4)
C1	0.0204 (6)	0.0298 (6)	0.0271 (6)	0.0012 (5)	0.0051 (5)	0.0078 (5)
C2	0.0182 (6)	0.0495 (8)	0.0282 (6)	−0.0041 (5)	0.0022 (5)	0.0102 (6)
C3	0.0283 (7)	0.0560 (9)	0.0275 (6)	−0.0157 (6)	0.0042 (5)	−0.0035 (6)
C4	0.0353 (7)	0.0459 (9)	0.0389 (8)	−0.0068 (6)	0.0120 (6)	−0.0098 (6)
C5	0.0223 (6)	0.0391 (7)	0.0390 (7)	0.0011 (5)	0.0055 (5)	−0.0028 (6)
C6	0.0194 (6)	0.0293 (6)	0.0262 (6)	−0.0012 (5)	0.0024 (5)	0.0052 (5)
C7	0.0191 (6)	0.0295 (6)	0.0278 (6)	0.0013 (5)	0.0016 (5)	0.0039 (5)
C8	0.0258 (6)	0.0259 (6)	0.0304 (6)	−0.0036 (5)	−0.0057 (5)	0.0046 (5)
C9	0.0208 (8)	0.0257 (8)	0.0214 (8)	0.000	−0.0004 (6)	0.000
C10	0.0224 (6)	0.0376 (7)	0.0232 (6)	−0.0006 (5)	0.0000 (5)	0.0051 (5)

Geometric parameters (Å, º)

Cl1—C1	1.7384 (14)	C5—C6	1.388 (2)
O1—C7	1.4181 (15)	C5—H5	0.9500
O1—C8	1.4361 (15)	C6—C7	1.5062 (17)
O2—C7	1.4141 (15)	C7—H7	1.0000
O2—C10	1.4402 (15)	C8—C9	1.5272 (16)
C1—C2	1.3906 (18)	C8—H8A	0.9900
C1—C6	1.3921 (17)	C8—H8B	0.9900
C2—C3	1.378 (2)	C9—C8^{i}	1.5272 (16)
C2—H2	0.9500	C9—C10	1.5293 (16)
C3—C4	1.392 (2)	C9—C10^{i}	1.5293 (16)
C3—H3	0.9500	C10—H10A	0.9900
C4—C5	1.385 (2)	C10—H10B	0.9900
C4—H4	0.9500		
C7—O1—C8	110.41 (9)	O1—C7—C6	106.55 (10)
C7—O2—C10	110.16 (10)	O2—C7—H7	110.2
C2—C1—C6	121.55 (13)	O1—C7—H7	110.2
C2—C1—Cl1	117.40 (10)	C6—C7—H7	110.2
C6—C1—Cl1	121.04 (10)	O1—C8—C9	111.25 (10)
C3—C2—C1	119.41 (12)	O1—C8—H8A	109.4
C3—C2—H2	120.3	C9—C8—H8A	109.4
C1—C2—H2	120.3	O1—C8—H8B	109.4
C2—C3—C4	120.21 (13)	C9—C8—H8B	109.4
C2—C3—H3	119.9	H8A—C8—H8B	108.0
C4—C3—H3	119.9	C8^{i}—C9—C8	107.86 (14)
C5—C4—C3	119.45 (14)	C8^{i}—C9—C10	111.27 (7)
C5—C4—H4	120.3	C8—C9—C10	107.75 (7)
C3—C4—H4	120.3	C8^{i}—C9—C10^{i}	107.75 (7)
C6—C5—C4	121.57 (12)	C8—C9—C10^{i}	111.27 (7)

C6—C5—H5	119.2	C10—C9—C10^{i}	110.92 (16)
C4—C5—H5	119.2	O2—C10—C9	111.10 (9)
C5—C6—C1	117.72 (12)	O2—C10—H10A	109.4
C5—C6—C7	119.39 (11)	C9—C10—H10A	109.4
C1—C6—C7	122.75 (12)	O2—C10—H10B	109.4
O2—C7—O1	110.28 (9)	C9—C10—H10B	109.4
O2—C7—C6	109.32 (10)	H10A—C10—H10B	108.0

C6—C1—C2—C3	2.55 (19)	C8—O1—C7—C6	177.43 (10)
Cl1—C1—C2—C3	−176.83 (11)	C5—C6—C7—O2	−50.34 (15)
C1—C2—C3—C4	0.1 (2)	C1—C6—C7—O2	133.95 (12)
C2—C3—C4—C5	−2.1 (2)	C5—C6—C7—O1	68.83 (15)
C3—C4—C5—C6	1.5 (2)	C1—C6—C7—O1	−106.88 (13)
C4—C5—C6—C1	1.0 (2)	C7—O1—C8—C9	58.27 (13)
C4—C5—C6—C7	−174.90 (13)	O1—C8—C9—C8^{i}	−171.63 (13)
C2—C1—C6—C5	−3.07 (19)	O1—C8—C9—C10	−51.41 (14)
Cl1—C1—C6—C5	176.29 (10)	O1—C8—C9—C10^{i}	70.39 (14)
C2—C1—C6—C7	172.71 (12)	C7—O2—C10—C9	−58.74 (14)
Cl1—C1—C6—C7	−7.93 (17)	C8^{i}—C9—C10—O2	169.66 (10)
C10—O2—C7—O1	64.19 (13)	C8—C9—C10—O2	51.61 (14)
C10—O2—C7—C6	−178.97 (10)	C10^{i}—C9—C10—O2	−70.41 (9)
C8—O1—C7—O2	−64.03 (13)		

Symmetry code: (i) $-x+1, y, -z+1/2$.