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3,3′-Bis(chloromethyl)-4,4′-diethoxy-1,1′-biphenyl

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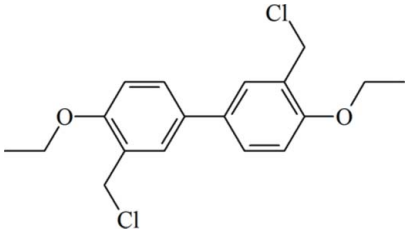
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.048; wR factor = 0.149; data-to-parameter ratio = 20.1.

The asymmetric unit of the title compound, $C_{18}H_{20}Cl_2O_2$, consists of a half-molecule, the other half being generated by an inversion center, located at the mid-point of the benzene–benzene bond. Except for the two Cl atoms, all other atoms of the compound are nearly coplanar, with the atomic displacements from the molecular mean plane ranging from 0.0037 (19) to 0.071 (2) Å. The two Cl atoms are in *trans* positions and are displaced with respect to the mean plane by 1.687 (2) and −1.693 (3) Å. The crystal packing is governed by van der Waals interactions.

Related literature

For general background and synthesis, see: Trad *et al.* (2006); Hrichi *et al.* (2013). For related structures, see: Huang *et al.* (2011); Trad *et al.* (2012).



Experimental

Crystal data

$C_{18}H_{20}Cl_2O_2$
M_r = 339.24
Monoclinic, $P2_1/n$
a = 4.984 (2) Å
b = 11.598 (5) Å
c = 14.578 (8) Å
β = 98.387 (2)°
V = 833.7 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.39 mm^{-1}
T = 298 K
0.20 × 0.14 × 0.10 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.937, T_{max} = 0.953
8808 measured reflections
2010 independent reflections
1252 reflections with $I > 2\sigma(I)$
R_{int} = 0.048

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.149
S = 1.05
2010 reflections
100 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Data collection: *COLLECT* (Nonius, 2002); cell refinement: *DENZO/SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO/SCALEPACK*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXL97*.

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Supporting information for this paper is available from the IUCr electronic archives (Reference: DS2239).

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supporting information

Acta Cryst. (2014). E**70**, o387 [doi:10.1107/S1600536814004528]

3,3′-Bis(chloromethyl)-4,4′-diethoxy-1,1′-biphenyl

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S1. Comment

The synthesis of the compound 3,3′-Bis(chloromethyl)-4,4′-diethoxy-1,1′-biphenyl (BipEt2Cl2) is a part of an ongoing program on the investigation of new π-conjugated electroluminescent polymers derived from bisphenols (Trad *et al.*, 2006; Hrichi *et al.*, 2013).

The asymmetric unit of the title compound contains one-half of the molecule (Fig. 1) with the other half generated by an inversion center lieing between the two phenyl groups, at the the mid-point of the carbon-carbon bond. All atoms of the compound (BipEt2Cl2) lie in the same plane, the largest deviation being 0.0709 (22) Å for atom C9, except the two chlorine atoms. A π-conjugated system accounts for the planarity of the molecule and probably prevents the free rotation around the central carbon-carbon bond between the phenyl groups. The planes containing respectively the chloromethyl group and the biphenyl group are nearly orthogonal to each other, with a dihedral angle equal to 88.98 (9)°, such a value is nearly close to that observed for the 1-benzyloxy-2,5-bis(chloromethyl)-4-methoxybenzene (Trad *et al.*, 2012). The values of bond lengths and angles agree with those reported for similar compounds (Huang *et al.* 2011; Trad *et al.* 2012). A projection of the crystal structure of the compound, on the (010) plane, is given the by the figure 2.

S2. Experimental

The compound 3,3′-Bis(chloromethyl)-4,4′-diethoxy-1,1′-biphenyl (BipEt2Cl2) was synthesized in two steps: a mixture of 4,4′-dihydroxy-1,1′-biphenyl (10 mmol), bromoethane (30 mmol) and anhydrous potassium carbonate (60 mmol) was added to 10 ml of DMF and was stirred for 24 h at room temperature. The reaction mixture was poured into distilled water and the intermediate product, 4,4′-diethoxy-1,1′-biphenyl (BipEt2), was extracted with diethyl ether, purified by recrystallization from ethanol/acetone (4/1) and then obtained as white fine powder. Yield: 80%; mp: 450–452 K. In a second step, a suspension of (BipEt2) (10 mmol) and paraformaldehyde (80 mmol), in a mixture of glacial acetic acid (30 ml) and 37% hydrochloric acid (8 ml), was left to stir for approximately 5 h at 328 K. After cooling, the resulting mixture was then poured into distilled water. The product (BipEt2Cl2) was extracted with diethylether and recrystallized from ethanol as colorless needle-like crystals. Yield: 65%; mp: 388 (2) K.

S3. Refinement

All H atoms were refined using a riding model with C—H = 0.96 (CH3), 0.97 (CH2), 0.93 (CArH) Å and U_{iso}(H) = 1.5 U_{eq}(C), 1.2 U_{eq}(C) and 1.2 U_{eq}(C) respectively.

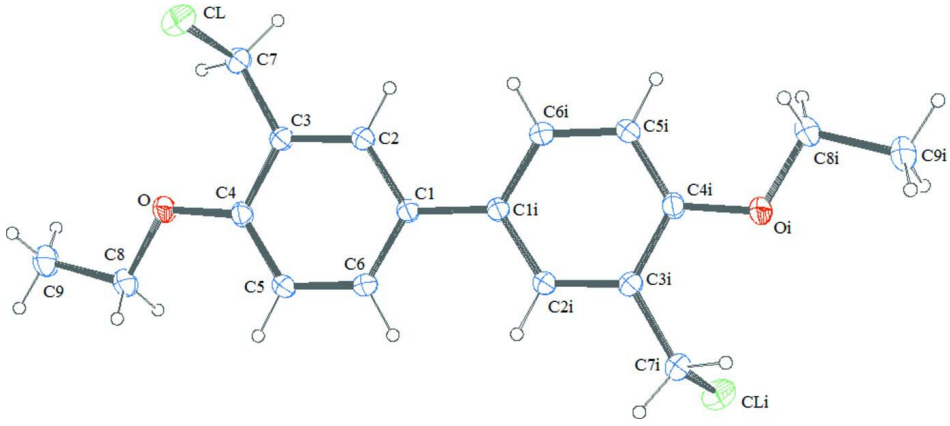

Figure 1

The molecular structure of the title compound BipEt2Cl2 with displacement ellipsoids drawn at 20% probability level for non hydrogen atoms.

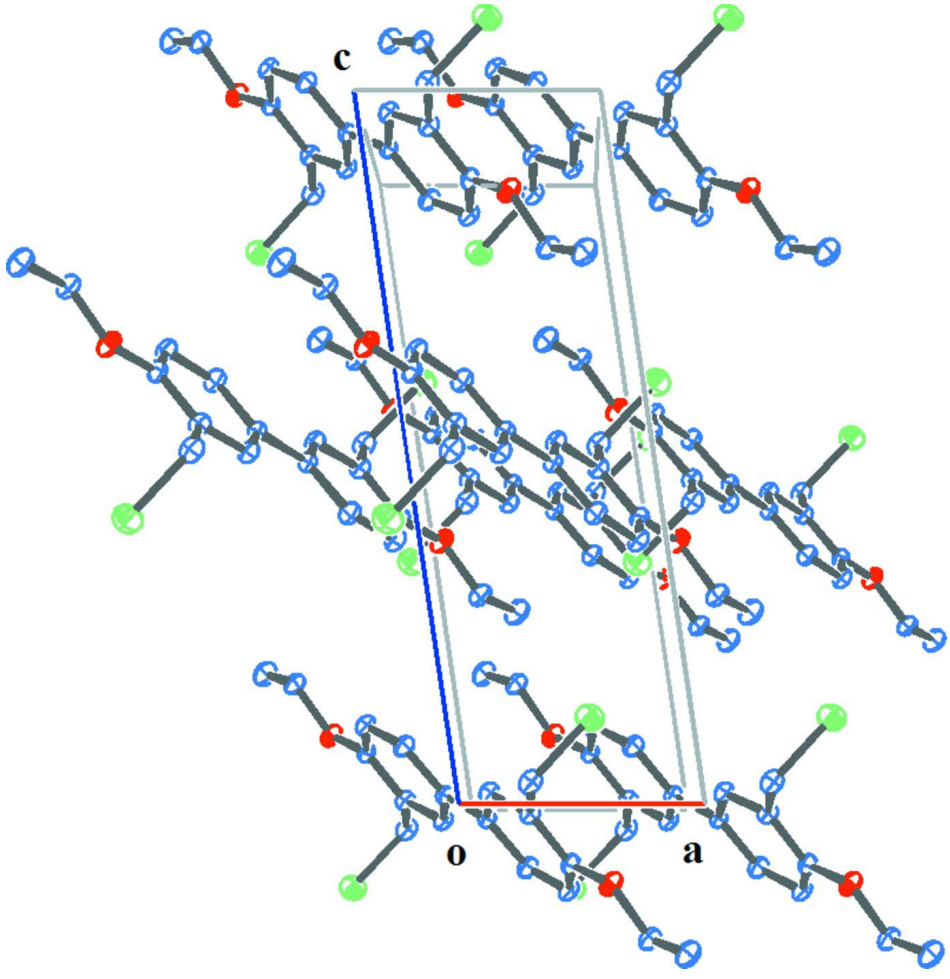

Figure 2

The crystal packing of compound BipEt2Cl2 viewed along *b* axis.

3,3′-Bis(chloromethyl)-4,4′-diethoxy-1,1′-biphenyl

Crystal data

$C_{18}H_{20}Cl_2O_2$
M_r = 339.24
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 4.984 (2) Å
b = 11.598 (5) Å
c = 14.578 (8) Å
β = 98.387 (2)°
V = 833.7 (7) Å^3
Z = 2
$F(000)$ = 356
D_x = 1.351 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8808 reflections
θ = 2.3–29.0°
μ = 0.39 mm^{-1}
T = 298 K
Needle, colourless
0.20 × 0.14 × 0.10 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi/w scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.937, T_{max} = 0.953
8808 measured reflections
2010 independent reflections
1252 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 28.1°, θ_{min} = 2.3°
h = −6→6
k = 0→15
l = 0→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.149
S = 1.05
2010 reflections
100 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
$w = 1/[\sigma^2(F_o^2) + (0.077P)^2 + 0.059P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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}
Cl	0.58814 (14)	0.13757 (6)	0.12470 (5)	0.0713 (3)
O	0.5754 (3)	0.20148 (12)	−0.11362 (11)	0.0483 (4)
C1	0.0870 (3)	0.45550 (16)	−0.01787 (13)	0.0345 (4)
C2	0.1168 (4)	0.34506 (17)	0.01950 (15)	0.0391 (5)

H2	0.0217	0.3264	0.0678	0.047*
C3	0.2804 (4)	0.26160 (17)	−0.01136 (13)	0.0385 (5)
C4	0.4206 (4)	0.28839 (17)	−0.08584 (14)	0.0388 (5)
C5	0.3944 (4)	0.39671 (18)	−0.12463 (14)	0.0425 (5)
H5	0.4869	0.4150	−0.1737	0.051*
C6	0.2315 (4)	0.47848 (18)	−0.09126 (14)	0.0424 (5)
H6	0.2173	0.5512	−0.1185	0.051*
C7	0.3035 (4)	0.14484 (18)	0.03221 (16)	0.0493 (6)
H7A	0.3263	0.0876	−0.0145	0.059*
H7B	0.1377	0.1272	0.0568	0.059*
C8	0.7054 (4)	0.2217 (2)	−0.19346 (16)	0.0500 (6)
H8A	0.8346	0.2844	−0.1816	0.060*
H8B	0.5720	0.2422	−0.2462	0.060*
C9	0.8482 (5)	0.1126 (2)	−0.21296 (19)	0.0618 (7)
H9A	0.9380	0.1234	−0.2663	0.093*
H9B	0.7183	0.0513	−0.2248	0.093*
H9C	0.9795	0.0932	−0.1603	0.093*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.0797 (5)	0.0631 (5)	0.0694 (5)	0.0113 (3)	0.0046 (3)	0.0182 (3)
O	0.0565 (9)	0.0371 (9)	0.0568 (9)	0.0086 (6)	0.0266 (7)	−0.0025 (7)
C1	0.0339 (10)	0.0325 (11)	0.0380 (10)	0.0000 (7)	0.0090 (7)	−0.0006 (8)
C2	0.0423 (10)	0.0346 (12)	0.0433 (11)	0.0010 (8)	0.0156 (8)	0.0015 (9)
C3	0.0444 (11)	0.0293 (11)	0.0435 (11)	−0.0001 (8)	0.0115 (9)	−0.0002 (9)
C4	0.0364 (10)	0.0359 (12)	0.0459 (12)	0.0019 (7)	0.0122 (8)	−0.0045 (9)
C5	0.0466 (12)	0.0398 (12)	0.0454 (12)	0.0020 (8)	0.0206 (9)	0.0042 (9)
C6	0.0485 (11)	0.0327 (11)	0.0490 (12)	0.0026 (8)	0.0169 (9)	0.0071 (9)
C7	0.0557 (13)	0.0360 (12)	0.0596 (15)	0.0039 (9)	0.0193 (11)	0.0031 (10)
C8	0.0524 (13)	0.0511 (14)	0.0503 (13)	0.0068 (10)	0.0206 (10)	−0.0039 (10)
C9	0.0685 (16)	0.0540 (16)	0.0679 (16)	0.0123 (11)	0.0267 (13)	−0.0088 (12)

Geometric parameters (Å, º)

Cl—C7	1.811 (2)	C2—C3	1.382 (3)
O—C4	1.366 (2)	C3—C4	1.409 (3)
O—C8	1.432 (3)	C3—C7	1.493 (3)
C1—C2	1.391 (3)	C4—C5	1.376 (3)
C1—C6	1.399 (3)	C5—C6	1.382 (3)
C1—C1^{i}	1.490 (3)	C8—C9	1.499 (3)
C4—O—C8	117.53 (16)	C3—C7—Cl	111.25 (15)
C2—C1—C6	115.93 (17)	C3—C7—H7A	109.4
C2—C1—C1^{i}	122.3 (2)	Cl—C7—H7A	109.4
C6—C1—C1^{i}	121.7 (2)	C3—C7—H7B	109.4
C3—C2—C1	123.47 (18)	Cl—C7—H7B	109.4
C3—C2—H2	118.3	H7A—C7—H7B	108.0

C1—C2—H2	118.3	O—C8—C9	107.42 (19)
C2—C3—C4	118.59 (18)	O—C8—H8A	110.2
C2—C3—C7	120.61 (18)	C9—C8—H8A	110.2
C4—C3—C7	120.78 (17)	O—C8—H8B	110.2
O—C4—C5	125.14 (18)	C9—C8—H8B	110.2
O—C4—C3	115.51 (17)	H8A—C8—H8B	108.5
C5—C4—C3	119.35 (17)	C8—C9—H9A	109.5
C4—C5—C6	120.47 (18)	C8—C9—H9B	109.5
C4—C5—H5	119.8	H9A—C9—H9B	109.5
C6—C5—H5	119.8	C8—C9—H9C	109.5
C5—C6—C1	122.17 (19)	H9A—C9—H9C	109.5
C5—C6—H6	118.9	H9B—C9—H9C	109.5
C1—C6—H6	118.9		

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