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1-Benzyloxy-2,5-bis(chloromethyl)-4-methoxybenzene

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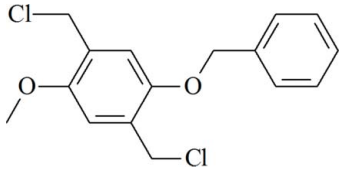
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.049; wR factor = 0.166; data-to-parameter ratio = 18.5.

In the title compound, $C_{16}H_{16}Cl_2O_2$, the dihedral angle between the two rings is 52.65 (10)°. The two Cl atoms are *trans* to one another being displaced by 1.644 (5) and −1.664 (4) Å from the plane of the benzene ring. Except for the two Cl atoms and the C atoms of the ring of the benzyloxy group, all the other atoms of the compound lie in the same plane [maximum deviation = 0.056 (3) Å]. In the crystal, no significant intermolecular interactions are observed.

Related literature

For general background, physical properties and synthesis of poly(*p*-phenylenevinylene) derivatives (PPVs), see: Trad *et al.* (2006). For related structures, see: Huang *et al.* (2011); Watanabe *et al.* (2005).



Experimental

Crystal data

$C_{16}H_{16}Cl_2O_2$
M_r = 311.19
Monoclinic, $P2_1/c$
a = 10.9026 (4) Å
b = 17.8127 (6) Å
c = 8.4221 (2) Å
β = 109.561 (4)°
V = 1541.21 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.42 mm^{-1}
T = 298 K
0.40 × 0.30 × 0.20 mm

Data collection

Enraf–Nonius κ-geometry TurboCAD-4 diffractometer
4141 measured reflections
3363 independent reflections
1562 reflections with $I > 2\sigma(I)$
R_{int} = 0.099
1 standard reflections every 60 min
intensity decay: 3%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.166
S = 1.00
3363 reflections
182 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); 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: NG5278).

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supporting information

Acta Cryst. (2012). E**68**, o2339 [https://doi.org/10.1107/S1600536812029558]

1-Benzyloxy-2,5-bis(chloromethyl)-4-methoxybenzene

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S1. Comment

The 1-benzyloxy-2,5-bis(chloromethyl)-4-methoxybenzene (MBzCl) was used as a monomer for the synthesis of π-conjugated polymers such as poly(*p*-phenylenevinylene) derivatives (PPVs) which have potential application as electroluminescent materials. The monomer (MBzCl) has been synthesized as described in literature (Trad *et al.*, 2006).

The asymmetric unit of the title compound (MBzCl) contains one molecule which is presented in Fig.1. The dihedral angle between the two phenyl rings is 52.65 (10)°. The two planes containing respectively the two chloromethyl groups and the substituted benzene ring are nearly orthogonal to each other, with a dihedral angle equal to 87.69 (9)°. The two chlorine atoms are in *trans* position with respect to the benzene substituted group. All atoms of the compound (MBzCl) lie in the same plane, the largest deviation being 0.0563 (28) Å for atom C9, except the two chlorine atoms and the carbons of the phenyl of the benzyloxy group. Some selected bond lengths are given in table 2 and agree with those reported for similar compounds (Huang *et al.*, 2011; Watanabe *et al.*, 2005). A strong intramolecular hydrogen bond C7—H7A···O1 is observed (table 1). In the crystal structure, weak intermolecular C—H···Cl hydrogen bonds link molecules of (MBzCl) into chains which propagate along [010] as shown in Fig. 2.

S2. Experimental

The compound 1-benzyloxy-2,5-bis(chloromethyl)-4-methoxybenzene (MBzCl) was prepared in two steps: a mixture of 4-methoxyphenol (10 mmol), benzylchloride (15 mmol) and K_2CO_3 (20 mmol) was added to 10 ml of DMF and was heated under stirring at 353 K for 24 h. The product, 1-benzyloxy-4-methoxybenzene (MBz), was purified by recrystallization from ethanol and was obtained as white powder.Yield: 95%; mp 346 (2) K. In a next step, a suspension of (MBz) (10 mmol) and paraformaldehyde (50 mmol), in a mixture of glacial acetic acid (20 ml) and 37% hydrochloric acid (10 ml), was left to stir for approximately 20 h at room temperature. The resulting mixture was then poured into distilled water. The product (MBzCl) was extracted with dichloromethane and recrystallized from ethanol as colorless needle-like white crystals. Yield: 40%; mp: 388 (2) K.

S3. Refinement

All H atoms were refined using a riding model with C—H = 0.96 (CH_3), 0.97 (CH_2), 0.93 ($C_{Ar}H$) Å and U_{iso}(H) = 1.5 U_{eq}(C), 1.2 U_{eq}(C) and 1.2 U_{eq}(C) respectively.

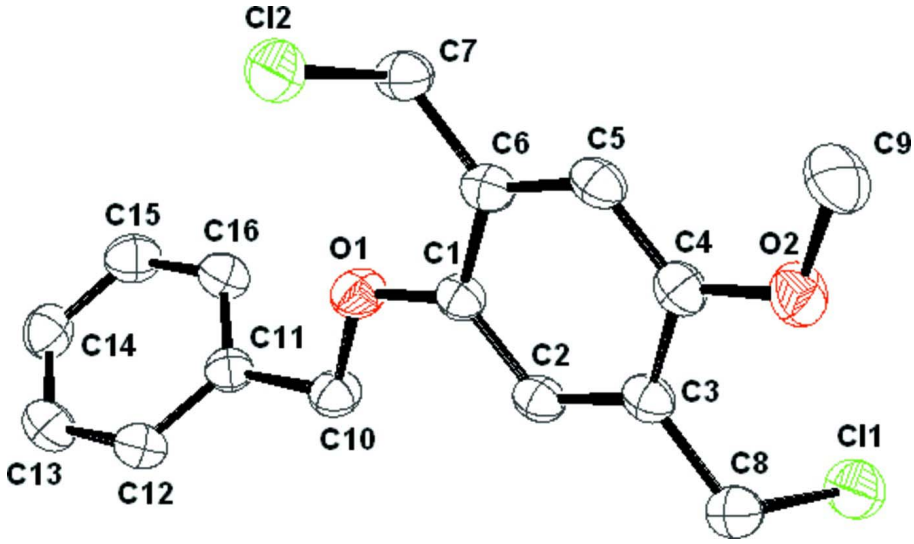

Figure 1

The molecular structure of the title compoud MBzCl with displacement ellipsoids drawn at the 30% probability.

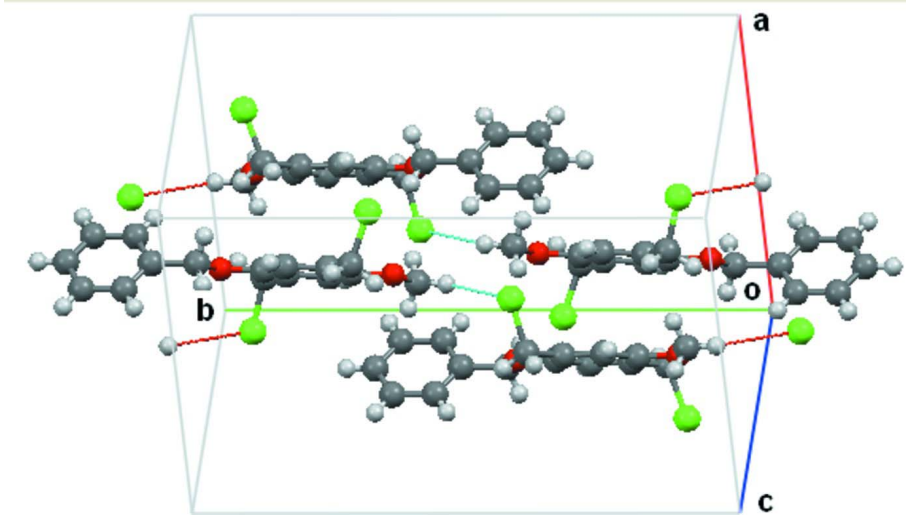

Figure 2

The lattice framework of the title compound MBzCl, showing molecules linked through weak intermolecular C—H···Cl hydrogen bonds to form chains which propagate along [010].

1-Benzyloxy-2,5-bis(chloromethyl)-4-methoxybenzene

Crystal data

$C_{16}H_{16}Cl_2O_2$
$M_r = 311.19$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 10.9026$ (4) Å
$b = 17.8127$ (6) Å
$c = 8.4221$ (2) Å
$\beta = 109.561$ (4)°
$V = 1541.21$ (9) Å^3
$Z = 4$
$F(000) = 648$
$D_x = 1.341$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4284 reflections
$\theta = 2.0$–27.0°
$\mu = 0.42$ mm^{-1}

T = 298 K
Needle-shaped, colourless

0.40 × 0.30 × 0.20 mm

Data collection

Enraf–Nonius κ-geometry TurboCAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
non–profiled $\omega/2\theta$ scans
4141 measured reflections
3363 independent reflections
1562 reflections with $I > 2\sigma(I)$

R_{int} = 0.099
θ_{max} = 27.0°, θ_{min} = 2.0°
h = −13→1
k = −22→0
l = −10→10
1 standard reflections every 60 min
intensity decay: 3%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.166
S = 1.00
3363 reflections
182 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
$w = 1/[\sigma^2(F_o^2) + (0.0682P)^2 + 0.2353P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 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}
Cl1	0.01947 (9)	0.37837 (6)	0.06080 (12)	0.0810 (3)
Cl2	0.62734 (9)	0.11217 (6)	0.35509 (13)	0.0854 (4)
O1	0.2844 (2)	0.08686 (11)	0.1620 (3)	0.0591 (6)
O2	0.3519 (2)	0.39330 (11)	0.2330 (3)	0.0676 (7)
C1	0.2962 (3)	0.16329 (16)	0.1757 (4)	0.0456 (7)
C2	0.2106 (3)	0.21379 (16)	0.0689 (4)	0.0488 (7)
H2	0.1388	0.1960	−0.0179	0.059*
C3	0.2311 (3)	0.29076 (16)	0.0903 (4)	0.0479 (7)
C4	0.3386 (3)	0.31709 (16)	0.2217 (4)	0.0510 (7)
C5	0.4231 (3)	0.26696 (17)	0.3285 (4)	0.0521 (7)
H5	0.4947	0.2849	0.4155	0.062*
C6	0.4029 (3)	0.18995 (16)	0.3084 (4)	0.0485 (7)
C7	0.4947 (3)	0.13664 (18)	0.4269 (4)	0.0596 (8)
H7A	0.4481	0.0915	0.4368	0.071*

H7B	0.5289	0.1595	0.5376	0.071*
C8	0.1388 (3)	0.34438 (18)	−0.0267 (4)	0.0590 (8)
H8A	0.0950	0.3195	−0.1330	0.071*
H8B	0.1872	0.3865	−0.0487	0.071*
C9	0.4639 (4)	0.4225 (2)	0.3585 (5)	0.0789 (11)
H9A	0.4628	0.4083	0.4679	0.118*
H9B	0.4640	0.4763	0.3503	0.118*
H9C	0.5408	0.4028	0.3421	0.118*
C10	0.1737 (3)	0.05788 (17)	0.0327 (4)	0.0555 (8)
H10A	0.1737	0.0752	−0.0765	0.067*
H10B	0.0946	0.0755	0.0491	0.067*
C11	0.1787 (3)	−0.02635 (16)	0.0394 (4)	0.0480 (7)
C12	0.1706 (3)	−0.06681 (18)	−0.1013 (4)	0.0599 (8)
H12	0.1642	−0.0419	−0.2009	0.072*
C13	0.1719 (4)	−0.14467 (19)	−0.0975 (5)	0.0704 (10)
H13	0.1663	−0.1717	−0.1941	0.084*
C14	0.1815 (3)	−0.18163 (19)	0.0491 (5)	0.0683 (9)
H14	0.1828	−0.2338	0.0522	0.082*
C15	0.1892 (3)	−0.14167 (19)	0.1900 (5)	0.0678 (9)
H15	0.1955	−0.1665	0.2896	0.081*
C16	0.1875 (3)	−0.06377 (17)	0.1849 (4)	0.0596 (8)
H16	0.1924	−0.0367	0.2813	0.071*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0677 (6)	0.0993 (7)	0.0726 (6)	0.0214 (5)	0.0192 (5)	−0.0109 (5)
Cl2	0.0632 (6)	0.1105 (8)	0.0773 (7)	0.0212 (5)	0.0167 (5)	−0.0182 (5)
O1	0.0492 (12)	0.0531 (12)	0.0632 (14)	−0.0077 (10)	0.0031 (10)	−0.0008 (10)
O2	0.0667 (15)	0.0520 (13)	0.0768 (17)	−0.0099 (11)	0.0145 (12)	−0.0128 (11)
C1	0.0400 (15)	0.0517 (17)	0.0442 (15)	−0.0052 (12)	0.0131 (12)	−0.0040 (13)
C2	0.0415 (16)	0.0598 (19)	0.0440 (16)	−0.0063 (13)	0.0128 (13)	−0.0056 (14)
C3	0.0436 (15)	0.0592 (18)	0.0431 (16)	−0.0039 (14)	0.0174 (13)	−0.0044 (13)
C4	0.0501 (17)	0.0543 (18)	0.0517 (17)	−0.0056 (14)	0.0212 (14)	−0.0068 (14)
C5	0.0444 (16)	0.0630 (19)	0.0467 (16)	−0.0106 (14)	0.0124 (13)	−0.0118 (14)
C6	0.0389 (14)	0.0627 (18)	0.0444 (15)	−0.0050 (13)	0.0147 (12)	−0.0015 (14)
C7	0.0509 (18)	0.072 (2)	0.0514 (18)	0.0006 (15)	0.0107 (15)	0.0008 (15)
C8	0.0568 (19)	0.065 (2)	0.0530 (19)	0.0010 (16)	0.0149 (15)	0.0020 (16)
C9	0.089 (3)	0.059 (2)	0.080 (3)	−0.0217 (19)	0.016 (2)	−0.0175 (18)
C10	0.0468 (16)	0.0556 (19)	0.0553 (19)	−0.0050 (14)	0.0054 (14)	−0.0018 (14)
C11	0.0381 (14)	0.0526 (17)	0.0493 (17)	−0.0051 (13)	0.0091 (12)	−0.0032 (14)
C12	0.063 (2)	0.066 (2)	0.0474 (19)	−0.0032 (16)	0.0135 (15)	−0.0014 (15)
C13	0.077 (2)	0.069 (2)	0.061 (2)	−0.0076 (19)	0.0188 (19)	−0.0162 (18)
C14	0.068 (2)	0.0512 (19)	0.082 (3)	−0.0034 (16)	0.0195 (19)	−0.0040 (19)
C15	0.070 (2)	0.068 (2)	0.062 (2)	−0.0075 (17)	0.0186 (17)	0.0099 (17)
C16	0.064 (2)	0.062 (2)	0.0509 (19)	−0.0076 (16)	0.0163 (15)	−0.0045 (15)

Geometric parameters (Å, º)

Cl1—C8	1.801 (3)	C8—H8B	0.9700
Cl2—C7	1.798 (3)	C9—H9A	0.9600
O1—C1	1.369 (3)	C9—H9B	0.9600
O1—C10	1.424 (3)	C9—H9C	0.9600
O2—C4	1.365 (3)	C10—C11	1.502 (4)
O2—C9	1.420 (4)	C10—H10A	0.9700
C1—C2	1.388 (4)	C10—H10B	0.9700
C1—C6	1.399 (4)	C11—C12	1.364 (4)
C2—C3	1.391 (4)	C11—C16	1.370 (4)
C2—H2	0.9300	C12—C13	1.387 (4)
C3—C4	1.397 (4)	C12—H12	0.9300
C3—C8	1.494 (4)	C13—C14	1.372 (5)
C4—C5	1.378 (4)	C13—H13	0.9300
C5—C6	1.391 (4)	C14—C15	1.363 (5)
C5—H5	0.9300	C14—H14	0.9300
C6—C7	1.493 (4)	C15—C16	1.388 (4)
C7—H7A	0.9700	C15—H15	0.9300
C7—H7B	0.9700	C16—H16	0.9300
C8—H8A	0.9700		

C1—O1—C10	117.2 (2)	H8A—C8—H8B	108.0
C4—O2—C9	117.4 (3)	O2—C9—H9A	109.5
O1—C1—C2	124.5 (2)	O2—C9—H9B	109.5
O1—C1—C6	115.8 (3)	H9A—C9—H9B	109.5
C2—C1—C6	119.8 (3)	O2—C9—H9C	109.5
C1—C2—C3	120.7 (3)	H9A—C9—H9C	109.5
C1—C2—H2	119.6	H9B—C9—H9C	109.5
C3—C2—H2	119.6	O1—C10—C11	108.8 (2)
C2—C3—C4	119.3 (3)	O1—C10—H10A	109.9
C2—C3—C8	120.1 (3)	C11—C10—H10A	109.9
C4—C3—C8	120.6 (3)	O1—C10—H10B	109.9
O2—C4—C5	124.5 (3)	C11—C10—H10B	109.9
O2—C4—C3	115.5 (3)	H10A—C10—H10B	108.3
C5—C4—C3	120.0 (3)	C12—C11—C16	119.0 (3)
C4—C5—C6	121.1 (3)	C12—C11—C10	120.3 (3)
C4—C5—H5	119.5	C16—C11—C10	120.7 (3)
C6—C5—H5	119.5	C11—C12—C13	120.7 (3)
C5—C6—C1	119.2 (3)	C11—C12—H12	119.7
C5—C6—C7	120.2 (3)	C13—C12—H12	119.7
C1—C6—C7	120.7 (3)	C14—C13—C12	119.9 (3)
C6—C7—Cl2	111.3 (2)	C14—C13—H13	120.1
C6—C7—H7A	109.4	C12—C13—H13	120.1
Cl2—C7—H7A	109.4	C15—C14—C13	119.8 (3)
C6—C7—H7B	109.4	C15—C14—H14	120.1
Cl2—C7—H7B	109.4	C13—C14—H14	120.1
H7A—C7—H7B	108.0	C14—C15—C16	119.9 (3)

C3—C8—Cl1	111.4 (2)	C14—C15—H15	120.0
C3—C8—H8A	109.4	C16—C15—H15	120.0
Cl1—C8—H8A	109.4	C11—C16—C15	120.7 (3)
C3—C8—H8B	109.4	C11—C16—H16	119.7
Cl1—C8—H8B	109.4	C15—C16—H16	119.7

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7*A*···O1	0.97	2.40	2.756 (4)	101