Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,2-Bis(3-chloromethyl-4-ethoxyphenyl)-propane

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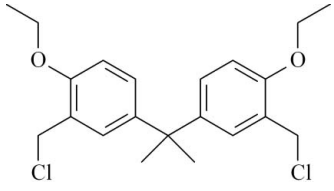
Received 11 June 2008; accepted 20 June 2008

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.036; wR factor = 0.118; data-to-parameter ratio = 11.8.

The title compound, $C_{21}H_{26}Cl_2O_2$, a bis-chloromethyl derivative of *O*-ethylated bisphenol A, exhibits C_2 molecular symmetry. It shows a bent conformation with the two benzene rings nearly perpendicular [dihedral angle = 87.17 (6)°].

Related literature

For more information on the synthesis, see: Miyazawa *et al.* (1999). For background to the investigation of new conjugated polymers derived from bisphenols as potential organic semiconducting materials, see: Jaballah *et al.* (2006). For the use of bis-chloromethyl bisphenol A ethers for the control of fungal and bacterial organisms, see: Priddy & Hennis (1970).



Experimental

Crystal data

$C_{21}H_{26}Cl_2O_2$ M_r = 381.32
Monoclinic, $C2/c$
a = 13.856 (5) Å
b = 15.185 (6) Å
c = 10.999 (4) Å
β = 118.82 (3)°
V = 2027.7 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.33 mm^{-1}
T = 293 (2) K
0.42 × 0.33 × 0.21 mm

Data collection

Enraf–Nonius TurboCAD-4 diffractometer
Absorption correction: none
2374 measured reflections
1960 independent reflections
1173 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
2 standard reflections
frequency: 120 min
intensity decay: 2%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.118
S = 1.02
1960 reflections
166 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 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: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors gratefully acknowledge financial support from the Ministry of Higher Education, Scientific Research and Technology of Tunisia.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KP2175).

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supporting information

Acta Cryst. (2008). E**64**, o1360 [doi:10.1107/S1600536808018783]

2,2-Bis(3-chloromethyl-4-ethoxyphenyl)propane

Nejmeddine Jaballah, Taha Guerfel and Mustapha Majdoub

S1. Comment

BPAEtCl was synthesized as part of an ongoing program on the investigation of new conjugated polymers derived from bisphenols as potential organic semi-conducting materials (Jaballah *et al.*, 2006). This intermediate is of value in synthetic work inasmuch as the CH_2C1 group can be converted to other groups such as CH_2CN, CH_2OH and CHO. Particularly, the bend-like structure of bisphenol A (BPA) nucleus offers a special interest in metacyclophanes synthesis (Miyazawa *et al.*, 1999). Bis-chloromethyl bisphenol A ethers are also useful as microbicides for control of fungal and bacterial organisms (Priddy & Hennis, 1970). The molecular structure of BPAEtCl is shown in Fig. 1. The two benzene rings are nearly perpendicular, forming a dihedral angle of 87.17 (6)°. The ethoxy group plan [O1—C8—C9] is almost parallel with the benzene ring with the dihedral angle of 6.82 (37)° whereas chloromethyl group plan [C1—C7—Cl] is close to be perpendicular [82.62 (13)°].

S2. Experimental

BPAEtCl was synthesized in two steps from 4,4′-isopropylidenediphenol [Bisphenol A, BPA]. To a stirred mixture of BPA (10 mmoles) and K_2CO_3 (40 mmoles) in 20 mL of dimethylformamide, was added dropwise bromoethane (30 mmoles). After stirring for 5 h at room temperature, the reaction mixture was poured into distilled water and extracted with diethyl ether. The extract was washed with distilled water, dried over anhydrous $MgSO_4$, and then evaporated. The resultant crude product was purified by recrystallization from ethanol/water (3/1) to afford the 2,2-bis-(4-ethoxyphenyl)-propane [BPAEt] as needle-like white crystals. A mixture of BPAEt (10 mmoles), paraformaldehyde (2.5 g), and 37% aqueous HCl (8.5 mL) in acetic acid (30 mL) was heated at 328 K for 5 h. The resulting mixture was then poured into distilled water and extracted with diethyl ether. The organic layer was washed several times with distilled water and dried over anhydrous $MgSO_4$. After solvent removal and two recrystallizations from hexane, we obtained BPAEtCl as colourless crystals. Yield: 75%; mp: 352–354 K.

S3. Refinement

Hydrogen atoms were located in a fourier map and refined freely with isotropic thermal parameters.

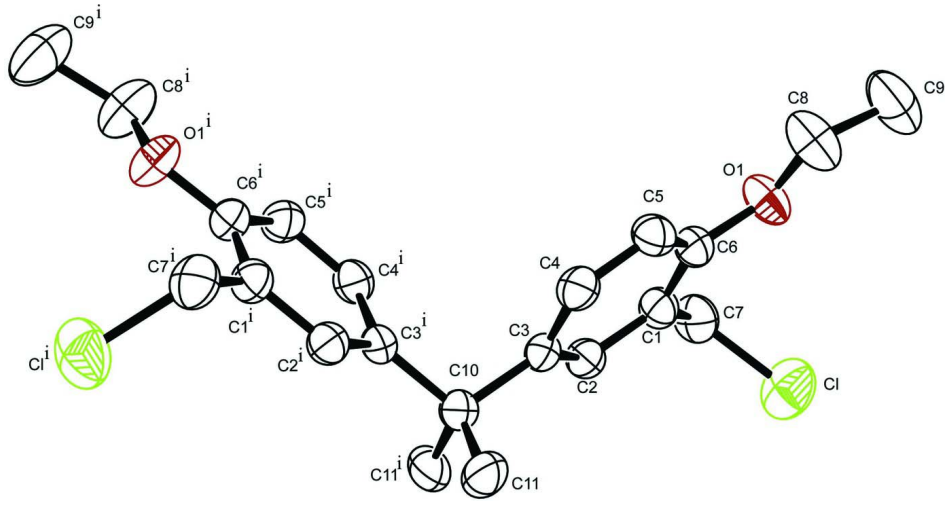

Figure 1

The molecular structure of BPAEtCl, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted. Symmetry code: (i) $-x + 1, y, -z + 5/2$.

2,2-Bis(3-chloromethyl-4-ethoxyphenyl)propane

Crystal data

$C_{21}H_{26}Cl_2O_2$
$M_r = 381.32$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 13.856\ (5)$ Å
$b = 15.185\ (6)$ Å
$c = 10.999\ (4)$ Å
$\beta = 118.82\ (3)°$
$V = 2027.7\ (13)$ Å^3
$Z = 4$
$F(000) = 808$
$D_x = 1.249$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 11.6–15.7°$
$\mu = 0.33$ mm^{-1}
$T = 293$ K
Prism, colourless
0.42 × 0.33 × 0.21 mm

Data collection

Enraf–Nonius TurboCAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
non–profiled ω scans
2374 measured reflections
1960 independent reflections
1173 reflections with $I > 2\sigma(I)$
$R_{int} = 0.022$
$\theta_{max} = 26.0°$, $\theta_{min} = 2.2°$
$h = -17\rightarrow17$
$k = -6\rightarrow18$
$l = -1\rightarrow13$
2 standard reflections every 120 min
intensity decay: 2%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.118$
$S = 1.03$
1960 reflections
166 parameters
0 restraints
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.052P)^2 + 0.7485P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
HC2	0.2827 (17)	0.0620 (14)	1.052 (2)	0.046 (6)*
HC4	0.5740 (19)	0.1686 (14)	1.147 (2)	0.050 (6)*
H111	0.641 (2)	0.0223 (19)	1.220 (3)	0.085 (9)*
H211	0.6149 (19)	−0.0548 (15)	1.306 (3)	0.052 (6)*
H1C7	0.124 (2)	0.1193 (16)	0.880 (3)	0.064 (8)*
HC5	0.4965 (18)	0.2588 (15)	0.961 (2)	0.051 (6)*
H311	0.5386 (19)	−0.0493 (15)	1.142 (3)	0.056 (6)*
H2C7	0.126 (2)	0.2044 (18)	0.786 (3)	0.077 (8)*
H2C8	0.403 (3)	0.278 (2)	0.719 (3)	0.102 (11)*
H1C9	0.318 (3)	0.390 (2)	0.568 (4)	0.109 (13)*
H2C9	0.217 (3)	0.403 (3)	0.598 (4)	0.128 (14)*
H3C9	0.224 (4)	0.315 (3)	0.524 (5)	0.152 (17)*
H1C8	0.392 (3)	0.362 (2)	0.807 (4)	0.107 (12)*
Cl	0.11729 (5)	0.08024 (5)	0.67435 (7)	0.0780 (3)
C10	0.5	0.04410 (18)	1.25	0.0424 (7)
O1	0.28391 (12)	0.26525 (10)	0.77132 (17)	0.0557 (5)
C3	0.43999 (15)	0.10349 (12)	1.1219 (2)	0.0360 (5)
C1	0.27565 (15)	0.15480 (13)	0.9159 (2)	0.0395 (5)
C5	0.45100 (18)	0.21933 (13)	0.9770 (2)	0.0436 (5)
C2	0.32750 (16)	0.10062 (13)	1.0324 (2)	0.0386 (5)
C4	0.49939 (17)	0.16456 (13)	1.0907 (2)	0.0420 (5)
C6	0.33857 (16)	0.21428 (12)	0.8875 (2)	0.0408 (5)
C7	0.15422 (18)	0.14840 (18)	0.8252 (3)	0.0522 (6)
C11	0.5796 (2)	−0.01497 (17)	1.2272 (3)	0.0600 (8)
C8	0.3483 (3)	0.3195 (2)	0.7315 (4)	0.0752 (9)
C9	0.2741 (4)	0.3638 (3)	0.5969 (4)	0.0863 (11)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.0520 (4)	0.0956 (5)	0.0591 (5)	0.0027 (3)	0.0050 (3)	−0.0129 (4)
C10	0.0475 (15)	0.0365 (14)	0.0313 (17)	0	0.0095 (13)	0
O1	0.0532 (9)	0.0566 (9)	0.0505 (11)	0.0097 (7)	0.0195 (8)	0.0228 (8)
C3	0.0406 (10)	0.0330 (9)	0.0282 (12)	−0.0002 (7)	0.0116 (8)	−0.0029 (8)
C1	0.0371 (10)	0.0412 (10)	0.0358 (12)	0.0050 (8)	0.0141 (9)	0.0017 (9)
C5	0.0467 (11)	0.0390 (10)	0.0434 (14)	−0.0058 (9)	0.0204 (10)	0.0022 (10)

C2	0.0382 (10)	0.0382 (10)	0.0367 (13)	−0.0020 (8)	0.0159 (9)	−0.0001 (9)
C4	0.0358 (10)	0.0433 (11)	0.0363 (13)	−0.0055 (8)	0.0090 (9)	−0.0046 (9)
C6	0.0450 (11)	0.0383 (10)	0.0344 (13)	0.0055 (8)	0.0154 (9)	0.0030 (9)
C7	0.0400 (11)	0.0592 (14)	0.0478 (16)	0.0077 (10)	0.0135 (10)	0.0061 (12)
C11	0.0727 (17)	0.0472 (13)	0.0393 (16)	0.0191 (12)	0.0104 (13)	−0.0055 (12)
C8	0.0762 (19)	0.078 (2)	0.071 (2)	0.0090 (16)	0.0357 (17)	0.0314 (17)
C9	0.102 (3)	0.087 (2)	0.080 (3)	0.027 (2)	0.052 (2)	0.042 (2)

Geometric parameters (Å, º)

Cl—C7	1.808 (3)	C5—HC5	0.95 (2)
C10—C11^{i}	1.533 (3)	C2—HC2	0.95 (2)
C10—C11	1.533 (3)	C4—HC4	0.92 (2)
C10—C3^{i}	1.537 (3)	C7—H1C7	0.99 (3)
C10—C3	1.537 (2)	C7—H2C7	0.95 (3)
O1—C6	1.368 (2)	C11—H111	1.06 (3)
O1—C8	1.430 (3)	C11—H211	0.97 (2)
C3—C2	1.386 (3)	C11—H311	0.98 (3)
C3—C4	1.388 (3)	C8—C9	1.494 (4)
C1—C6	1.392 (3)	C8—H2C8	1.04 (3)
C1—C2	1.395 (3)	C8—H1C8	1.00 (4)
C1—C7	1.489 (3)	C9—H1C9	0.91 (4)
C5—C4	1.377 (3)	C9—H2C9	0.99 (4)
C5—C6	1.387 (3)	C9—H3C9	1.07 (5)
C8—C9	1.494 (4)		
C11^{i}—C10—C11	108.4 (3)	C1—C7—Cl	112.38 (17)
C11^{i}—C10—C3^{i}	107.90 (14)	C1—C7—H1C7	107.3 (15)
C11—C10—C3^{i}	112.29 (13)	Cl—C7—H1C7	106.6 (15)
C11^{i}—C10—C3	112.29 (13)	C1—C7—H2C7	109.6 (16)
C11—C10—C3	107.90 (14)	Cl—C7—H2C7	102.7 (17)
C3^{i}—C10—C3	108.1 (2)	H1C7—C7—H2C7	118 (2)
C6—O1—C8	117.78 (18)	C10—C11—H111	111.7 (16)
C2—C3—C4	116.32 (18)	C10—C11—H211	108.1 (14)
C2—C3—C10	123.99 (17)	H111—C11—H211	109 (2)
C4—C3—C10	119.69 (16)	C10—C11—H311	109.7 (14)
C6—C1—C2	119.21 (18)	H111—C11—H311	109 (2)
C6—C1—C7	121.3 (2)	H211—C11—H311	109.5 (18)
C2—C1—C7	119.5 (2)	O1—C8—C9	109.2 (3)
C4—C5—C6	120.0 (2)	O1—C8—H2C8	107.3 (17)
C4—C5—HC5	118.4 (14)	C9—C8—H2C8	109.7 (19)
C6—C5—HC5	121.6 (14)	O1—C8—H1C8	110 (2)
C3—C2—C1	122.58 (19)	C9—C8—H1C8	113 (2)
C3—C2—HC2	119.3 (13)	H2C8—C8—H1C8	108 (3)
C1—C2—HC2	118.1 (13)	C8—C9—H1C9	107 (2)
C5—C4—C3	122.72 (19)	C8—C9—H2C9	115 (2)
C5—C4—HC4	118.3 (14)	H1C9—C9—H2C9	115 (3)
C3—C4—HC4	119.0 (14)	C8—C9—H3C9	109 (2)

O1—C6—C5	123.96 (19)	H1C9—C9—H3C9	110 (3)
O1—C6—C1	116.89 (18)	H2C9—C9—H3C9	101 (3)
C5—C6—C1	119.15 (19)		

Symmetry code: (i) $-x+1, y, -z+5/2$.