1,1,1-Trichloro-2,2-bis(4-ethoxyphenyl)ethane

Graham Smith

Acta Cryst. (2012). E68, o3219

This open-access article is distributed under the terms of the Creative Commons Attribution Licence http://creativecommons.org/licenses/by/2.0/uk/legalcode, which permits unrestricted use, distribution, and reproduction in any medium, provided the original authors and source are cited.

Acta Crystallographica Section E: Structure Reports Online is the IUCr’s highly popular open-access structural journal. It provides a simple and easily accessible publication mechanism for the growing number of inorganic, metal-organic and organic crystal structure determinations. The electronic submission, validation, refereeing and publication facilities of the journal ensure very rapid and high-quality publication, whilst key indicators and validation reports provide measures of structural reliability. The journal publishes over 4000 structures per year. The average publication time is less than one month.

Crystallography Journals Online is available from journals.iucr.org
1,1,1-Trichloro-2,2-bis(4-ethoxyphenyl)ethane

Graham Smith
Science and Engineering Faculty, Queensland University of Technology, GPO Box 2434, Brisbane, Queensland 4001, Australia
Correspondence e-mail: g.smith@qut.edu.au

Received 16 October 2012; accepted 22 October 2012

Key indicators: single-crystal X-ray study; T = 200 K; mean C–C = 0.004 Å; R factor = 0.046; wR factor = 0.092; data-to-parameter ratio = 14.8.

In the title compound, C_{18}H_{19}Cl_{3}O_{2}, which is the 4-ethoxy-phenyl analogue of the insecticidally active 4-methoxyphenyl compound methoxychlor, the dihedral angle between the two benzene rings is 60.38 (13)°. An intramolecular aromatic C–H···Cl interaction is present.

Related literature
For background to DDT-type insecticides, see: Läuger et al. (1944). For unit-cell data for the title compound, see: Schneider & Fankuchen (1946). For the structures of analogous p-alkoxy-substituted DDT compounds, see: Smith et al. (1976); Smith (2012).

Experimental

Crystal data
C_{18}H_{19}Cl_{3}O_{2}
Mr = 373.68
Monoclinic, P_{2}1/c
a = 23.4405 (7) Å
b = 9.8835 (2) Å
c = 7.7924 (2) Å
V = 1780.35 (8) Å^{3}
Z = 4

Mo Kα radiation
μ = 0.52 mm^{-1}
T = 200 K
0.30 × 0.15 × 0.08 mm

Data collection
Oxford Diffraction Gemini-S CCD-detector diffractometer
Absorption correction: multi-scan
(CrysAlis PRO; Agilent, 2012)
T_{min} = 0.960, T_{max} = 0.980
10942 measured reflections
3109 independent reflections
2282 reflections with I > 2σ(I)

Refinement
R[F^2 > 2σ(F^2)] = 0.046
wR(F^2) = 0.092
S = 0.91
3109 reflections
210 parameters
H-atom parameters constrained
Δρ_{max} = 0.38 e Å^{-3}
Δρ_{min} = -0.25 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A
D—H
H···A
D···A
D—H···A

C2B—H2B···Cl2 0.93 2.67 3.321 (3) 128

Data collection: CrysAlis PRO (Agilent, 2012); cell refinement: CrysAlis PRO; data reduction: CrysAlis PRO; program(s) used to solve structure: SIR92 (Altomare et al., 1993); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008) within WinGX (Farrugia, 1999); molecular graphics: PLATON (Spek, 2009); software used to prepare material for publication: PLATON.

The author acknowledges financial support from the Australian Research Council and the Science and Engineering Faculty and the University Library, Queensland University of Technology.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5546).

References
Agilent (2012). CrysAlis PRO. Agilent Technologies Ltd, Yarnton, England.
Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1993). J. Appl. Cryst. 26, 343–350.
Farrugia, L. J. (1999). J. Appl. Cryst. 32, 837–838.
Läuger, P., Martin, H. & Müller, P. H. (1944). Helv. Chim. Acta. 27, 892–928.
Schneider, M. & Fankuchen, I. (1946). J. Am. Chem. Soc. 68, 2669–2670.
Sheldrick, G. M. (2008). Acta Cryst. A64, 112–122.
Smith, G. (2012). Acta Cryst. E68, o2544.
Smith, G., Kennard, C. H. L. & White, A. H. (1976). Aust. J. Chem. 29, 743–747.
Spek, A. L. (2009). Acta Cryst. D65, 148–155.
The title compound, C_{18}H_{19}Cl_{3}O_{2} is the 4-ethoxyphenyl analogue of the insecticides DDT [1,1,1-trichloro-2,2-bis(4-chlorophenyl)ethane] and methoxychlor (the 4-methoxyphenyl analogue), and has similar insecticidal activity (Läuger et al., 1944), but was not used as a commercial product. The crystal structures of methoxychlor (Smith et al., 1976) and the p-butoxy analogue (Smith, 2012) are known and the unit cell data for the title compound has been reported (Schneider & Fankuchen, 1946). The structure of the title compound is reported herein.

The molecular structure of the title compound is shown in Fig. 1. The unit cell and space group are consistent with those previously reported. The dihedral angle between the two benzene ring mean planes is 60.38 (13)°. The value of this angle is 77.7° (no s.u. available) in the structure of methoxychlor (Smith et al., 1976). The conformations of the two ethoxy side chains relative to their benzene rings (A and B) are similar [comparative torsion angles C3—C4—O4—C11, -173.3 (3) and 162.2 (2), respectively]. The B ring conformation is stabilized by an intramolecular aromatic C2B—H···Cl2 interaction (Table 1). No significant intermolecular interactions are present (Fig. 2).

Experimental
The title compound was obtained as an analytical reference standard from the US Public Health Service. Colourless crystal prisms suitable for X-ray analysis were obtained by room temperature evaporation of a solution of the title compound in ethanol.

Refinement
Hydrogen atoms were included in the refinement at calculated positions [C—H = 0.93–0.98 Å, with \(U_{iso}(H) = 1.2U_{eq}(C)\) (aromatic, methylene and methine) or 1.5\(U_{eq}(C)\)(methyl), using a riding-model approximation.

Computing details
Data collection: CrysAlis PRO (Agilent, 2012); cell refinement: CrysAlis PRO (Agilent, 2012); data reduction: CrysAlis PRO (Agilent, 2012); program(s) used to solve structure: SIR92 (Altomare et al., 1993); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008) within WinGX (Farrugia, 1999); molecular graphics: PLATON (Spek, 2009); software used to prepare material for publication: PLATON (Spek, 2009).
Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

Figure 2
A perspective view of the crystal packing in the unit cell viewed approximately along the b axis.

1,1,1-Trichloro-2,2-bis(4-ethoxyphenyl)ethane

Crystal data

\[ \text{C}_{18}\text{H}_{19}\text{Cl}_{3}\text{O}_{2} \]

\[ M_r = 373.68 \]

Monoclinic, \( P2_1/c \)

Hall symbol: -P 2yb\( b \)

\[ a = 23.4405 (7) \text{ Å} \]

\[ b = 9.8835 (2) \text{ Å} \]

\[ c = 7.7924 (2) \text{ Å} \]

\[ \beta = 99.536 (3)^\circ \]

\[ V = 1780.35 (8) \text{ Å}^3 \]

\[ Z = 4 \]

\[ F(000) = 776 \]

\[ D_x = 1.394 \text{ Mg m}^{-3} \]

\( \text{Mo} \text{ K}\alpha \) radiation, \( \lambda = 0.71073 \text{ Å} \)

Cell parameters from 3070 reflections

\[ \theta = 3.1–28.8^\circ \]

\[ \mu = 0.52 \text{ mm}^{-1} \]

\[ T = 200 \text{ K} \]

Prism, colourless

\[ 0.30 \times 0.15 \times 0.08 \text{ mm} \]
supplementary materials

**Data collection**

Oxford Diffraction Gemini-S CCD-detector
diffractometer

Radiation source: Enhance (Mo) X-ray source
Graphite monochromator

Detector resolution: 16.077 pixels mm^{-1}

ω scans
Absorption correction: multi-scan
(CrysAlis PRO; Agilent, 2012)

\[ \frac{T_{\text{min}}}{T_{\text{max}}} = 0.960 \text{, } 0.980 \]

10942 measured reflections
3109 independent reflections
2282 reflections with \( I > 2\sigma(I) \)

\[ R_{\text{int}} = 0.106, \quad \theta_{\text{max}} = 25.0^\circ, \quad \theta_{\text{min}} = 3.4^\circ \]

\[ h = -27 \rightarrow 23, \quad k = -11 \rightarrow 11, \quad l = -9 \rightarrow 9 \]

**Refinement**

Refinement on \( F^2 \)

Least-squares matrix: full

\[ R[F^2 > 2\sigma(F^2)] = 0.046 \]

\[ wR(F^2) = 0.092 \]

\[ S = 0.91 \]

3109 reflections
210 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

\[ w = 1/(\sigma^2(F_o^2) + (0.013P)^2) \]

where \( P = (F_o^2 + 2F_c^2)/3 \)

\[ \Delta/\sigma \text{max} < 0.001 \]

\[ \Delta \rho_{\text{max}} = 0.38 \text{ e Å}^{-3} \]

\[ \Delta \rho_{\text{min}} = -0.25 \text{ e Å}^{-3} \]

**Special details**

**Geometry.** Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

**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)**

|     | \( x \)   | \( y \)   | \( z \)   | \( U_{iso} \) |
|-----|-----------|-----------|-----------|--------------|
| Cl1 | 0.33099 (3)| −0.05016 (8)| 0.40168 (9)| 0.0385 (3)   |
| Cl2 | 0.23442 (3)| −0.19217 (7)| 0.50376 (9)| 0.0374 (3)   |
| Cl3 | 0.21520 (3)| 0.01715 (8)| 0.24488 (9)| 0.0412 (3)   |
| O4A | 0.02901 (9)| 0.0931 (2)| 0.7508 (3)| 0.0442 (8)   |
| O4B | 0.44289 (8)| 0.0498 (2)| 1.1563 (2)| 0.0406 (7)   |
| Cl  | 0.25902 (12)| −0.0312 (3)| 0.4447 (3)| 0.0274 (9)   |
| Cl1A| 0.19612 (11)| 0.0869 (3)| 0.6338 (3)| 0.0228 (9)   |
| Cl1B| 0.30558 (11)| 0.0680 (3)| 0.7397 (3)| 0.0247 (9)   |
| C2  | 0.25640 (11)| 0.0785 (3)| 0.5851 (3)| 0.0240 (9)   |
| C2A | 0.17938 (12)| 0.0081 (3)| 0.7642 (3)| 0.0293 (10)  |
| C2B | 0.31987 (12)| −0.0486 (3)| 0.8366 (3)| 0.0293 (9)   |
| C3A | 0.12369 (13)| 0.0129 (3)| 0.7994 (4)| 0.0325 (10)  |
| C3B | 0.36559 (12)| −0.0512 (3)| 0.9749 (4)| 0.0322 (10)  |
| C4A | 0.08312 (12)| 0.0979 (3)| 0.7062 (4)| 0.0304 (10)  |
| C4B | 0.39827 (12)| 0.0635 (3)| 1.0180 (3)| 0.0301 (10)  |
| C5A | 0.09942 (12)| 0.1816 (3)| 0.5803 (3)| 0.0316 (10)  |
| C5B | 0.38476 (12)| 0.1820 (3)| 0.9249 (3)| 0.0314 (10)  |
supplementary materials

|       | U^11   | U^22   | U^33   | U^12   | U^13   | U^23   |
|-------|--------|--------|--------|--------|--------|--------|
| Cl1   | 0.0348 (5) | 0.0443 (5) | 0.0374 (5) | 0.0061 (4) | 0.0093 (3) | -0.0034 (4) |
| Cl2   | 0.0452 (5) | 0.0235 (4) | 0.0428 (5) | -0.0032 (3) | 0.0053 (4) | -0.0059 (3) |
| Cl3   | 0.0435 (5) | 0.0481 (5) | 0.0263 (4) | 0.0079 (4) | -0.0054 (3) | -0.0015 (4) |
| O4A   | 0.0280 (13) | 0.0556 (15) | 0.0488 (14) | 0.0015 (11) | 0.0054 (10) | -0.0016 (11) |
| O4B   | 0.0327 (12) | 0.0469 (14) | 0.0373 (12) | -0.0064 (10) | -0.0081 (10) | 0.0016 (10) |
| C1    | 0.0303 (17) | 0.0255 (16) | 0.0252 (15) | 0.0056 (13) | 0.0014 (13) | 0.0001 (13) |
| C1A   | 0.0249 (16) | 0.0193 (14) | 0.0230 (15) | 0.0016 (12) | 0.0005 (12) | -0.0031 (12) |
| C1B   | 0.0250 (16) | 0.0248 (15) | 0.0244 (15) | 0.0002 (13) | 0.0044 (12) | 0.0024 (13) |
| C2    | 0.0288 (16) | 0.0162 (14) | 0.0254 (15) | 0.0014 (12) | -0.0003 (12) | 0.0003 (12) |
| C2A   | 0.0333 (18) | 0.0210 (15) | 0.0316 (17) | 0.0031 (13) | -0.0001 (14) | 0.0034 (13) |
| C2B   | 0.0276 (17) | 0.0269 (15) | 0.0314 (17) | -0.0041 (13) | 0.0007 (13) | 0.0021 (13) |
| C3A   | 0.0357 (18) | 0.0270 (16) | 0.0350 (18) | -0.0015 (14) | 0.0062 (14) | 0.0031 (14) |
| C3B   | 0.0337 (18) | 0.0307 (17) | 0.0315 (17) | 0.0014 (14) | 0.0030 (14) | 0.0064 (14) |
| C4A   | 0.0262 (17) | 0.0338 (17) | 0.0303 (17) | -0.0023 (14) | 0.0025 (14) | -0.0093 (14) |
| C4B   | 0.0289 (17) | 0.0385 (18) | 0.0219 (16) | 0.0016 (14) | 0.0014 (13) | -0.0041 (14) |
| C5A   | 0.0319 (18) | 0.0286 (17) | 0.0312 (17) | 0.0052 (14) | -0.0036 (14) | 0.0021 (14) |
| C5B   | 0.0306 (17) | 0.0301 (17) | 0.0328 (17) | -0.0063 (14) | 0.0036 (14) | -0.0070 (14) |
| C6A   | 0.0347 (18) | 0.0250 (15) | 0.0262 (16) | -0.0008 (13) | 0.0007 (13) | 0.0023 (13) |
| C6B   | 0.0302 (17) | 0.0251 (16) | 0.0268 (16) | 0.0010 (13) | 0.0051 (13) | 0.0018 (13) |
| C11A  | 0.0314 (19) | 0.050 (2) | 0.061 (2) | 0.0063 (17) | -0.0013 (17) | -0.0054 (18) |
| C11B  | 0.0329 (19) | 0.051 (2) | 0.0425 (19) | -0.0057 (16) | 0.0028 (15) | -0.0063 (16) |

Acta Cryst. (2012). E68, o3219
Geometric parameters (Å, °)

| Bond/Angle | Distance/Angle |
|------------|---------------|
| C11—C1     | 1.784 (3)     |
| C11A—C21A  | 1.496 (5)     |
| C12—C1     | 1.779 (3)     |
| C11B—C21B  | 1.508 (4)     |
| C13—C1     | 1.783 (3)     |
| C2—H2      | 0.9800        |
| O4A—C4A    | 1.370 (4)     |
| C2A—H2A    | 0.9300        |
| C11A—C21A  | 1.496 (5)     |
| C11B—C21B  | 1.508 (4)     |
| Geometric parameters (Å, °) |
| C1—C2      | 1.549 (4)     |
| C5A—H5A    | 0.9300        |
| C1A—C2     | 1.525 (4)     |
| C5B—H5B    | 0.9300        |
| C1A—C2A    | 1.388 (4)     |
| C6A—H6A    | 0.9300        |
| C1A—C2A    | 1.388 (4)     |
| C6B—H6B    | 0.9300        |
| C1B—C2     | 1.527 (3)     |
| C11A—H11A  | 0.9700        |
| C1B—C2B    | 1.388 (4)     |
| C11A—H12A  | 0.9700        |
| C1B—C6B    | 1.391 (4)     |
| C11B—H11B  | 0.9700        |
| C2A—C3A    | 1.379 (4)     |
| C1B—C6B    | 1.391 (4)     |
| C2B—C3B    | 1.389 (4)     |
| C1C—C2     | 1.112 (2)     |
| C2A—C3A    | 1.225 (2)     |
| C2B—C3B    | 1.246 (3)     |
| C2B—C3B    | 1.112 (2)     |
| C2C—C3A    | 1.112 (2)     |
| C2B—C3B    | 1.112 (2)     |
| C2B—C3B    | 1.112 (2)     |
| C2B—C3B    | 1.112 (2)     |
| C2B—C3B    | 1.112 (2)     |

Acta Cryst. (2012). E68, o3219
supplementary materials

O4A—C4A—C5A  125.2 (3)  C21B—C11B—H11B  110.00
C3A—C4A—C5A  119.3 (3)  C21B—C11B—H12B  110.00
O4B—C4B—C3B  115.4 (2)  H11B—C11B—H12B  109.00
O4B—C4B—C5B  124.8 (3)  C11A—C21A—H21A  109.00
C3B—C4B—C5B  119.9 (2)  C11A—C21A—H22A  109.00
C4A—C5A—C6A  119.4 (3)  C11A—C21A—H22A  109.00
C4B—C5B—C6B  119.4 (3)  H21A—C21A—H22A  109.00
C1A—C6A—C5A  122.1 (2)  H21A—C21A—H23A  109.00
C1B—C6B—C5B  121.8 (3)  H22A—C21A—H23A  110.00
O4A—C11A—C21A  107.8 (3)  C11B—C21B—H21B  109.00
O4B—C11B—C21B  107.8 (2)  C11B—C21B—H22B  109.00
C1—C2—H2  106.00  C11B—C21B—H23B  110.00
C1A—C2—H2  106.00  H21B—C21B—H22B  109.00
C1B—C2—H2  106.00  H21B—C21B—H23B  109.00
C1A—C2A—H2A  119.00  H22B—C21B—H23B  109.00
C11A—O4A—C4A—C3A  −173.3 (3)  C2B—C1B—C2—C1  −52.8 (3)
C11A—O4A—C4A—C5A  8.0 (4)  C2B—C1B—C2—C1A  76.3 (3)
C4A—O4A—C11A—C21A  173.7 (3)  C6B—C1B—C2—C1  127.1 (3)
C11B—O4B—C4B—C3B  162.2 (2)  C6B—C1B—C2—C1A  −103.8 (3)
C11B—O4B—C4B—C5B  −18.4 (4)  C2—C1B—C2B—C3B  179.4 (3)
C4B—O4B—C11B—C21B  −174.3 (2)  C6B—C1B—C2B—C3B  −0.5 (4)
C1—C1—C2—C1A  −175.16 (18)  C2—C1B—C6B—C5B  −179.1 (2)
C11—C1—C2—C1B  −44.3 (3)  C2B—C1B—C6B—C5B  0.8 (4)
C12—C1—C2—C1A  −53.5 (3)  C1A—C2A—C3A—C4A  −0.7 (4)
C12—C1—C2—C1B  77.3 (3)  C1B—C2B—C3B—C4B  −0.5 (4)
C13—C1—C2—C1A  66.8 (2)  C2A—C3A—C4A—O4A  179.2 (3)
C13—C1—C2—C1B  162.36 (19)  C2A—C3A—C4A—C5A  −2.0 (4)
C2A—C1A—C2—C1  88.5 (3)  C2B—C3B—C4B—O4B  −179.5 (2)
C2A—C1A—C2—C1B  −41.7 (4)  C2B—C3B—C4B—C5B  1.1 (4)
C6A—C1A—C2—C1  −91.0 (3)  O4A—C4A—C5A—C6A  −179.0 (3)
C6A—C1A—C2—C1B  138.9 (3)  C3A—C4A—C5A—C6A  2.4 (4)
C2—C1A—C2A—C3A  −176.7 (3)  O4B—C4B—C5B—C6B  179.9 (2)
C6A—C1A—C2A—C3A  2.8 (4)  C3B—C4B—C5B—C6B  −0.8 (4)
C2—C1A—C6A—C5A  177.1 (2)  C4A—C5A—C6A—C1A  −0.2 (4)
C2A—C1A—C6A—C5A  −2.4 (4)  C4B—C5B—C6B—C1B  −0.2 (4)

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|-------|-------|---------|
| C2B—H2B···Cl2  | 0.93 | 2.67  | 3.321 (3) | 128 |