# 2-[Bis(4-methoxyphenyl)methyl]benzyl chloroacetate

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Key indicators

Single-crystal X-ray study
$T = 133$ K
Mean σ(C–C) = 0.002 Å
R factor = 0.041
wR factor = 0.116
Data-to-parameter ratio = 23.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_{24}H_{23}ClO_4$, the side chain displays an extended conformation. The main secondary interactions involve a bifurcated hydrogen-bond system (C—H···)$_2$O to the carbonyl O atom. Each ring is involved in a long C—H···π interaction.

Comment

Triphenylmethane is well known as a basic framework for many organic dyes and pigments (*e.g.* crystal violet, malachite green and phenolphthalein). Its structure has been reported by Riche & Pascard-Billy (1974) and at low temperature by Veldman *et al.* (1996). The trityl group is used as a protecting group for hydroxyl functions. Our search for starting materials for the synthesis of new classes of cyclophanes led us to consider phenolphthalein as a suitable candidate. This compound has two *para*-hydroxyphenyl units that can be incorporated into the cyclophane ring; these units are connected by a methylene spacer, which is a very important feature. During our investigations we obtained the title triphenylmethyl derivative, (I). The synthesis of the structurally similar compound {bis(4-methoxyphenyl)[2-methoxyphenyl]}methane was reported previously by Gomberg & Snow (1925). The crystal structure of the related but more symmetrical compound 1,1,1-tris(4-hydroxyphenyl)methane was reported, and its hydrogen bonding discussed in detail, by Aitipamula *et al.* (2003).

(I)
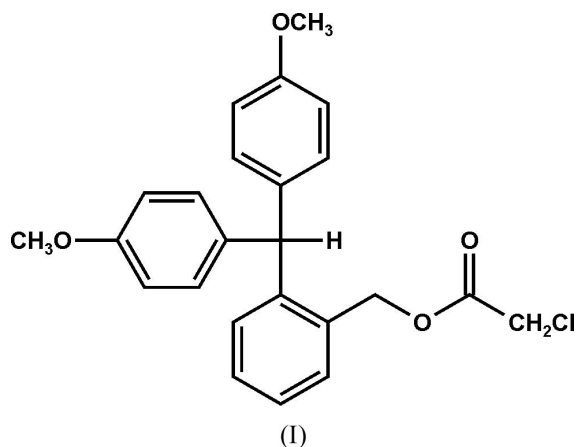
Bond lengths and angles in (I) may be regarded as normal. The largest angle at C1 is C31—C1—C11 114.30 (9)°. The side chain displays an extended conformation (Table 1). Ring orientations are defined by the torsion angles about C1—CX1 (X = 1–3; Table 1).

The molecules of (I) are connected by a series of secondary interactions. Four weak hydrogen bonds are shown in Table 2.
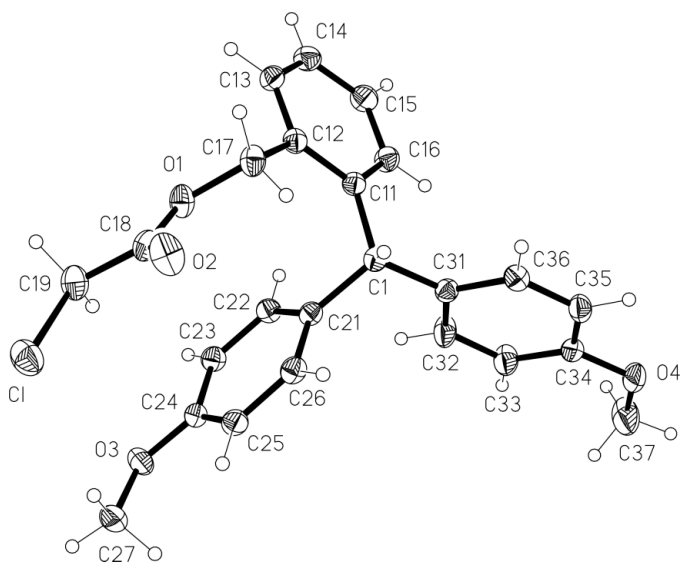
Figure 1
The molecule of (I) in the crystal structure, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atom radii are arbitrary.

The two shortest C—H⋯O interactions form a bifurcated system with the common acceptor, O2 (the carbonyl O atom), and link the molecules in pairs *via* inversion symmetry. Each ring centroid (*CgX*, for C*X*1–C*X*6) is involved in a C—H⋯π interaction [H33⋯*Cg*1 2.76 Å, symmetry code (1 + *x*, *y*,*z*); H17*B*⋯*Cg*2 2.94 Å, symmetry code (−1 + *x*, *y*, *z*); H15⋯*Cg*3 2.88 Å, symmetry code (-*x*, −*y*, −*z*)], but the H⋯*Cg* distances, here calculated for normalized C—H = 1.08 Å, are quite long.

Experimental

The title compound was obtained in 30% yield during hydrogenation of dimethylphenolphthalein (1mmol) by borane–dimethyl sulfide in tetrahydrofuran solution, followed by acylation with chloroacetic anhydride (1mmol) in dichloromethane in the presence of pyridine. In the first step of this reaction, the molecule lost a hydroxyl unit, which was replaced by hydrogen. Analytical and spectroscopic data for (I): analysis (of crude product before recrystallization) calculated for $C_{24}H_{23}ClO_4 \cdot H_2O$: C 67.21, H 5.87%; found: C 67.83, H 5.59; m.p. 348–350K; ^{1}H NMR (CDCl$_3$, 400 MHz, δ, p.p.m.): 3.71 (*s*, 6H), 5.11 (*s*, 2H), 5.63 (*s*, 1H), 6.74, 6.87 (*dd*, 8H, *J* = 8.8 Hz), 7.14–7.22 (*m*, 3H), 7.30 (*d*, 1H); ^{13}C NMR (CDCl$_3$, δ, p.p.m.): 40.81, 51.04, 55.38, 66.21, 113.88, 126.76, 129.06, 130.33, 130.47, 130.62, 133.01, 135.72, 143.77, 158.22, 167.13; IR (KBr pellet, v, cm^{-1}): 1758 (C=O).

Crystal data

$C_{24}H_{23}ClO_4$	$Z = 2$
$M_r = 410.87$	$D_x = 1.320$ Mg m^{-3}
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 7.6817\ (4)$ Å	Cell parameters from 8877
$b = 10.6398\ (6)$ Å	reflections
$c = 13.4992\ (8)$ Å	$\theta = 2.7$–30.5°
$\alpha = 108.529\ (4)°$	$\mu = 0.21$ mm^{-1}
$\beta = 92.712\ (4)°$	$T = 133\ (2)$ K
$\gamma = 97.109\ (4)°$	Prism, colourless
$V = 1033.67\ (10)$ Å^3	0.35 × 0.21 × 0.16 mm

Data collection

Bruker SMART 1000 CCD	6274 independent reflections
diffractometer	4988 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.024$
Absorption correction: multi-scan	$\theta_{max} = 30.5°$
(*SADABS*; Bruker, 1998)	$h = -10 \rightarrow 10$
$T_{min} = 0.890$, $T_{max} = 0.967$	$k = -14 \rightarrow 15$
22 190 measured reflections	$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o{}^2) + (0.0613P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.041$	$+ 0.2812P]$
$wR(F^2) = 0.116$	where $P = (F_o{}^2 + 2F_c{}^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.001$
6274 reflections	$\Delta\rho_{max} = 0.41$ e Å^{-3}
264 parameters	$\Delta\rho_{min} = -0.21$ e Å^{-3}
H-atom parameters constrained	Extinction correction: none

Table 1
Selected geometric parameters (°).

C31—C1—C11	114.30 (9)		
C18—O1—C17—C12	160.60 (10)	H1—C1—C11—C12	−51.5
C17—O1—C18—C19	−179.76 (10)	H1—C1—C21—C26	−27.9
O1—C18—C19—Cl	171.84 (8)	H1—C1—C31—C36	−26.1

Table 2
Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C1—H1⋯O2^i	1.00	2.43	3.3793 (14)	157
C26—H26⋯O2^i	0.95	2.58	3.4039 (15)	145
C27—H27*B*⋯O4^{ii}	0.98	2.65	3.4856 (16)	143
C37—H37*B*⋯Cl^{iii}	0.98	3.00	3.9767 (16)	179

Symmetry codes: (i) −*x*, −*y* + 1, −*z* + 1; (ii) *x*, *y*, *z* + 1; (iii) *x* + 1, *y*, *z* − 1.

Methyl H atoms were identified in difference syntheses, idealized and then refined using rigid methyl groups (C—H = 0.98 Å and H—C—H = 109.5°) allowed to rotate but not tip. Other H atoms were included using a riding model with C—H = 0.95 (*sp*^{2}), 0.99 (methylene) or 1.00 (methine) Å. For all H, U_{iso}(H) = 1.2U_{eq}(parent)..

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL97*.

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