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Benzyl 2,3,4,6-tetramethylbenzoate

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Key indicators

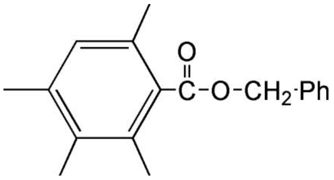
Single-crystal X-ray study
T = 110 K
Mean σ(C–C) = 0.002 Å
R factor = 0.044
wR factor = 0.123
Data-to-parameter ratio = 19.6

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

In the title compound, $C_{18}H_{20}O_2$, the C(=O)O group is twisted by 68.1 (1)° from the plane of the 2,3,4,6-tetramethylbenzyl ring. The methylene C atom is almost coplanar with the carbonylate group.

Comment

The title compound, (I), was prepared in connection with studies (Pinkus, Klausmeyer *et al.*, 2005; Pinkus & Lin, 1975) of sterically hindered esters for the purpose of investigating whether magnetic non-equivalence (Pinkus & Lin, 1975; van Gorkom & Hall, 1968; Welch, 1997; Hoye *et al.*, 1994) in the methylene H atoms could be detected in the NMR spectrum. The ^{1}H NMR spectrum (360 MHZ) showed only a singlet for the methylene H atoms at 6.906 p.p.m. This may be because of the distance of the methylene H atoms from the asymmetrically situated 4-methyl group, or insufficient hindrance to rotation involving the methylene group, or a combination of the two possibilities. The distance would be greater than any previously reported to our knowledge (van Gorkom & Hall, 1968; Hoye *et al.*, 1994). Crystallographically, the methylene H atoms are in slightly different environments with respect to the 2,3,4,6-tetramethylbenzyl ring. The twist of the C(=O)O group from the latter plane is described by the torsion angle C2—C1—C7—O2 [68.1 (1)°]. The methylene C atom is nearly coplanar with respect to the CC(=O)O group, with an O1—C7—O2—C8 torsion angle of 2.7 (1)°.



(I)

Experimental

The title compound, (I), was synthesized by a previously described method (Pinkus, Klausmeyer *et al.*, 2005) for ethyl 2,3,5,6-tetramethylbenzoate. 2,3,4,6-Tetramethylbenzoic acid was first converted to the acid chloride and then to the benzyl ester. The reagents used were 2,3,4,6-tetramethylbenzoic acid (9.10 g, 0.0512 mol), thionyl chloride (18.0 g, 0.153 mol) benzyl alcohol (15 g, 0.138 mol) and pyridine (8.10 g, 0.103 mol). A brown liquid which was obtained was dried over calcium chloride and distilled through a fractionating column at reduced pressure, b.p. 439.5–440.50 K (< 1 mm), nD25 (refractive index at 298 K) 1.5514, yielding a colorless liquid (11.8 g) in 86.5% yield based on 2,3,4,6-tetramethylbenzoic acid. The liquid solidified on standing (m.p. 312.5–313.5 K). IR (neat) (cm^{-1}): 1719 (C=O str), 1262 (C—O str), 1606, 1456, 1148, 1038, 740, 701. ^{1}H

NMR (360 MHz, $CDCl_3$, TMS, p.p.m.): 7.297–7.436 (*m*, 5 Me-C_6H_5), 6.906 (*s*, 4-H), 5.417 (*s*, CH_2), 2.305, 2.259, 2.240, 2.191 (four *s*-CH_3). Analysis (MHW Labs) calculated for $C_{18}H_{20}O_2$: C 80.56, H 7.51%; found: C 80.73, H 7.4%.

Crystal data

$C_{18}H_{20}O_2$
$M_r = 268.34$
Monoclinic, $P2_1/n$
$a = 15.4921\ (19)$ Å
$b = 5.5915\ (8)$ Å
$c = 17.1846\ (19)$ Å
$\beta = 97.815\ (5)°$
$V = 1474.8\ (3)$ Å^3
$Z = 4$

$D_x = 1.209$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 4540 reflections
$\theta = 2.7$–$28.3°$
$\mu = 0.08$ mm^{-1}
$T = 110\ (2)$ K
Block, colorless
0.13 × 0.12 × 0.11 mm

Data collection

Bruker X8 APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.984$, $T_{max} = 0.993$
11515 measured reflections

3631 independent reflections
3026 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 28.3°$
$h = -20 \rightarrow 17$
$k = -6 \rightarrow 7$
$l = -12 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.123$
$S = 1.04$
3631 reflections
185 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0578P)^2 + 0.5741P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C7	1.2116 (14)	C1—C7	1.4973 (17)
O2—C7	1.3412 (14)	C8—C9	1.5048 (17)
O2—C8	1.4642 (15)		
C7—O2—C8	115.92 (9)	O2—C7—C1	111.56 (9)
O1—C7—O2	123.84 (11)	O2—C8—C9	107.21 (9)
O1—C7—C1	124.58 (11)		
C2—C1—C7—O2	68.18 (15)	C1—C7—O2—C8	−178.81 (10)

H atoms were included in calculated positions (C—H = 0.93 Å); isotropic displacement parameters were fixed [$U_{iso}(H) = 1.2U_{iso}(C)$]. The methyl groups were allowed to rotate but not to tip.

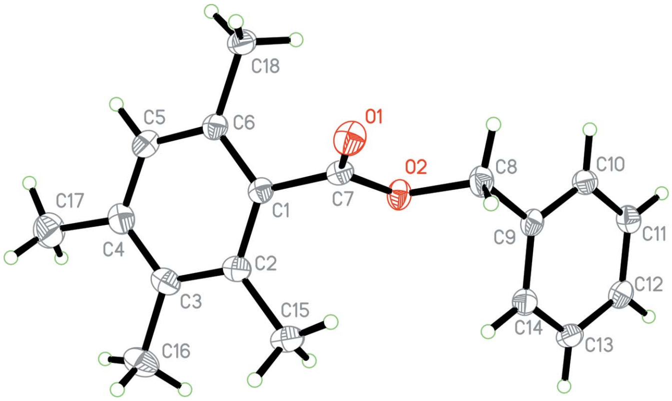


Figure 1
A view of the molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

Data collection: *APEX2* (Bruker, 2003); cell refinement: *APEX2*; data reduction: *SAINT-Plus* (Bruker, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2000); software used to prepare material for publication: *SHELXTL*.

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