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Ethyl 2,3,5,6-tetramethylbenzoate

A. G. Pinkus,* Kevin K. Klausmeyer, Rodney P. Feazell and Ellen C. H. Y. Lin

Department of Chemistry and Biochemistry, Baylor University, Waco, TX 76798, USA

Correspondence e-mail:
a_g_pinkus@baylor.edu

Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.002 Å
R factor = 0.039
wR factor = 0.108
Data-to-parameter ratio = 16.6

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

The crystal structure of ethyl 2,3,5,6-tetramethylbenzoate, $C_{13}H_{18}O_2$, has been determined and torsion angles are compared to those previously reported in a dipole moment study of substituted benzoates.

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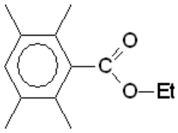
Comment

The crystal structure of the title compound, (I), was determined to compare data on the conformation of the carboethoxy group with the data obtained from previous dipole moment studies obtained in solution. The interplanar angle between the duryl ring plane and the O=C—O plane calculated from the Braude–Sondheimer (1955) equation in the latter case was 49°, whereas the angle determined for ethyl benzoate was 0° (Pinkus & Lin, 1975). The corresponding angle for the title compound determined crystallographically was 79.9 (2)°. This demonstrates the steric factor of the methyl groups overcoming the resonance energy that would favor a planar system. The molecular structure of compound (I) is displayed in Fig. 1. Selected interatomic distances and angles are given in Table 1. All bond lengths and angles are as expected for an *o*-substituted benzoate.



(I)

Experimental

The title compound was prepared in 85% yield by the reaction of 2,3,5,6-tetramethylbenzoyl chloride and ethanol following the procedure described by Newman & Lloyd (1952) (m.p. 353.9–354.7 K; literature 351.6–352.4 K). IR (cm^{-1}) 1729 (C=O str). ^{1}H NMR (60 MHz, CCl_4, TMS, p.p.m.): 2.16 (*s*, *o*-Me), 2.06 (*s*, *m*-Me), 6.83 (*s*, *p*-H), 4.32 (quart, —CH_2—), 1.33 (trip, —CH_3).

Crystal data

$C_{13}H_{18}O_2$
M_r = 206.27
Monoclinic, $P2_1/c$
a = 18.223 (1) Å
b = 5.5837 (4) Å
c = 11.5842 (7) Å
β = 101.725 (4)°
V = 1154.12 (13) Å^3
Z = 4

D_x = 1.187 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2643 reflections
θ = 2.3–27.7°
μ = 0.08 mm^{-1}
T = 110 (2) K
Block, colorless
0.19 × 0.10 × 0.08 mm

Data collection

Bruker X8 APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.98$, $T_{\max} = 0.99$
10368 measured reflections
2342 independent reflections
1777 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.035$
$\theta_{\max} = 26.4°$
$h = -22 \rightarrow 22$
$k = -6 \rightarrow 6$
$l = -14 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.108$
$S = 1.02$
2342 reflections
141 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0492P)^2 + 0.3725P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.25$ e Å^{-3}
$\Delta\rho_{\min} = -0.19$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.3369 (16)	C4—C5	1.3921 (19)
O1—C12	1.4591 (15)	C4—C9	1.5056 (19)
O2—C1	1.2048 (16)	C5—C6	1.3894 (19)
C1—C2	1.5055 (18)	C6—C7	1.4034 (18)
C2—C3	1.3986 (19)	C6—C10	1.5079 (18)
C2—C7	1.4001 (18)	C7—C11	1.5064 (19)
C3—C4	1.4015 (18)	C12—C13	1.498 (2)
C3—C8	1.5087 (18)		
C1—O1—C12	115.66 (10)	O2—C1—C2	124.66 (12)
O2—C1—O1	123.81 (12)	O1—C1—C2	111.53 (11)
C3—C2—C1—O1	79.95 (15)		

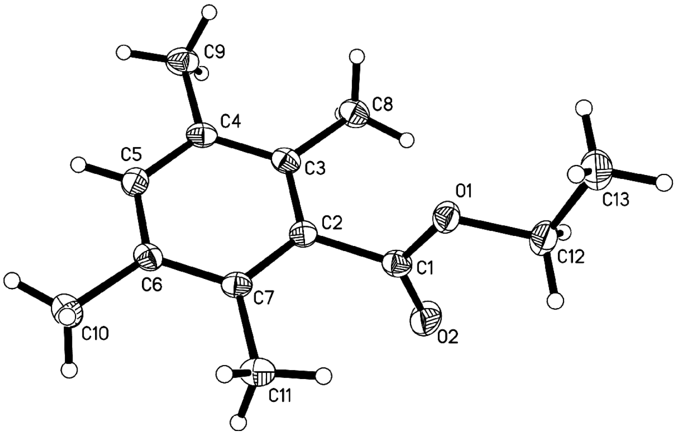


Figure 1
A view of the molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level

H atoms were included in calculated positions (C—H = 0.93 and 0.96 Å) and their isotropic displacement parameters were fixed [$U_{\rm iso}$(H) = 1.2 and 1.5$U_{\rm iso}$(C) for aromatic and methyl H atoms, respectively].

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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