

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Methyl 4-methylbenzoate

 Aamer Saeed,^a Hummera Rafique^a and Ulrich Flörke^{b*}
^aDepartment of Chemistry, Quaid-i-Azam University Islamabad, Pakistan, and

^bDepartment Chemie, Fakultät für Naturwissenschaften, Universität Paderborn, Warburgerstrasse 100, D-33098 Paderborn, Germany

Correspondence e-mail: aamersaeed@yahoo.com

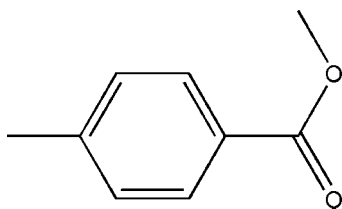
Received 29 March 2008; accepted 1 April 2008

 Key indicators: single-crystal X-ray study; $T = 120$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.042; wR factor = 0.125; data-to-parameter ratio = 18.2.

The structure of the title compound, $\text{C}_9\text{H}_{10}\text{O}_2$, is related to that of 4-methylphenyl 4-methylbenzoate and ethylene di-4-methylbenzoate showing similar bond parameters. The molecule is planar, the dihedral angle between the aromatic ring and the $-\text{COOMe}$ group being 0.95 (6)°. The crystal structure exhibits intermolecular $\text{C}-\text{H}\cdots\text{O}$ contacts that link molecules into infinite chains extended in the $[001]$ direction.

Related literature

For related literature, see: Deguire & Brisse (1988); Gowda *et al.* (2007); Gray & Whalley (1971); Harris & Mantle (2001); Saeed & Rama (1994); Simpson (1978).



Experimental

Crystal data

 $\text{C}_9\text{H}_{10}\text{O}_2$
 $M_r = 150.17$
 Monoclinic, $P2_1/c$
 $a = 5.9134$ (11) Å
 $b = 7.6048$ (14) Å
 $c = 17.484$ (3) Å

 $\beta = 97.783$ (4)°
 $V = 779.0$ (2) Å³
 $Z = 4$
 Mo $K\alpha$ radiation

 $\mu = 0.09$ mm⁻¹
 $T = 120$ (2) K
 $0.45 \times 0.43 \times 0.39$ mm

Data collection

 Bruker SMART APEX diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 2004)
 $T_{\min} = 0.961$, $T_{\max} = 0.967$

 6617 measured reflections
 1855 independent reflections
 1482 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.038$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.124$
 $S = 1.06$
 1855 reflections

 102 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.31$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.20$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|-------|-------------|-------------|---------------|
| $\text{C9}-\text{H9B}\cdots\text{O2}^i$ | 0.98 | 2.51 | 3.4930 (16) | 177 |

 Symmetry code: (i) $x, -y + \frac{1}{2}, z - \frac{1}{2}$.

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

AS gratefully acknowledges a research grant from Quaid-I-Azam University, Islamabad.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SG2231).

References

- Bruker (2002). SMART and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.
 Deguire, S. & Brisse, F. (1988). *Can. J. Chem.* **66**, 2545–2552.
 Gowda, B. T., Foro, S., Babitha, K. S. & Fuess, H. (2007). *Acta Cryst.* **E63**, o3867.
 Gray, R. W. & Whalley, W. B. (1971). *J. Chem. Soc. C*, pp. 3575–3577.
 Harris, J. P. & Mantle, P. G. (2001). *Phytochemistry*, **58**, 709–716.
 Saeed, A. & Rama, N. H. (1994). *J. Sci. I. R. Iran*, **5**, 173–175.
 Sheldrick, G. M. (2004). SADABS. University of Göttingen, Germany.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Simpson, T. J. (1978). *J. Chem. Soc. Chem. Commun.* pp. 627–628.

supplementary materials

Acta Cryst. (2008). E64, o821 [doi:10.1107/S1600536808008738]

Methyl 4-methylbenzoate

A. Saeed, H. Rafique and U. Flörke

Comment

The title ester is an important intermediate in the synthesis of a variety of natural products. These include the sclerotiorin group of fungal metabolites (Gray & Whalley, 1971), isochromans related to sclerotiorin pigments (Saeed & Rama, 1994) and isocoumarins like the 7-methylmellein (Harris & Mantle, 2001) and stellatin (Simpson, 1978).

Experimental

The title ester was prepared from commercial *p*-toluic acid according to standard procedure.

Refinement

Hydrogen atoms were located in difference syntheses, refined at idealized positions riding on the carbon or nitrogen atoms ($C-H = 0.88-0.99 \text{ \AA}$) with isotropic displacement parameters $U_{iso}(H) = 1.2U(C_{eq})$.

Figures

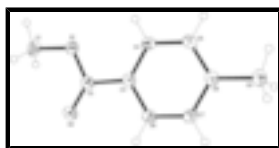


Fig. 1. Molecular structure of title compound. Displacement ellipsoids are drawn at the 50% probability level.



Fig. 2. Crystal packing viewed along [100] with intermolecular hydrogen bonding pattern indicated as dashed lines. H-atoms not involved in hydrogen bonding are omitted.

Methyl 4-methylbenzoate

Crystal data

$C_9H_{10}O_2$

$M_r = 150.17$

Monoclinic, $P2_1/c$

Hall symbol: $-P 2_1/c$

$a = 5.9134 (11) \text{ \AA}$

$b = 7.6048 (14) \text{ \AA}$

$c = 17.484 (3) \text{ \AA}$

$\beta = 97.783 (4)^\circ$

$V = 779.0 (2) \text{ \AA}^3$

$F_{000} = 320$

$D_x = 1.280 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 806 reflections

$\theta = 2.4-27.8^\circ$

$\mu = 0.09 \text{ mm}^{-1}$

$T = 120 (2) \text{ K}$

Block, colourless

$0.45 \times 0.43 \times 0.39 \text{ mm}$

supplementary materials

Z = 4

Data collection

| | |
|---|--|
| Bruker SMART APEX diffractometer | 1855 independent reflections |
| Radiation source: sealed tube | 1482 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{\text{int}} = 0.039$ |
| $T = 120(2)$ K | $\theta_{\text{max}} = 27.9^\circ$ |
| φ and ω scans | $\theta_{\text{min}} = 2.4^\circ$ |
| Absorption correction: multi-scan (SADABS; Sheldrick, 2004) | $h = -7 \rightarrow 7$ |
| $T_{\text{min}} = 0.961$, $T_{\text{max}} = 0.967$ | $k = -10 \rightarrow 9$ |
| 6617 measured reflections | $l = -23 \rightarrow 23$ |

Refinement

| | |
|--|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.042$ | H-atom parameters constrained |
| $wR(F^2) = 0.124$ | $w = 1/[\sigma^2(F_o^2) + (0.0752P)^2 + 0.0208P]$ |
| $S = 1.06$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 1855 reflections | $(\Delta/\sigma)_{\text{max}} < 0.001$ |
| 102 parameters | $\Delta\rho_{\text{max}} = 0.31 \text{ e } \text{\AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | $\Delta\rho_{\text{min}} = -0.20 \text{ e } \text{\AA}^{-3}$ |
| | Extinction correction: none |

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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|-------------|----------------------------------|
| O1 | 0.39091 (14) | 0.28701 (11) | 0.44793 (5) | 0.0280 (2) |
| O2 | 0.68910 (15) | 0.15425 (13) | 0.51751 (5) | 0.0325 (3) |
| C1 | 0.2956 (2) | 0.31740 (17) | 0.51874 (7) | 0.0320 (3) |
| H1A | 0.2793 | 0.2050 | 0.5448 | 0.048* |
| H1B | 0.1456 | 0.3732 | 0.5068 | 0.048* |

| | | | | |
|-----|--------------|--------------|-------------|------------|
| H1C | 0.3974 | 0.3946 | 0.5526 | 0.048* |
| C2 | 0.59091 (19) | 0.20144 (15) | 0.45593 (6) | 0.0234 (3) |
| C3 | 0.67753 (18) | 0.17434 (15) | 0.38071 (6) | 0.0223 (3) |
| C4 | 0.55841 (19) | 0.23124 (15) | 0.31083 (7) | 0.0247 (3) |
| H4A | 0.4154 | 0.2888 | 0.3098 | 0.030* |
| C5 | 0.6496 (2) | 0.20350 (15) | 0.24261 (7) | 0.0262 (3) |
| H5A | 0.5675 | 0.2426 | 0.1952 | 0.031* |
| C6 | 0.8588 (2) | 0.11953 (15) | 0.24239 (7) | 0.0244 (3) |
| C7 | 0.97615 (19) | 0.06389 (15) | 0.31291 (7) | 0.0253 (3) |
| H7A | 1.1195 | 0.0068 | 0.3140 | 0.030* |
| C8 | 0.88716 (19) | 0.09050 (15) | 0.38126 (7) | 0.0242 (3) |
| H8A | 0.9693 | 0.0515 | 0.4287 | 0.029* |
| C9 | 0.9593 (2) | 0.08897 (17) | 0.16858 (7) | 0.0312 (3) |
| H9A | 1.1213 | 0.1213 | 0.1764 | 0.047* |
| H9B | 0.8782 | 0.1613 | 0.1273 | 0.047* |
| H9C | 0.9438 | -0.0355 | 0.1542 | 0.047* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|----|------------|------------|------------|-------------|-------------|-------------|
| O1 | 0.0268 (5) | 0.0322 (5) | 0.0260 (4) | 0.0042 (3) | 0.0071 (3) | 0.0018 (3) |
| O2 | 0.0343 (5) | 0.0389 (5) | 0.0234 (5) | 0.0042 (4) | 0.0004 (4) | 0.0016 (3) |
| C1 | 0.0340 (7) | 0.0342 (7) | 0.0299 (7) | 0.0024 (5) | 0.0121 (5) | -0.0009 (5) |
| C2 | 0.0249 (6) | 0.0201 (6) | 0.0248 (6) | -0.0033 (4) | 0.0022 (5) | 0.0003 (4) |
| C3 | 0.0236 (6) | 0.0203 (6) | 0.0232 (6) | -0.0031 (4) | 0.0031 (4) | 0.0006 (4) |
| C4 | 0.0214 (5) | 0.0252 (6) | 0.0271 (6) | 0.0008 (4) | 0.0022 (4) | 0.0024 (4) |
| C5 | 0.0271 (6) | 0.0281 (6) | 0.0222 (6) | -0.0019 (5) | -0.0006 (5) | 0.0029 (4) |
| C6 | 0.0275 (6) | 0.0210 (6) | 0.0251 (6) | -0.0058 (4) | 0.0049 (4) | -0.0010 (4) |
| C7 | 0.0231 (6) | 0.0215 (6) | 0.0316 (6) | 0.0008 (4) | 0.0042 (5) | 0.0002 (4) |
| C8 | 0.0245 (6) | 0.0221 (6) | 0.0251 (6) | -0.0017 (4) | 0.0000 (4) | 0.0032 (4) |
| C9 | 0.0355 (7) | 0.0320 (7) | 0.0271 (6) | -0.0005 (5) | 0.0079 (5) | -0.0018 (5) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|-----------|-------------|-----------|-------------|
| O1—C2 | 1.3405 (14) | C5—C6 | 1.3927 (17) |
| O1—C1 | 1.4468 (14) | C5—H5A | 0.9500 |
| O2—C2 | 1.2065 (14) | C6—C7 | 1.3962 (17) |
| C1—H1A | 0.9800 | C6—C9 | 1.5101 (16) |
| C1—H1B | 0.9800 | C7—C8 | 1.3843 (16) |
| C1—H1C | 0.9800 | C7—H7A | 0.9500 |
| C2—C3 | 1.4890 (16) | C8—H8A | 0.9500 |
| C3—C8 | 1.3929 (16) | C9—H9A | 0.9800 |
| C3—C4 | 1.3940 (16) | C9—H9B | 0.9800 |
| C4—C5 | 1.3899 (16) | C9—H9C | 0.9800 |
| C4—H4A | 0.9500 | | |
| C2—O1—C1 | 115.38 (9) | C4—C5—H5A | 119.3 |
| O1—C1—H1A | 109.5 | C6—C5—H5A | 119.3 |
| O1—C1—H1B | 109.5 | C5—C6—C7 | 118.16 (10) |

supplementary materials

| | | | |
|-------------|--------------|-------------|--------------|
| H1A—C1—H1B | 109.5 | C5—C6—C9 | 121.71 (11) |
| O1—C1—H1C | 109.5 | C7—C6—C9 | 120.13 (11) |
| H1A—C1—H1C | 109.5 | C8—C7—C6 | 121.10 (10) |
| H1B—C1—H1C | 109.5 | C8—C7—H7A | 119.5 |
| O2—C2—O1 | 123.28 (10) | C6—C7—H7A | 119.5 |
| O2—C2—C3 | 124.43 (11) | C7—C8—C3 | 120.20 (10) |
| O1—C2—C3 | 112.28 (9) | C7—C8—H8A | 119.9 |
| C8—C3—C4 | 119.46 (10) | C3—C8—H8A | 119.9 |
| C8—C3—C2 | 118.00 (10) | C6—C9—H9A | 109.5 |
| C4—C3—C2 | 122.54 (10) | C6—C9—H9B | 109.5 |
| C5—C4—C3 | 119.76 (11) | H9A—C9—H9B | 109.5 |
| C5—C4—H4A | 120.1 | C6—C9—H9C | 109.5 |
| C3—C4—H4A | 120.1 | H9A—C9—H9C | 109.5 |
| C4—C5—C6 | 121.33 (10) | H9B—C9—H9C | 109.5 |
| C1—O1—C2—O2 | -1.07 (16) | C3—C4—C5—C6 | 0.00 (17) |
| C1—O1—C2—C3 | 179.72 (9) | C4—C5—C6—C7 | -0.20 (17) |
| O2—C2—C3—C8 | -0.70 (18) | C4—C5—C6—C9 | -179.94 (10) |
| O1—C2—C3—C8 | 178.50 (10) | C5—C6—C7—C8 | 0.28 (17) |
| O2—C2—C3—C4 | -179.94 (11) | C9—C6—C7—C8 | -179.98 (10) |
| O1—C2—C3—C4 | -0.74 (16) | C6—C7—C8—C3 | -0.16 (17) |
| C8—C3—C4—C5 | 0.12 (17) | C4—C3—C8—C7 | -0.05 (17) |
| C2—C3—C4—C5 | 179.36 (10) | C2—C3—C8—C7 | -179.32 (10) |

Hydrogen-bond geometry (\AA , $^\circ$)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---------------------------------|-------|-------------|-------------|---------------|
| C9—H9B \cdots O2 ⁱ | 0.98 | 2.51 | 3.4930 (16) | 177 |

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

Fig. 1

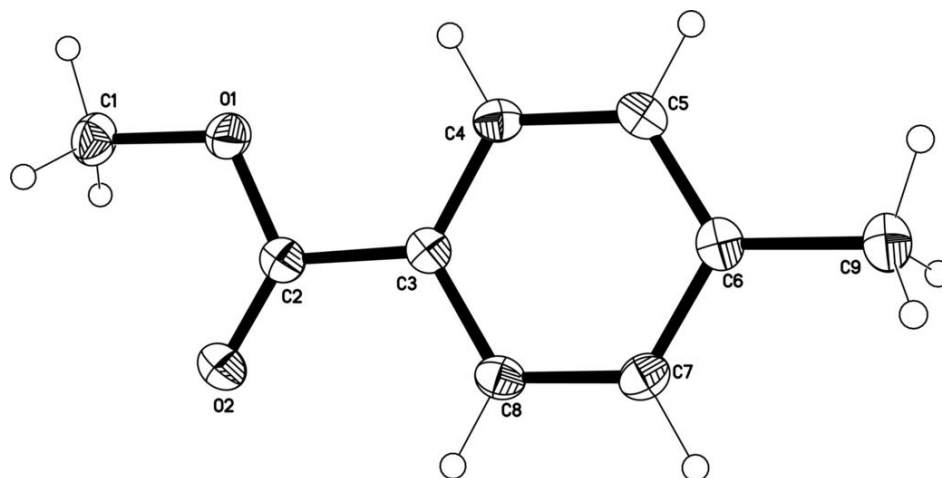


Fig. 2

