Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Methyl 3,4,5-trimethoxybenzoate

Aamer Saeed,[a] Rasheed Ahmad Khera[a] and Michael Bolte[b]*

[a]Department of Chemistry, Quaid-i-Azam University Islamabad, Pakistan, and [b]Institut für Anorganische Chemie, J. W. Goethe-Universität Frankfurt, Max-von-Laue-Strasse 7, 60438 Frankfurt/Main, Germany
Correspondence e-mail: aamersaeed@yahoo.com

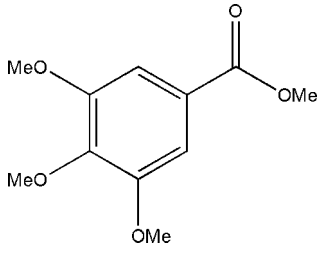
Received 18 September 2007; accepted 30 October 2007

Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.040; wR factor = 0.114; data-to-parameter ratio = 14.0.

In the molecule of the title compound, $C_{11}H_{14}O_5$, the methyl ester and the two methoxy residues in *meta* positions to the ester residue are almost coplanar, while the methoxy residue in the *para* position is almost perpendicular with respect to the aromatic ring.

Related literature

For related literature, see: Hellyer *et al.* (1966); Cooke & Rainbow (1974); Suzuki *et al.* (2004); Aboul-Enein & Eid (1979); Tanaka *et al.* (2001); Saeed & Ehsan (2005*a*, 2005*b*); Saeed (2007). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{11}H_{14}O_5$
M_r = 226.22
Monoclinic, $P2_1/c$
a = 17.6930 (15) Å
b = 4.5050 (5) Å
c = 14.6383 (12) Å
β = 106.545 (7)°
V = 1118.47 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 173 (2) K
0.42 × 0.37 × 0.21 mm

Data collection

STOE IPDS II diffractometer
Absorption correction: none
8229 measured reflections
2094 independent reflections
1796 reflections with $I > 2\sigma(I)$
R_{int} = 0.068

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.114
S = 1.06
2094 reflections
150 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2329).

References

Aboul-Enein, M. N. & Eid, A. I. (1979). *Acta Pharm. Suecia*, **16**, 267–270.
Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Cooke, R. G. & Rainbow, I. J. (1974). *Aust. J. Chem.* **27**, 2291–2292.
Hellyer, R., Lassak, E. V. & McKern, H. H. G. (1966). *Aust. J. Chem.* **19**, 1765–1767.
Saeed, A. (2007). *Synth. Commun.* **37**, 1485–1490.
Saeed, A. & Ehsan, S. (2005*a*). *J. Braz. Chem. Soc.* **16**, 739–742.
Saeed, A. & Ehsan, S. (2005*b*). *Chem. Heterocycl. C.* **11**, 1644–48.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Stoe & Cie (2002). *X-AREA* and *X-RED32*. Stoe & Cie, Darmstadt, Germany.
Suzuki, K., Tsuruga, T., Hiranuma, K. & Yamaura, M. (2004). *Synlett*, pp. 116–118.
Tanaka, T., Nonaka, G., Ishimatsu, M., Nishioka, I. & Kouno, I. (2001). *Chem. Pharm. Bull.* **49**, 486–487.

  doi:10.1107/S1600536807054384

supplementary materials

Acta Cryst. (2007). E**63**, o4582 [**doi:10.1107/S1600536807054384**]

Methyl 3,4,5-trimethoxybenzoate

A. Saeed, R. A. Khera and M. Bolte

Comment

The title ester is a constituent of the volatile oils of the genus eucalyptus (Hellyer *et al.*, 1966) and an important intermediate in the synthesis of a variety of natural products. These include lomandrone, the principal pigment of Lomandra hastilis (Cooke & Rainbow 1974), the aglycon of Actinoflavoside (Suzuki *et al.*, 2004), the cactus alkaloid, mescaline well known on account of its interesting effects on the psychic states of human subjects (Aboul-Enein & Eid 1979) and Cercidinin A, an ellagitannin isolated from the bark of Cercidiphyllum japonicum (Tanaka *et al.*, 2001). In addition a number of isocoumarins like kigelin (Saeed & Ehesan, 2005*a*), reticulol (Saeed & Ehesan, 2005*b*) and cAMP phosphodiesterase inhibitor 8-hydroxy-6,7-dimethoxy −3-hydroxymethylisocoumarin (Saeed, 2007). We report herein the crystal structure of the title compound, (I).

In the molecule of (I) (Fig. 1), the ligand bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The methyl acetate and the two O—Me residues in *meta* position to the ester residue are almost coplanar, while the O—Me residue in *para*-position is almost perpendicular with respect to the aromatic ring.

Experimental

Dimethyl sulfate (5.6 g, 45 mmol) and anhydrous potassium carbonate (6.1 g, 50 mmol) were added to a stirred solution of gallic acid (1.7 g, 10 mmol) in acetone (60 ml), and the mixture was refluxed under nitrogen for 6 h. The acetone was rotary evaporated, the residue was mixed with ice-water, and then extracted with ethyl acetate (3 *x* 50 ml). The extract was washed with $NaHCO_3$ (5%), dried ($MgSO_4$) and evaporated. The residue was recrystallized from aqueous methanol as needles (yield; 2.29 g, 92%, m.p. 355–356 K). The spectroscopic data was in agreement to that reported in literature (Aboul-Enein & Eid, 1979).

Refinement

H atoms were positioned geometrically, with C—H = 0.95 and 0.98 Å for aromatic and methyl H, and constrained to ride on their parent atoms, with $U_{iso}(H) = xU_{eq}(C)$, where $x = 1.2$ for aromatic H and $x = 1.5$ for methyl H atoms.

Figures

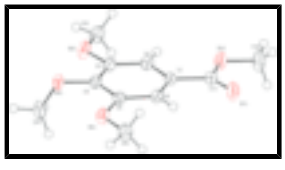


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

Methyl 3,4,5-trimethoxybenzoate

Crystal data

$C_{11}H_{14}O_5$	$F_{000} = 480$
$M_r = 226.22$	$D_x = 1.343$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 7934 reflections
$a = 17.6930$ (15) Å	$\theta = 3.5$–25.6º
$b = 4.5050$ (5) Å	$\mu = 0.11$ mm^{-1}
$c = 14.6383$ (12) Å	$T = 173$ (2) K
$\beta = 106.545$ (7)º	Block, colorless
$V = 1118.47$ (18) Å^3	0.42 × 0.37 × 0.21 mm
$Z = 4$	

Data collection

STOE IPDS-II diffractometer	1796 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.068$
Monochromator: graphite	$\theta_{max} = 25.6$º
$T = 173$(2) K	$\theta_{min} = 3.6$º
ω scans	$h = -21 \rightarrow 21$
Absorption correction: none	$k = -4 \rightarrow 5$
8229 measured reflections	$l = -15 \rightarrow 17$
2094 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.040$	$w = 1/[\sigma^2(F_o^2) + (0.0714P)^2 + 0.0891P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.114$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.06$	$\Delta\rho_{max} = 0.29$ e Å^{-3}
2094 reflections	$\Delta\rho_{min} = -0.20$ e Å^{-3}
150 parameters	Extinction correction: SHELXL, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.035 (5)
Secondary atom site location: difference Fourier map	

Special details

Experimental. ;

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.69462 (7)	0.3640 (3)	0.37200 (9)	0.0242 (3)
C2	0.69852 (7)	0.5101 (3)	0.28963 (9)	0.0257 (3)
H2	0.6538	0.6118	0.2510	0.031*
C3	0.76894 (7)	0.5054 (3)	0.26446 (9)	0.0253 (3)
C4	0.83503 (7)	0.3570 (3)	0.32254 (9)	0.0231 (3)
C5	0.83048 (7)	0.2175 (3)	0.40627 (9)	0.0233 (3)
C6	0.75980 (7)	0.2157 (3)	0.43092 (9)	0.0246 (3)
H6	0.7561	0.1157	0.4866	0.030*
C11	0.62009 (7)	0.3662 (3)	0.40168 (9)	0.0277 (3)
O11	0.61194 (5)	0.2474 (3)	0.47227 (7)	0.0393 (3)
O12	0.56223 (5)	0.5192 (3)	0.34019 (7)	0.0395 (3)
C12	0.48913 (8)	0.5425 (5)	0.36571 (12)	0.0513 (5)
H12A	0.4968	0.6739	0.4209	0.077*
H12B	0.4732	0.3452	0.3816	0.077*
H12C	0.4480	0.6241	0.3118	0.077*
O31	0.77982 (5)	0.6370 (2)	0.18506 (7)	0.0333 (3)
C31	0.71604 (8)	0.8117 (3)	0.12770 (10)	0.0335 (3)
H31A	0.7012	0.9633	0.1674	0.050*
H31B	0.6707	0.6830	0.0999	0.050*
H31C	0.7326	0.9081	0.0766	0.050*
O41	0.90383 (5)	0.34196 (19)	0.29594 (7)	0.0259 (3)
C41	0.95168 (8)	0.6036 (3)	0.32068 (12)	0.0336 (3)
H41A	0.9677	0.6291	0.3900	0.050*
H41B	0.9213	0.7772	0.2905	0.050*
H41C	0.9987	0.5830	0.2984	0.050*
O51	0.89959 (5)	0.0934 (2)	0.46006 (7)	0.0287 (3)
C51	0.89795 (7)	−0.0633 (3)	0.54457 (10)	0.0322 (3)
H51A	0.8620	−0.2330	0.5273	0.048*
H51B	0.8796	0.0701	0.5867	0.048*
H51C	0.9511	−0.1342	0.5775	0.048*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0216 (6)	0.0282 (7)	0.0245 (7)	−0.0041 (5)	0.0092 (5)	−0.0066 (5)
C2	0.0232 (6)	0.0300 (7)	0.0244 (7)	0.0002 (5)	0.0075 (5)	−0.0024 (5)
C3	0.0288 (6)	0.0256 (7)	0.0246 (7)	−0.0014 (5)	0.0125 (5)	−0.0023 (5)
C4	0.0244 (6)	0.0211 (6)	0.0278 (7)	−0.0008 (4)	0.0137 (5)	−0.0047 (5)
C5	0.0245 (6)	0.0211 (6)	0.0257 (7)	−0.0008 (5)	0.0096 (5)	−0.0044 (5)
C6	0.0271 (6)	0.0260 (7)	0.0231 (6)	−0.0035 (5)	0.0109 (5)	−0.0027 (5)
C11	0.0229 (6)	0.0367 (8)	0.0241 (7)	−0.0028 (5)	0.0076 (5)	−0.0039 (5)
O11	0.0280 (5)	0.0589 (7)	0.0355 (6)	0.0020 (4)	0.0162 (4)	0.0110 (5)
O12	0.0223 (5)	0.0683 (8)	0.0305 (5)	0.0077 (4)	0.0119 (4)	0.0096 (5)
C12	0.0246 (6)	0.0905 (14)	0.0431 (9)	0.0153 (7)	0.0166 (6)	0.0177 (9)
O31	0.0325 (5)	0.0423 (6)	0.0304 (5)	0.0080 (4)	0.0173 (4)	0.0112 (4)
C31	0.0355 (7)	0.0372 (8)	0.0299 (7)	0.0059 (6)	0.0127 (6)	0.0062 (6)
O41	0.0261 (4)	0.0219 (5)	0.0362 (5)	0.0001 (3)	0.0191 (4)	−0.0029 (4)
C41	0.0311 (6)	0.0245 (7)	0.0531 (9)	−0.0036 (5)	0.0245 (6)	−0.0031 (6)
O51	0.0239 (4)	0.0346 (5)	0.0301 (5)	0.0054 (4)	0.0118 (4)	0.0057 (4)
C51	0.0300 (6)	0.0367 (8)	0.0311 (7)	0.0013 (5)	0.0105 (5)	0.0070 (6)

Geometric parameters (Å, °)

C1—C2	1.3925 (19)	C12—H12A	0.9800
C1—C6	1.3978 (18)	C12—H12B	0.9800
C1—C11	1.5013 (15)	C12—H12C	0.9800
C2—C3	1.3964 (17)	O31—C31	1.4345 (16)
C2—H2	0.9500	C31—H31A	0.9800
C3—O31	1.3663 (16)	C31—H31B	0.9800
C3—C4	1.4039 (18)	C31—H31C	0.9800
C4—O41	1.3813 (13)	O41—C41	1.4362 (16)
C4—C5	1.4001 (19)	C41—H41A	0.9800
C5—O51	1.3710 (15)	C41—H41B	0.9800
C5—C6	1.3967 (15)	C41—H41C	0.9800
C6—H6	0.9500	O51—C51	1.4317 (16)
C11—O11	1.2082 (17)	C51—H51A	0.9800
C11—O12	1.3448 (16)	C51—H51B	0.9800
O12—C12	1.4485 (15)	C51—H51C	0.9800
C2—C1—C6	121.64 (11)	O12—C12—H12C	109.5
C2—C1—C11	120.77 (11)	H12A—C12—H12C	109.5
C6—C1—C11	117.58 (11)	H12B—C12—H12C	109.5
C1—C2—C3	119.30 (12)	C3—O31—C31	117.27 (9)
C1—C2—H2	120.3	O31—C31—H31A	109.5
C3—C2—H2	120.3	O31—C31—H31B	109.5
O31—C3—C2	124.78 (11)	H31A—C31—H31B	109.5
O31—C3—C4	115.28 (10)	O31—C31—H31C	109.5
C2—C3—C4	119.94 (12)	H31A—C31—H31C	109.5
O41—C4—C5	119.98 (11)	H31B—C31—H31C	109.5

O41—C4—C3	120.10 (11)	C4—O41—C41	113.09 (9)
C5—C4—C3	119.89 (11)	O41—C41—H41A	109.5
O51—C5—C6	124.54 (11)	O41—C41—H41B	109.5
O51—C5—C4	114.94 (10)	H41A—C41—H41B	109.5
C6—C5—C4	120.51 (11)	O41—C41—H41C	109.5
C5—C6—C1	118.68 (12)	H41A—C41—H41C	109.5
C5—C6—H6	120.7	H41B—C41—H41C	109.5
C1—C6—H6	120.7	C5—O51—C51	117.59 (9)
O11—C11—O12	123.08 (11)	O51—C51—H51A	109.5
O11—C11—C1	124.97 (12)	O51—C51—H51B	109.5
O12—C11—C1	111.95 (11)	H51A—C51—H51B	109.5
C11—O12—C12	115.46 (11)	O51—C51—H51C	109.5
O12—C12—H12A	109.5	H51A—C51—H51C	109.5
O12—C12—H12B	109.5	H51B—C51—H51C	109.5
H12A—C12—H12B	109.5		

C6—C1—C2—C3	0.84 (19)	C2—C1—C6—C5	0.49 (19)
C11—C1—C2—C3	179.53 (11)	C11—C1—C6—C5	−178.24 (11)
C1—C2—C3—O31	179.20 (12)	C2—C1—C11—O11	−179.39 (13)
C1—C2—C3—C4	−0.63 (19)	C6—C1—C11—O11	−0.6 (2)
O31—C3—C4—O41	−2.40 (17)	C2—C1—C11—O12	0.29 (17)
C2—C3—C4—O41	177.45 (11)	C6—C1—C11—O12	179.03 (12)
O31—C3—C4—C5	179.26 (11)	O11—C11—O12—C12	2.1 (2)
C2—C3—C4—C5	−0.89 (19)	C1—C11—O12—C12	−177.58 (13)
O41—C4—C5—O51	5.05 (17)	C2—C3—O31—C31	5.12 (19)
C3—C4—C5—O51	−176.61 (11)	C4—C3—O31—C31	−175.04 (11)
O41—C4—C5—C6	−176.09 (10)	C5—C4—O41—C41	−99.28 (14)
C3—C4—C5—C6	2.25 (18)	C3—C4—O41—C41	82.39 (14)
O51—C5—C6—C1	176.71 (11)	C6—C5—O51—C51	3.72 (18)
C4—C5—C6—C1	−2.03 (18)	C4—C5—O51—C51	−177.48 (11)

Fig. 1