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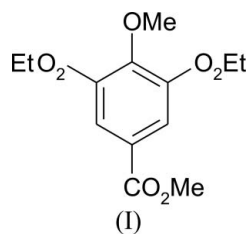
Key indicators

Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{C}-\text{C}) = 0.007 \text{ \AA}$
R factor = 0.073
wR factor = 0.208
Data-to-parameter ratio = 13.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

Methyl 3,5-diacetoxy-4-methoxybenzoate

The title compound, $\text{C}_{13}\text{H}_{14}\text{O}_7$, is derived from methyl gallate, a protective biological antioxidant. It crystallizes with two independent molecules in the asymmetric unit.Received 30 May 2006
Accepted 17 November 2006

Comment

Methyl gallate (methyl 3,4,5-trihydroxybenzoate) can be isolated from *Acer truncatum* Bunge and it is considered to be a major pharmacological antioxidant (Whang *et al.*, 2005). It is widely distributed and is used to treat coronary arteriosclerosis, cerebrovascular diseases and angina pectoris (Liu, 2003). In order to investigate a similar biological compound derived from methyl gallate, the title compound, (I), was prepared and its crystal structure is reported here.

The asymmetric unit consists of two independent molecules of (I) (Fig. 1). In both molecules, the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The C1–C6 and C14–C19 benzene rings are planar, with r.m.s. deviations of 0.0043 and 0.0085 Å, respectively. The two independent molecules in the asymmetric unit are very similar in conformation, but show significant small differences in the magnitudes of their torsion angles. For example, the torsion angles C9–O5–C2–C3 [–73.6 (5)°] and C11–O7–C6–C1 [–77.0 (5)°] are comparable to those for C24–O11–C19–C18 [88.7 (5)°] and C22–O13–C15–C14 [82.9 (5)°].

In the crystal packing, the molecules are linked by several weak intermolecular C–H...O interactions, shown as dashed lines in Fig. 2.

Experimental

All the solvents and reagents were commercial and purified according to standard laboratory techniques. To a solution of methyl 3,4,5-triacetoxybenzoate (1 mmol, 310 mg) in DMF (10 ml) was added K_2CO_3 (3 mmol, 414 mg) and CH_3I (2 mmol, 0.13 ml). After stirring for 3 h at 413 K, the inorganic salt in the resulting mixture was removed by filtration, and the filtrate was diluted with EtOAc. The organic phase was washed with distilled water, dried over Na_2SO_4 and evaporated to give 279 mg (90% yield) of methyl 3,5-diacetoxy-4-

methoxybenzoate after recrystallization from EtOAc–heptane (3:1) (m.p. 356 K).

Crystal data

$C_{13}H_{14}O_7$
 $M_r = 282.24$
 Monoclinic, $P2_1/c$
 $a = 9.0530$ (18) Å
 $b = 25.769$ (5) Å
 $c = 11.864$ (2) Å
 $\beta = 92.57$ (3)°
 $V = 2764.9$ (10) Å³

$Z = 8$
 $D_x = 1.356$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.11$ mm⁻¹
 $T = 293$ (2) K
 Prism, colorless
 $0.30 \times 0.20 \times 0.15$ mm

Data collection

Bruker APEX area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{min} = 0.967$, $T_{max} = 0.984$

5484 measured reflections
 5146 independent reflections
 2421 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.164$
 $\theta_{max} = 25.5^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.073$
 $wR(F^2) = 0.208$
 $S = 1.05$
 5146 reflections
 369 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0788P)^2 + 0.5721P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.27$ e Å⁻³
 $\Delta\rho_{min} = -0.31$ e Å⁻³

The residual factor for equivalent reflections is large ($R_{int} = 0.16$) because the data are very weak. H atoms were included in calculated positions and refined using the riding-model approximation, with C–H(aromatic) = 0.93 Å and C–H(methyl) = 0.96 Å, and with $U_{iso}(H) = 1.2U_{eq}(C)$ for aromatic and $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms.

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

The support by the Modern Analytical Center at Nanjing University is gratefully acknowledged.

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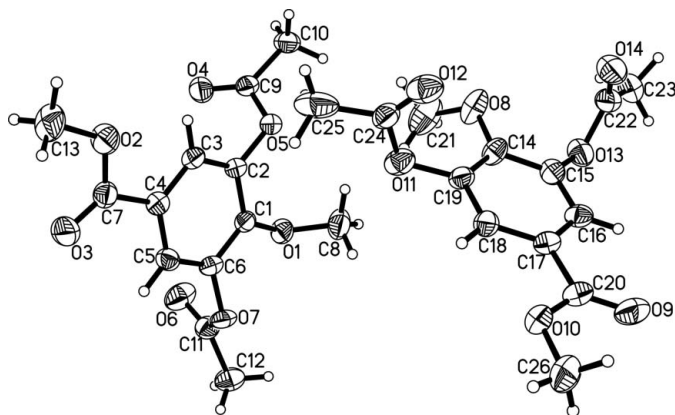


Figure 1 The asymmetric unit of the title compound, (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

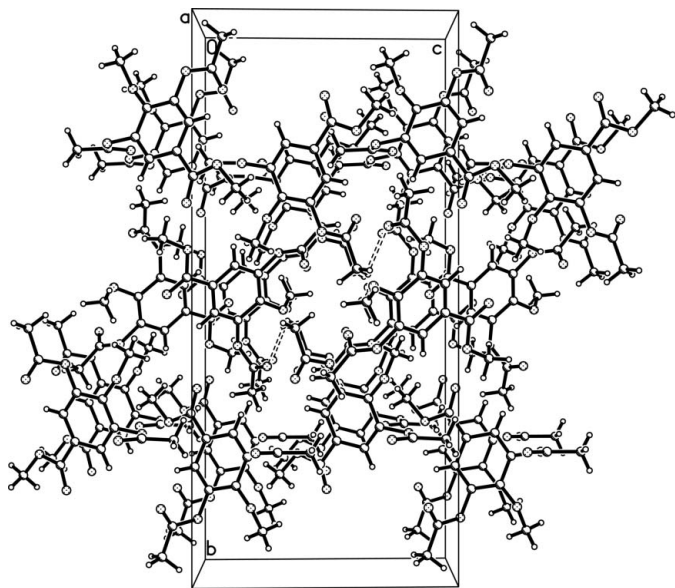


Figure 2 The crystal packing of (I), viewed along the a axis. Weak intermolecular C–H...O interactions are shown as dashed lines.

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