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Ethyl 3-(4-geranyloxy-3-methoxyphenyl)-2-propenoate

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The title compound, $C_{22}H_{30}O_4$, is known as a chemopreventive agent against colon tumor development. The molecular structure is nearly planar, showing a *trans* arrangement with respect to the C=C bond of the phenylpropenoate moiety.

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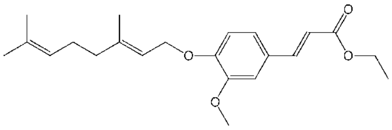
Key indicators

Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.003 Å
R factor = 0.059
wR factor = 0.120
Data-to-parameter ratio = 10.5

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

Comment

The title compound, (I), is known as a chemopreventive agent against colon tumor development (Tsuda *et al.*, 1999). It was prepared from ferulic acid, which was easily prepared in large quantities from the oily component of rice bran (Taniguchi *et al.*, 1996).



EGMP

The molecular structure of (I) is shown in Fig. 1, confirming the *trans* arrangement with respect to the C7=C8 double bond, similar to ferulic acid (Nethaji *et al.*, 1988). The orientations of the phenyl ring and the geranyl group are defined by the torsion angles in Table 1. The molecule is nearly planar. The molecules in the crystal lattice are in a conventional herring-bone-type arrangement (Fig. 2). No close contacts were found that indicated any relevant intermolecular interaction.

Experimental

To a solution of 1.0 g (4.5 mmol) of ethyl ferulate, ethyl ester of ferulic acid, 1.2 g (4.6 mmol) of triphenylphosphine and 0.69 g (4.5 mmol) of geraniol in 10 ml anhydrous tetrahydrofuran was added 0.93 g (4.6 mmol) diisopropyl azodicarboxylate. The mixture was stirred for 5 min at room temperature and the solvent then removed under reduced pressure. After the residue was extracted with 30 ml of hexane, the hexane was removed under reduced pressure. Recrystallization from methanol gave the pure title compound (I) (1.37 g, 85%). Crystals of (I) were obtained by slow evaporation of an ethanol solution at 277–279 K.

Crystal data

$C_{22}H_{30}O_4$
$M_r = 358.48$
Triclinic, $P\overline{1}$
$a = 11.094$ (1) Å
$b = 11.943$ (1) Å
$c = 9.3399$ (9) Å
$\alpha = 110.864$ (5)°
$\beta = 113.510$ (5)°
$\gamma = 68.358$ (4)°
$V = 1022.5$ (2) Å^3
$Z = 2$
$D_x = 1.164$ Mg m^{-3}
Cu Kα radiation
Cell parameters from 2787 reflections
$\theta = 4.1$–67.5°
$\mu = 0.63$ mm^{-1}
$T = 193.2$ K
Prism, colorless
0.60 × 0.58 × 0.30 mm

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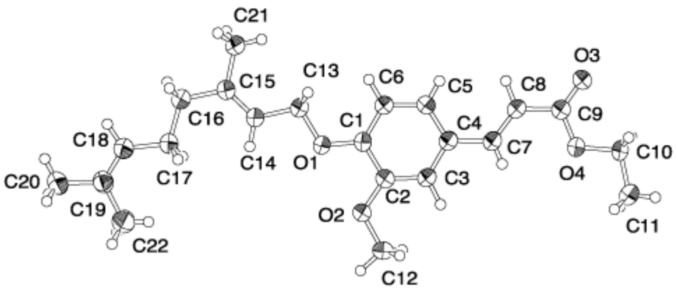

Figure 1
The molecular structure of (I) showing 50% probability displacement ellipsoids.

Data collection

Rigaku R-AXIS-RAPID image-plate diffractometer
ω scans
Absorption correction: multi-scan (Higashi, 1995)
$T_{\min} = 0.587$, $T_{\max} = 0.828$
5896 measured reflections
3429 independent reflections
3094 reflections with $F^2 > 2.0\sigma(F^2)$
$R_{\rm int} = 0.028$
$\theta_{\max} = 68.2°$
$h = -13 \rightarrow 13$
$k = -14 \rightarrow 14$
$l = -11 \rightarrow 11$

Refinement

Refinement on F
$R = 0.059$
$wR = 0.121$
$S = 1.65$
3429 reflections
326 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o) + 0.00403|F_o|^2]$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.51$ e Å^{-3}
$\Delta\rho_{\min} = -0.28$ e Å^{-3}
Extinction correction: Zachariasen (1967) type 2 Gaussian isotropic
Extinction coefficient: 0.39 (6)

Table 1
Selected torsion angles (°).

O(1)—C(13)—C(14)—C(15)	180.0 (1)
O(3)—C(9)—O(4)—C(10)	−1.1 (3)
O(3)—C(9)—C(8)—C(7)	−176.8 (2)
O(4)—C(9)—C(8)—C(7)	3.2 (3)
C(1)—O(1)—C(13)—C(14)	−178.2 (1)
C(1)—C(2)—O(2)—C(12)	177.6 (2)
C(2)—C(1)—O(1)—C(13)	179.4 (1)
C(3)—C(2)—O(2)—C(12)	−3.6 (3)
C(3)—C(4)—C(7)—C(8)	174.6 (2)
C(4)—C(7)—C(8)—C(9)	178.4 (2)
C(5)—C(4)—C(7)—C(8)	−5.9 (3)
C(6)—C(1)—O(1)—C(13)	0.6 (2)
C(8)—C(9)—O(4)—C(10)	178.9 (1)
C(9)—O(4)—C(10)—C(11)	−177.5 (2)
C(13)—C(14)—C(15)—C(16)	178.3 (1)
C(13)—C(14)—C(15)—C(21)	−0.1 (3)
C(14)—C(15)—C(16)—C(17)	−2.3 (2)
C(15)—C(16)—C(17)—C(18)	−177.1 (1)
C(16)—C(17)—C(18)—C(19)	170.5 (2)
C(17)—C(16)—C(15)—C(21)	176.1 (1)
C(17)—C(18)—C(19)—C(20)	179.6 (2)
C(17)—C(18)—C(19)—C(22)	0.8 (3)

The measured fraction ($\theta_{\max} = 68.2°$) of 0.917 is relatively low, which may be ascribed to either a short exposure time of the image plate or the instrument having only one rotation axis, leaving some parts of reciprocal space inaccessible.

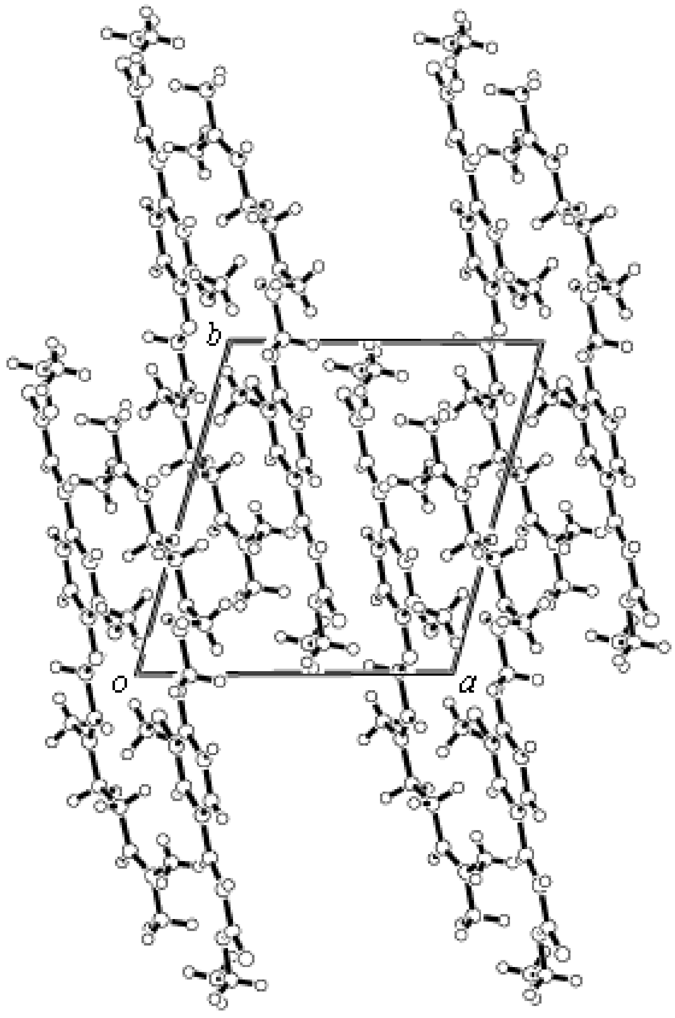

Figure 2
Projection down the *c* axis of the unit cell of (I)

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1999); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *TEXSAN*; software used to prepare material for publication: *TEXSAN*.

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