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Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.039
 wR factor = 0.101
Data-to-parameter ratio = 14.1For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.4-(*E*)-But-1-enyl-2,6-dimethoxyphenyl
nicotinate

In the title compound, 4-(*E*)-but-1-enyl-2,6-dimethoxyphenyl pyridine-3-carboxylate, $\text{C}_{18}\text{H}_{19}\text{NO}_4$, the butenyl substituent is in the *trans* configuration. The molecules are linked by weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, leading to the formation of a chain extending through the whole crystal structure.

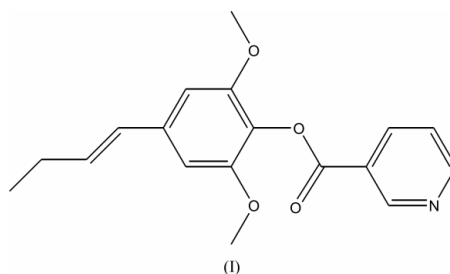
Received 29 April 2003

Accepted 14 May 2003

Online 23 May 2003

Comment

α -Asarone is a principal active ingredient of the bark extract of *Guatteria gaumeri* (Enriquez & Chàvez, 1980) and *Acorus calamus* (Gracza & Spaich, 1978). These extracts are used to prevent elevation of plasma lipids. It has been found, in earlier studies of α -asarone derivatives, that some of them could be effective hypolipidaemic agents (Popławski *et al.*, 2000; Labarrios *et al.*, 1999). α -Asarones have already been investigated by X-ray crystallography (Wolska *et al.*, 1998). In those studies, the occurrence of weak intra- and intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonding was reported. In this paper, the crystal and molecular structure of a new α -asarone derivative, (I), is presented.



The molecule of (I) consists of two aromatic rings, *viz.* a pyridine ring present as a nicotinic acid fragment and a benzene ring forming the basic α -asarone moiety. The least-squares planes of these rings form a dihedral angle of $76.39(6)^\circ$. The carboxylic acid group is almost coplanar with the pyridine ring; the plane through atoms C3/C30/O30/O31 forms an angle of $6.28(7)^\circ$ with the plane of the pyridine ring. The atoms of the methoxy groups do not deviate significantly from the benzene-ring plane, the maximum deviation being $0.274(4)$ Å for atom C360. Such an arrangement of methoxy groups attached to the benzene ring is most advantageous energetically, as has been proved both experimentally and theoretically (Ferdeselel *et al.*, 2001). The butenyl group is in the *trans* configuration. The C34/C40/C50/C60 plane makes an angle of $3.9(2)^\circ$ with the plane of the benzene ring. Atom C70 deviates from the C34/C40/C50/C60 plane by $1.162(4)$ Å.

Detailed geometrical analysis suggests the presence of a weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bond (Table 2).

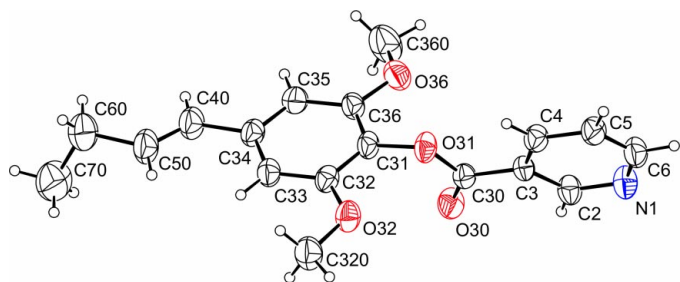


Figure 1
A view of the title compound, with the atom-labelling scheme. Displacements ellipsoids are drawn at the 40% probability level.

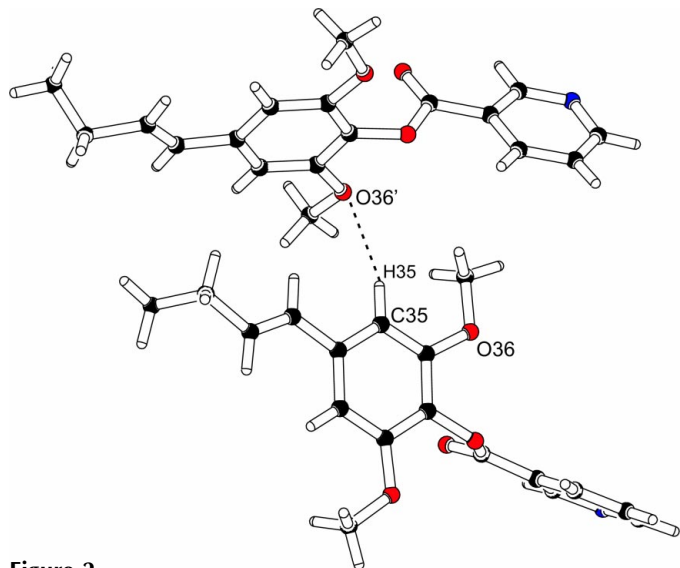


Figure 2
Intermolecular hydrogen bonding in the crystal structure of the title compound.

Finally, the molecules link together, forming a chain whose topological motif corresponds to the first-order graph-set descriptor $C(4)$ (Bernstein *et al.*, 1995).

Experimental

Details of the synthesis will be published elsewhere (Grabowski *et al.*, 2003). Crystals were obtained by slow evaporation of an ethanol/hexane solution at room temperature.

Crystal data

$C_{18}H_{19}NO_4$
 $M_r = 313.34$
Monoclinic, $P2_1/c$
 $a = 9.105$ (2) Å
 $b = 22.729$ (3) Å
 $c = 8.157$ (3) Å
 $\beta = 98.47$ (2)°
 $V = 1669.7$ (8) Å³
 $Z = 4$

$D_x = 1.247$ Mg m⁻³
Cu $K\alpha$ radiation
Cell parameters from 20 reflections
 $\theta = 9.9$ – 13.2 °
 $\mu = 0.72$ mm⁻¹
 $T = 293$ (2) K
Needle, colourless
 $0.43 \times 0.15 \times 0.08$ mm

Data collection

Rigaku AFC-5S diffractometer
 ω scans
2927 measured reflections
2927 independent reflections
1259 reflections with $I > 2\sigma(I)$
 $\theta_{\max} = 67.5$ °

$h = -10 \rightarrow 10$
 $k = 0 \rightarrow 27$
 $l = 0 \rightarrow 9$
3 standard reflections every 150 reflections
intensity decay: <2%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.101$
 $S = 1.03$
2927 reflections
208 parameters
H-atom parameters constrained

$$w = \frac{\exp[-4(\sin\theta/\lambda)^2]}{(0.0485P)^2}$$

$$\text{where } P = 0.3333F_o^2 + 0.6667F_c^2$$

$$(\Delta/\sigma)_{\max} < 0.001$$

$$\Delta\rho_{\max} = 0.26 \text{ e \AA}^{-3}$$

$$\Delta\rho_{\min} = -0.14 \text{ e \AA}^{-3}$$

Table 1

Selected geometric parameters (Å, °).

O31—C30	1.348 (2)	O36—C36	1.356 (2)
O31—C31	1.399 (2)	O36—C360	1.407 (3)
O32—C32	1.361 (2)	C2—N1	1.330 (2)
O32—C320	1.413 (2)	N1—C6	1.336 (3)
O30—C30	1.199 (2)		
C30—O31—C31	117.81 (15)	C36—C31—O31	119.4 (2)
C32—O32—C320	118.58 (16)	C32—C31—O31	118.7 (2)
C36—O36—C360	117.53 (18)	O32—C32—C31	115.52 (18)
O36—C36—C31	116.13 (19)	O32—C32—C33	125.3 (2)
O36—C36—C35	125.2 (2)	O30—C30—O31	123.18 (17)
N1—C2—C3	124.6 (2)	O30—C30—C3	125.7 (2)
C2—N1—C6	116.1 (2)	O31—C30—C3	111.08 (17)
C360—O36—C36—C31	167.2 (2)	C320—O32—C32—C33	2.1 (3)
C360—O36—C36—C35	-13.9 (3)	C36—C31—C32—O32	179.06 (18)
C34—C35—C36—O36	-178.69 (19)	O31—C31—C32—O32	-6.2 (3)
O36—C36—C31—C32	179.86 (18)	O31—C31—C32—C33	173.48 (17)
O36—C36—C31—O31	5.2 (3)	C31—O31—C30—O30	-6.2 (3)
C35—C36—C31—O31	-173.83 (18)	C31—O31—C30—C3	172.84 (18)
C30—O31—C31—C36	-79.5 (2)	C2—C3—C30—O30	5.1 (3)
C30—O31—C31—C32	105.7 (2)	C4—C3—C30—O30	-177.2 (2)
C320—O32—C32—C31	-178.25 (19)	C40—C50—C60—C70	117.0 (4)

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C35-H35\cdots O36^i$	0.93	2.70	3.564 (3)	154

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

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å. For methoxy H atoms, $U_{\text{iso}} = 1.5U_{\text{eq}}(\text{C})$; for all other H atoms, $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1989); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1989); program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1998); software used to prepare material for publication: *PARST97* (Nardelli, 1995).

The author thanks the Rector of the University of Łódź for financial support (University Research Grants) and A. T. Dubis for a sample of the title compound.

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