Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-1-(2-Naphthyl)-3-(3,4,5-trimethoxyphenyl)-prop-2-en-1-one

Zhi-Ke Lu,[a]* Peng-Mian Huang[a] and Jiang-Fan Yu[b]

[a]School of Pharmaceuticals & Biotechnology, Tianjin University, Tianjin 300072, People's Republic of China, and [b]College of Resources and the Environment, Central South Forestry University, Changsha, 410004, People's Republic of China

Correspondence e-mail: lukz1886@yahoo.com.cn

Received 30 October 2006
Accepted 12 November 2006

Key indicators

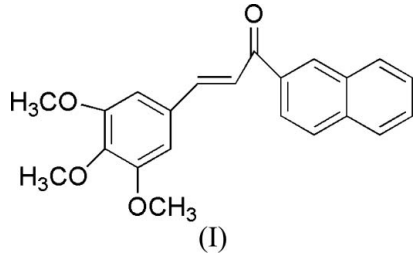
Single-crystal X-ray study
T = 113 K
Mean σ(C–C) = 0.003 Å
R factor = 0.066
wR factor = 0.146
Data-to-parameter ratio = 14.5

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

In the title compound, $C_{22}H_{20}O_4$, the naphthalene ring system makes a dihedral angle of 36.3 (5)° with the benzene ring. Neighboring molecules are linked *via* weak C—H···O hydrogen bonding.

Comment

(*E*)-1-(Naphthalen-2-yl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one, (I), is a valuable pharmaceutical intermediate (Jardine *et al.*, 1980; Issell *et al.*, 1984; Stahelin & Wartburg, 1989). We report here the crystal structure of (I).



(I)

The molecular structure of (I) is shown in Fig. 1. The dihedral angle between the naphthalene ring system and the benzene ring is 36.3 (5)°. Of the three methoxy groups, two are coplanar with the benzene ring, but the O3—C21 bond makes an angle of 55.75 (13)° with the C14-benzene ring plane, minimizing repulsion between the methoxy groups. Neighboring molecules are linked together *via* weak C—H···O hydrogen bonding (Table 1).

Experimental

A mixture of 3,4,5-trimethoxybenzaldehyde (5 mmol) and 2-(prop-1-en-2-yl)naphthalenecarbaldehyde (10 mmol) was dissolved in a water–ethanol solution (100 ml, *v*/*v* = 6:4). A solution of KOH (4.0 ml, 7.1 mmol) was added slowly with stirring at 323 K over 5 h. The resulting mixture was neutralized with 2 *N* HCl solution and extracted with CH_2Cl_2. The organic layer was washed with brine, dried with Na_2SO_4 and evaporated under vacuum to give (I). Compound (I) (40 mg) was dissolved in a mixture of ethyl acetate (5 ml) and petroleum ether (50 ml), and the solution was allowed to stand at room temperature for 16 d, yielding single crystals of (I).

Crystal data

$C_{22}H_{20}O_4$
M_r = 348.38
Orthorhombic, *Pbca*
a = 13.5903 (13) Å
b = 8.6378 (9) Å
c = 30.169 (3) Å
V = 3541.5 (6) Å^3
Z = 8
D_x = 1.307 Mg m^{-3}
Mo *Kα* radiation
μ = 0.09 mm^{-1}
T = 113 (2) K
Platelet, colorless
0.34 × 0.22 × 0.06 mm

Data collection

Rigaku Saturn-70 diffractometer
ω scans
Absorption correction: none
26883 measured reflections
3477 independent reflections
3229 reflections with $I > 2\sigma(I)$
$R_{int} = 0.073$
$\theta_{max} = 26.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.066$
$wR(F^2) = 0.146$
$S = 1.23$
3477 reflections
240 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0553P)^2 + 1.8606P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0066 (9)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12···O1^{i}	0.95	2.56	3.508 (3)	176
C22—H22B···O1^{i}	0.98	2.51	3.405 (3)	153

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

Methyl H atoms were placed in calculated positions, with C—H = 0.98 Å, and torsion angles were refined to fit the electron density, U_{iso}(H) = 1.5U_{eq}(C). Aromatic H were placed in calculated positions, with C—H = 0.93 Å, and refined in riding mode, with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *CrystalStructure* (Rigaku/MSC, 2005).

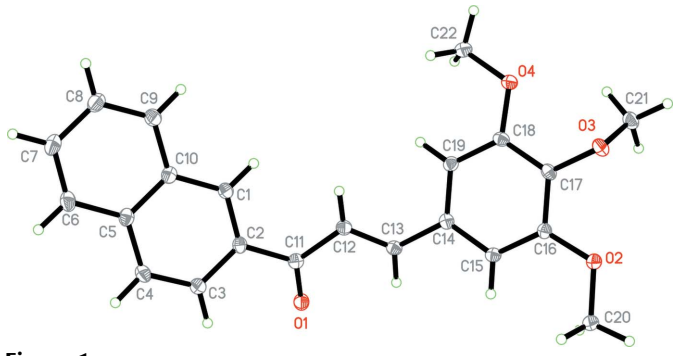


Figure 1
The molecular structure of (I) with 30% probability displacement ellipsoids (arbitrary spheres for H atoms).

The authors thank the Science Foundation of Tianjin University for financial support.

References

Bruker (1997). *SHELXTL*. Version 6.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Issell, B. F., Muggia, F. M. & Carter, S. K. (1984). Editors. *Etoposide (VP-16), Current Status and New Developments*, pp. 33–47. Orlando: Academic Press.
Jardine, I. (1980). *Podophyllotoxins*. In *Anticancer Agents Based on Natural Products Models*. Orlando: Academic Press.
Rigaku/MSC (2005). *CrystalClear* (Version 1.36) and *CrystalStructure* (Version 3.7.0). Rigaku/MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Stahelin, H. & Wartburg, A. V. (1989). *Progr. Drug. Res.* **33**, 169–266.