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1-(4-Aminophenyl)-3-(3,4-dimethoxyphenyl)-prop-2-en-1-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.088
wR factor = 0.217
Data-to-parameter ratio = 13.8

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

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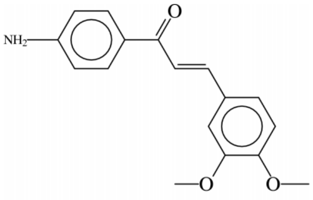
In the title molecule, $C_{17}H_{17}NO_3$, the configuration of the keto group with respect to the olefinic double bond is s-*cis*. The dihedral angle between the two aromatic rings is 9.5 (2)°. Both the H atoms of the amino group are involved in N—H···O hydrogen bonding to form molecular layers parallel to the (100) plane. The layers are stacked along the *b* axis and are interlinked by C—H···π and C—H···O interactions.

Comment

Chalcones and their heterocyclic analogues exhibit anti-inflammatory, antitumour (Shibata, 1994; Xia *et al.*, 2000), antibacterial, antifungal (Gafner *et al.*, 1996; Popova *et al.*, 2001), antituberculous, antiviral, antiprotozoal and gastro-protective (Opletalova & Sedivy, 1999) activities. The different bioactivities of a variety of chalcones have been reviewed by Dimmock *et al.* (1999). Hydroxychalcones can be readily reacted with acryloyl and methacryloyl chloride to obtain acrylate and methacrylate monomers, which are useful for the preparation of photo-cross-linkable polymers (Subramanian *et al.*, 2001; Balaji & Nanjundan, 2002). The structure determination of the title compound, (I), was undertaken as part of our study of chalcones.



(I)

In the title molecule, the keto group is in the s-*cis* configuration with respect to the olefinic double bond [C7—C8—C9—O1 = −4.0 (6)°; Fig. 1]. The methoxy groups are coplanar with the attached ring [C17—O3—C4—C5 = 0.1 (6)° and C16—O2—C3—C2 = −2.7 (6)°]. The molecule is slightly distorted from planarity, the dihedral angle between the two aromatic rings being 9.5 (2)°. The bond lengths and angles are comparable to those reported for related structures (Raj *et al.*, 1996, 1997; Jeyabharathi *et al.*, 2002; Ravishankar, Chinnakali, Nanjundan, Radhakrishnan *et al.*, 2003; Ravishankar, Chinnakali, Nanjundan, Jone Selvamalar *et al.*, 2003). The short H5···H8 (2.23 Å) and H8···H11 (2.11 Å) contacts result in the widening of the C6—C7—C8 [129.3 (4)°] and C9—C10—C11 [124.6 (3)°] angles, respectively, from 120°. The narrowing of the O2—C3—C4 [115.8 (3)°] and O3—C4—C3 [115.4 (3)°] angles and the resultant widening of the O2—C3—C2 [124.9 (4)°] and O3—C4—C5 [125.1 (4)°] angles from normal values are a result of the close H···H contacts (H2···H16B = 2.31 Å, H2···H16C = 2.27 Å, H5···H17B = 2.34 Å and H5···H17C = 2.23 Å). Similar features have been previously reported for related structures (Ravishankar, Chinnakali, Nanjundan, Radhakrishnan et al., 2003; Ravishankar, Chinnakali, Nanjundan, Jone Selvamalar *et al.*, 2003).

In the crystal structure, molecules translated by one unit along the b axis are linked by N1—H1A···O1^{i} hydrogen bonds to form chains (symmetry codes are given in Table 2). The adjacent symmetry-related chains are interlinked by N1—H1B···O2^{ii} hydrogen bonds to form molecular layers parallel to the (100) plane (Fig. 2). This layer structure is further stabilized by C—H···O interactions (Table 2), whose geometry agrees with those reported by Jeffrey (1997) and Desiraju & Steiner (1999). The N—H···O and C—H···O hydrogen bonds in a layer generate rings of graph set motifs $R_2^2(9)$ and $R_3^2(7)$ (Etter *et al.*, 1990). The layers are stacked alternately at distances of 3.501 (6) and 3.538 (6) Å along the b axis. Adjacent layers are linked by C—H···π and C—H···O interactions (Table 2). No significant $\pi\cdots\pi$ interaction is observed between the benzene rings of adjacent layers.

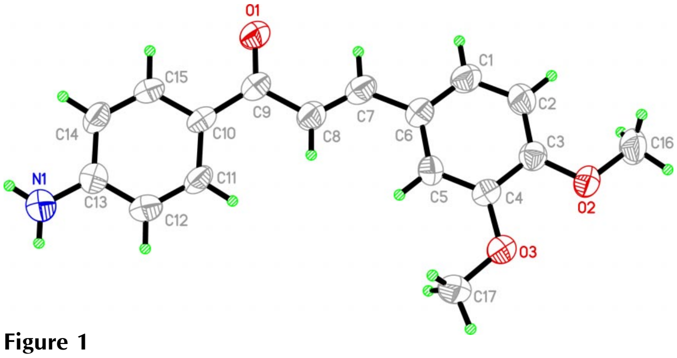


Figure 1
A view of the title molecule, with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

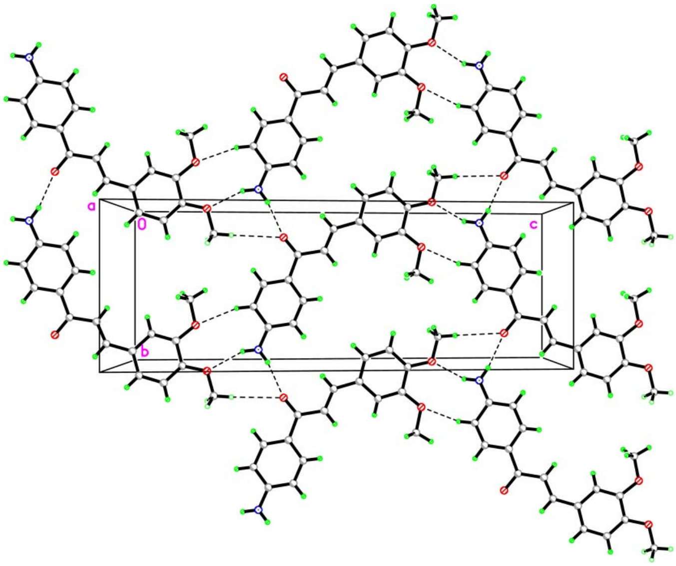


Figure 2
A view of the molecular layer, showing N—H···O hydrogen bonds and C—H···O interactions.

Experimental

The title compound was prepared by stirring an alcoholic solution of 3,4-dimethoxybenzaldehyde with 4-aminoacetophenone in the presence of 10% sodium hydroxide for 3 d at 273–283 K. The crude, filtered and washed product was recrystallized from a 1:1 ethylacetate/chloroform mixture.

Crystal data

$C_{17}H_{17}NO_3$
M_r = 283.32
Monoclinic, $C2/c$
a = 13.856 (2) Å
b = 8.8243 (13) Å
c = 24.775 (4) Å
β = 97.630 (3)°
V = 3002.4 (8) Å^3
Z = 8
D_x = 1.254 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2797 reflections
θ = 2.7–28.3°
μ = 0.09 mm^{-1}
T = 293 (2) K
Block, yellow
0.44 × 0.40 × 0.36 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
7214 measured reflections
2644 independent reflections
1787 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.034
$\theta_{\rm max}$ = 25.0°
$h = -15 \rightarrow 16$
$k = -10 \rightarrow 9$
$l = -29 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.088
$wR(F^2)$ = 0.217
S = 1.20
2644 reflections
192 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + 6.2455P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max}$ = 0.20 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.27 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C9	1.231 (4)	O3—C17	1.429 (5)
O2—C3	1.364 (4)	N1—C13	1.355 (5)
O2—C16	1.421 (5)	C7—C8	1.308 (5)
O3—C4	1.359 (4)		
O2—C3—C2	124.9 (4)	O3—C4—C3	115.4 (3)
O2—C3—C4	115.8 (3)	C8—C7—C6	129.3 (4)
O3—C4—C5	125.1 (4)	C11—C10—C9	124.6 (3)
C16—O2—C3—C2	−2.7 (6)	C7—C8—C9—O1	−4.0 (6)
C17—O3—C4—C5	0.1 (6)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.86	2.15	2.995 (5)	166
N1—H1B···O2^{ii}	0.86	2.40	3.176 (5)	151
C7—H7···O1	0.93	2.45	2.799 (5)	102
C15—H15···O1	0.93	2.47	2.780 (5)	100
C14—H14···O3^{ii}	0.93	2.63	3.490 (5)	154
C16—H16A···O1^{iii}	0.96	2.95	3.891 (5)	168
C16—H16A···O3^{iv}	0.96	2.98	3.518 (6)	117
C16—H16C···O1^{v}	0.96	2.87	3.758 (6)	155
C17—H17C···O1^{vi}	0.96	2.73	3.513 (6)	139
C17—H17B···$Cg1^{vii}$	0.96	2.96	3.739 (6)	139

Symmetry codes: (i) $x, 1+y, z$; (ii) $x, 1-y, z-\frac{1}{2}$; (iii) $x, -y, \frac{1}{2}+z$; (iv) $\frac{3}{2}-x, y-\frac{1}{2}, \frac{3}{2}-z$; (v) $1-x, -y, 1-z$; (vi) $\frac{3}{2}-x, \frac{1}{2}-y, 1-z$; (vii) $1-x, 1-y, 1-z$.

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All H atoms were positioned geometrically and allowed to ride on their parent atoms, with aromatic C—H distances of 0.93 Å, methyl C—H distances of 0.96 Å and N—H distances of 0.86 Å. The $U_{\rm iso}$(H) values were set at $1.5U_{\rm eq}$(C) for the methyl H atoms and at $1.2U_{\rm eq}$(C) for other C-bound H atoms; $U_{\rm iso}$(H) for the amino H atoms was set equal to $1.5U_{\rm eq}$(N). The 2θ maximum value was limited to 50° because of the large fraction of weak data at higher angles. The higher *R* and *wR* values may be a result of the weak data set.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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