

**(Z)-2-(4-Methoxyphenyl)-3-phenylacrylonitrile**

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**Key indicators**

Single-crystal X-ray study  
 $T = 295$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å  
 $R$  factor = 0.046  
 $wR$  factor = 0.144  
Data-to-parameter ratio = 12.1

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

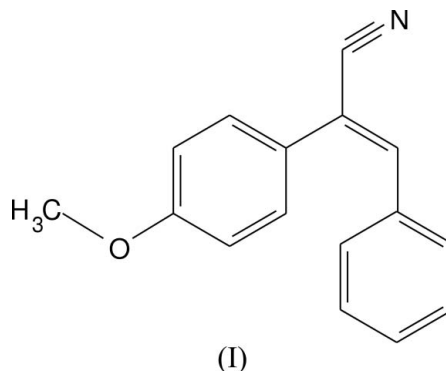
A new dipolarophile for the construction of bioactive isoxazoli(di)nes, (Z)-2-(4-methoxyphenyl)-3-phenylacrylonitrile,  $\text{C}_{16}\text{H}_{13}\text{NO}$ , was synthesized by the base-catalysed reaction of benzaldehyde with 4-methoxyphenylacetonitrile. The olefinic bond connecting the 4-methoxyphenylacetonitrile and the phenyl groups has *Z* geometry. There are two molecules in the asymmetric unit. In the crystal structure, the molecules are linked by  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds.

Received 11 August 2006

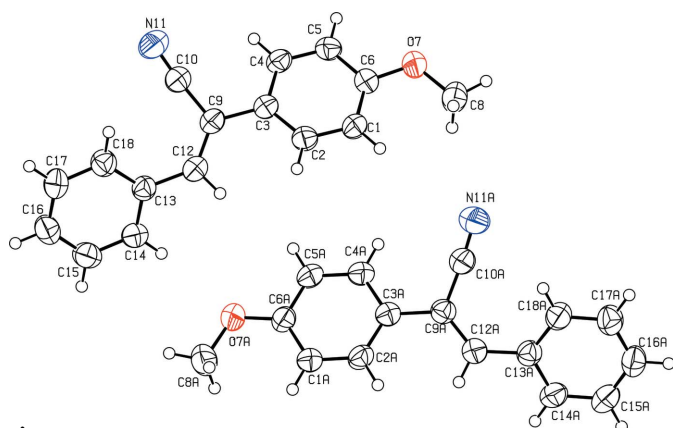
Accepted 16 August 2006

**Comment**

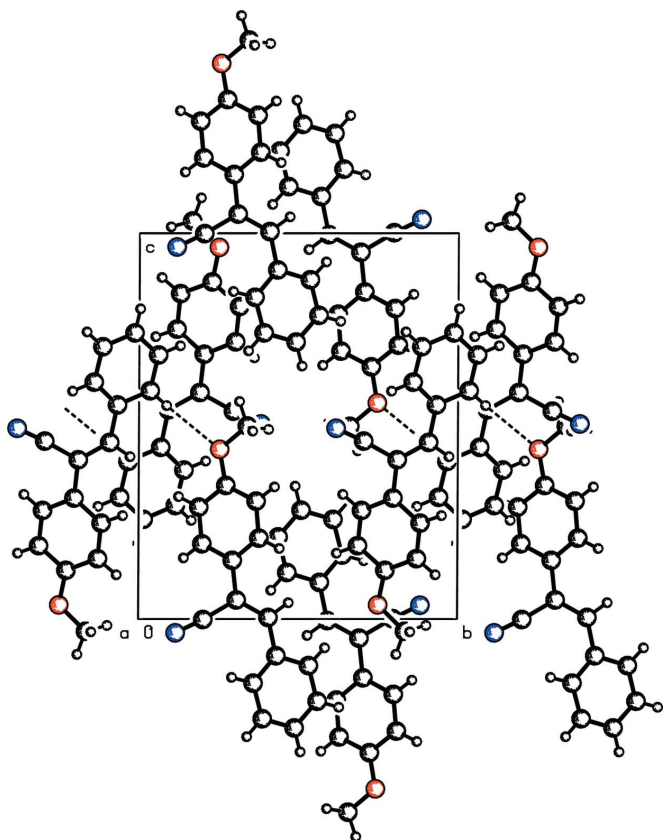
Acrylonitriles represent an interesting class of biologically active compounds. Many derivatives of acrylonitriles have been shown to possess antitumor (Ohsumi *et al.*, 1998), anti-tubercular (Sanna *et al.*, 2000) and antiproliferative activities (Carta *et al.*, 2002). It is well known that acrylonitriles are useful intermediates in organic synthesis and are capable of undergoing many useful organic transformations (Ambrosi *et al.*, 1994); for example, transformation into pyrazole, isoxazole and pyrimidine derivatives (Dawood *et al.*, 1999). Recently, the crystal structures of two bioactive heteroarylacrylonitriles have been reported (Sonar *et al.*, 2005). In both of these compounds the configuration about the olefinic double bond is *Z*. The X-ray crystal structure of the title compound, (I), is reported here.



The molecular structure and atom-numbering scheme are shown in Fig. 1. There are two molecules in the asymmetric unit. The olefinic bond connecting the 4-methoxyphenylacetonitrile and 3,4,5-trimethoxyphenyl groups has *Z* geometry. Significant deviations from the ideal bond-angle geometry around the  $\text{Csp}^2$  atoms of the double bond are observed. The bond angles (corresponding values for the second molecule of the asymmetric unit are given within square brackets)  $\text{C13}-\text{C12}=\text{C9} = 130.6(2)^\circ$  [ $130.7(2)^\circ$ ],  $\text{C12}=\text{C9}-\text{C3} = 124.6(2)^\circ$  [ $125.4(2)^\circ$ ] and  $\text{C10}-\text{C9}-\text{C3} =$



**Figure 1**  
View of the two molecules of the asymmetric unit of (I), with 50% probability displacement ellipsoids.



**Figure 2**  
The crystal packing in (I), viewed down the *c* axis. Dashed lines indicate hydrogen bonds.

114.7 (2)° [114.0 (2)°] are distorted due to the steric hindrance of the double bond linking the two ring systems. The olefinic double bond length, 1.351 (3) Å [1.342 (3) Å], is comparable with the values [1.356 (3) and 1.356 (2) Å] in the two compounds reported by Sonar *et al.* (2005); in a very closely related compound (Naveen *et al.*, 2006) the value is 1.348 (4) Å. A search of the Cambridge Structural Database [Version of January 2006; Allen (2002)] for Ph—C(CN)=CH—Ph with no cyclic substituents gave 20 hits; for these, the average value of the olefinic bond length is

1.353 (3) Å. The torsion angle of 154.1 (2)° [155.4 (2)°] for C14—C13—C12=C9 indicates the deviation of the phenyl ring from the plane of the olefinic double bond. The dihedral angle between the C1- and C13-benzene rings is 53.2 (1)°; that between the C1A- and C13A-benzene rings is 48.5 (1)°.

The structure exhibits intermolecular hydrogen bonds of the type C—H···O (Table 2) which help in stabilizing the crystal structure. The molecules form hydrogen-bonded dimers and are stacked in pairs. (Fig. 2).

## Experimental

To a well stirred suspension of benzaldehyde (0.72 g, 6.8 mmol) in 5% NaOH (10 ml) solution, 2-(4-methoxyphenyl)acetonitrile (1 g, 6.8 mmol) was added, along with a catalytic amount of *tert*-butylammonium bromide. The mixture was stirred at room temperature for 1 h, saturated with sodium chloride solution and extracted with diethyl ether (3 × 15 ml). The combined organic layer was dried over anhydrous sodium sulfate and evaporated under vacuum to obtain a crude mass which, on recrystallization with methanol, gave (I) as a pale-yellow crystalline solid (m.p. 359.15 K). Analysis calculated: C 81.68, H 5.57, N 5.95%; found: C 81.67, H 5.58, N 5.94%.

## Crystal data

$C_{16}H_{13}NO$	$V = 1253 (2) \text{ \AA}^3$
$M_r = 235.27$	$Z = 4$
Triclinic, $P\bar{1}$	$D_x = 1.247 \text{ Mg m}^{-3}$
$a = 7.362 (9) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 11.859 (12) \text{ \AA}$	$\mu = 0.08 \text{ mm}^{-1}$
$c = 14.481 (13) \text{ \AA}$	$T = 295 (2) \text{ K}$
$\alpha = 89.658 (6)^\circ$	Block, pale yellow
$\beta = 82.467 (6)^\circ$	$0.25 \times 0.20 \times 0.20 \text{ mm}$
$\gamma = 89.604 (5)^\circ$	

## Data collection

MacScience DIPLabo 32001 diffractometer	3954 independent reflections
$\omega$ scans	3150 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{\text{int}} = 0.036$
6395 measured reflections	$\theta_{\text{max}} = 25.0^\circ$

## Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0739P)^2 + 0.1197P]$
$R[F^2 > 2\sigma(F^2)] = 0.046$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.144$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.15$	$\Delta\rho_{\text{max}} = 0.23 \text{ e \AA}^{-3}$
3954 reflections	$\Delta\rho_{\text{min}} = -0.22 \text{ e \AA}^{-3}$
328 parameters	Extinction correction: <i>SHELXL97</i>
H-atom parameters constrained	Extinction coefficient: 0.085 (8)

**Table 1**

Selected geometric parameters (Å, °).

O7—C6	1.365 (2)	O7A—C6A	1.362 (2)
O7—C8	1.421 (3)	N11—C10	1.139 (3)
O7A—C8A	1.421 (3)	N11A—C10A	1.140 (3)
C6—O7—C8	117.96 (15)	N11—C10—C9	175.4 (2)
C6A—O7A—C8A	117.91 (16)	O7A—C6A—C1A	125.07 (17)
O7—C6—C1	124.52 (17)	O7A—C6A—C5A	115.76 (17)
O7—C6—C5	115.83 (16)	N11A—C10A—C9A	175.8 (2)

**Table 2**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C14-H14\cdots O7A$	0.93	2.55	3.427 (5)	157
$C14A-H14A\cdots O7^i$	0.93	2.57	3.453 (5)	158

Symmetry code: (i)  $x, y + 1, z$ .

H atoms were placed at idealized positions and allowed to ride on their parent atoms, with  $C-H = 0.93-0.96$  Å and  $U_{iso}(H) = 1.2U_{eq}(C)$ .

Data collection: *XPRESS* (MacScience, 2002); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003) and *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *PLATON*.

We thank the DST, Government of India, for financial assistance under projects DV6/15/DST/2005-06 and SP/I2/FOO/93.

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