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(*Z*)-3-(3,4-Dimethoxyphenyl)-2-(4-methoxyphenyl)acrylonitrile

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
R factor = 0.054
wR factor = 0.168
Data-to-parameter ratio = 12.6

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

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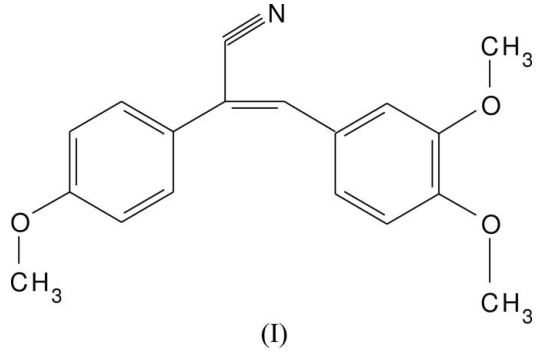
A new dipolarophile used in the construction of bioactive heterocycles, (*Z*)-3-(3,4-dimethoxyphenyl)-2-(4-methoxyphenyl)acrylonitrile, $C_{18}H_{17}NO_3$, has been synthesized by base-catalysed reaction of 3,4-dimethoxybenzaldehyde with (4-methoxyphenyl)acetonitrile. The olefinic bond has *Z* geometry and the molecules are linked by C—H···O and C—H···N hydrogen bonds.

Comment

2,3-Disubstituted acrylonitriles represent an interesting class of biologically active compounds and are capable of undergoing many useful organic transformations and have been transformed into bioactive heterocycles (Urska *et al.*, 2003). Using the nitrile function for C—C bond formation reaction is very important in organic chemistry (Collier *et al.*, 2004). The deprotonation of the α-carbon and alkylation is an important reaction (Murahashi *et al.*, 2004). Combretastatin A-4, (II), shows potent cytotoxicity against a wide variety of human cancer cell lines, including MDR cancer cell lines (El-Zayat *et al.*, 1993) and is thus an attractive lead compound for the development of anticancer drugs. The title compound (I) was designed as an analog of (II) in which the 3-hydroxy-4-methoxyphenyl unit was replaced by a 4-methoxyphenyl unit and olefinic bond carrying nitrile group..



(I)

Recently, the crystal structures of some bioactive heteroarylacrylonitriles have been reported (Maturana *et al.*, 2005). It has been found from the literature that the olefinic bond hase *Z* geometry, irrespective of the size of the substituents on the heterocyclic rings (Sonar *et al.*, 2005). The X-ray structure determination was carried out in order to confirm the olefinic bond geometry connected to 4-methoxyphenylacetonitrile and the 3,4-dimethoxyphenyl ring.

The molecular structure and atom-numbering scheme of (I) are shown in Fig. 1. In (I), the olefinic bond connecting the (4-methoxyphenyl)acetonitrile and 3,4-dimethoxyphenyl groups

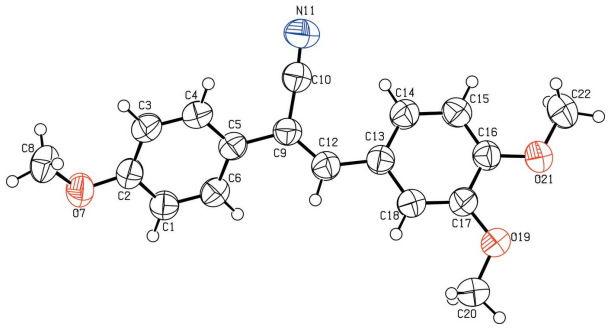


Figure 1
View of (I), with 50% probability displacement ellipsoids.

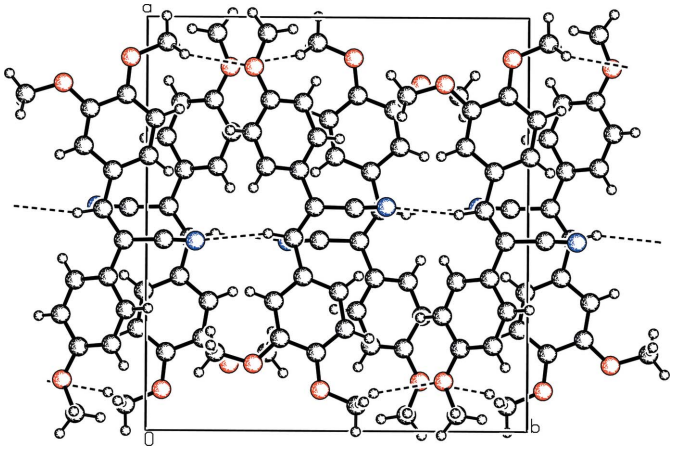


Figure 2
The crystal packing in (I), viewed down the *c* axis. Dashed lines indicate hydrogen bonds.

has *Z* geometry. Significant deviations from the ideal bond-angle geometry around the Csp^2 atoms of the double bond are observed. The bond angles C13—C12═C9 = 132.13 (19)°, C12═C9—C5 = 123.86° and C10—C9—C5 = 114.50 (17)° are distorted due to steric hindrance of the double bond linking the two ring systems. The olefinic double bond bearing the three conjugated substituents in (I) has a length of 1.348 (4) Å, slightly longer than that observed in (*Z*)-2-(3-thienyl)-3-(3,4-dimethoxyphenyl)acrylonitrile [1.353 (3) Å; Sonar *et al.*, 2005] and 2-styrylbenzimidazole [1.304 (4) Å; Bacelo *et al.*, 1997], suggesting some delocalization of the unsaturated bridging units. The C18—C13—C12═C9 torsion angle of 169.4 (2)° indicates the deviation of the 3,4-dimethoxyphenyl ring from the plane of the olefinic double bond.

The structure exhibits intermolecular hydrogen bonds of the type C—H···O and C—H···N (Table 2), which help to stabilize the crystal structure. These intermolecular hydrogen bonds link the molecules into chains (Fig. 2).

Experimental

To a well stirred suspension of 3,4-dimethoxybenzaldehyde (1.13 g, 6.8 mmol) in 5% NaOH (10 ml) solution, was added (4-methoxyphenyl)acetonitrile (1 g, 6.8 mmol) along with a catalytic amount of *tert*-butylammonium bromide. The mixture was stirred at room temperature for 45 min, saturated sodium chloride solution (10 ml) added, 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 from methanol gave (I) as a pale-yellow crystalline solid (m.p 383.15 K). Analysis calculated for $C_{18}H_{17}NO_3$: C 73.20; H 5.80, N 4.74%; found: C 73.21, H 5.80, N 4.73%.

Crystal data

$C_{18}H_{17}NO_3$
M_r = 295.33
Monoclinic, $P2_1/c$
a = 14.830 (9) Å
b = 13.688 (7) Å
c = 7.445 (16) Å
β = 91.724 (2)°
V = 1511 (3) Å^3
Z = 4
D_x = 1.299 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 295 (2) K
Block, pale yellow
0.25 × 0.20 × 0.20 mm

Data collection

MacScience DIPLabo 32001 diffractometer
ω scans
Absorption correction: none
4731 measured reflections
2549 independent reflections
1955 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.168
S = 1.05
2549 reflections
203 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1039P)^2 + 0.2197P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.042 (7)

Table 1
Selected geometric parameters (Å, °).

O7—C2	1.371 (4)	O21—C16	1.368 (4)
O7—C8	1.421 (4)	O21—C22	1.425 (4)
O19—C17	1.368 (4)	N11—C10	1.148 (4)
O19—C20	1.428 (4)		
C2—O7—C8	118.21 (18)	N11—C10—C9	176.5 (2)
C17—O19—C20	117.06 (17)	O21—C16—C15	124.50 (18)
C16—O21—C22	116.98 (17)	O21—C16—C17	116.14 (17)
O7—C2—C1	115.83 (18)	O19—C17—C16	115.41 (18)
O7—C2—C3	124.77 (18)	O19—C17—C18	125.39 (18)

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

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12···N11^{i}	0.93	2.62	3.522 (8)	164
C22—H22C···O7^{ii}	0.96	2.55	3.341 (8)	140

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

H atoms were placed at idealized positions and allowed to ride on their parent atoms, with C—H = 0.92–0.98 Å and U_{iso}(H) = xU_{eq}(C), where x = 1.5 for methyl H atoms and 1.2 for other H atoms.

Data collection: *XPRESS* (MacScience, 2002); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski and Minor, 1997) and *SCALEPACK*; 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*.

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