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3-[4-(Dimethylamino)phenyl]-2-(4-methoxyphenyl)acrylonitrile

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.003 Å
R factor = 0.049
wR factor = 0.146
Data-to-parameter ratio = 13.0

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

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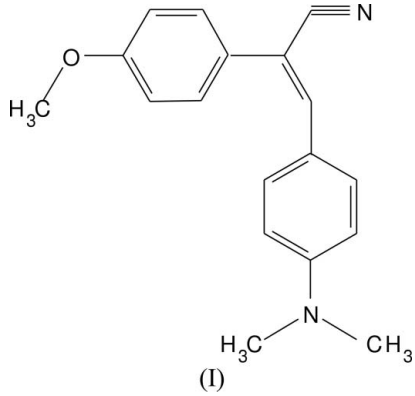
A new dipolarophile for the construction of the bioactive title compound, $C_{18}H_{18}N_2O$, was synthesized by base-catalysed reaction of benzaldehyde with (4-methoxyphenyl)acetonitrile. The olefinic bond connecting the (4-methoxyphenyl)-acetonitrile and the dimethylaminophenyl groups has a *Z* geometry.

Comment

Acrylonitriles represent an interesting class of biologically active compounds. Many derivatives of acrylonitriles have been shown to possess antitumor (Ohsumi *et al.*, 1998), antitubercular (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, into pyrazole, isoxazole and pyrimidine derivatives (Dawood *et al.*, 1999). Recently, the crystal structures of some bioactive heteroarylacrylonitriles have been reported (Sonar *et al.*, 2005; Naveen *et al.*, 2006). We found from the literature that the olefinic bond has a *Z* configuration irrespective of the size of the substituents on the heterocyclic rings (Sonar *et al.*, 2005). In order to confirm the olefinic bond geometry and to obtain detailed information on the conformation of the molecule of (I), its X-ray crystal structure determination has been carried out.



(I)

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 (*N*,*N*-dimethylamino)phenyl groups has *Z* geometry. Significant deviations from the ideal bond-angle geometry around the C*sp*^{2} atoms of the double bond are observed. The bond angles C13—C12═C9 = 132.01 (19)°, C12═C9—C2 = 125.76 (17)° and C10—C9—C2 = 114.14 (16)° are distorted due to the steric hindrance about

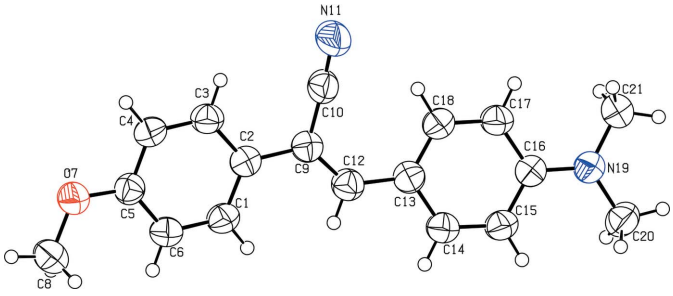


Figure 1
The molecular structure of (I), with 50% probability displacement ellipsoids.

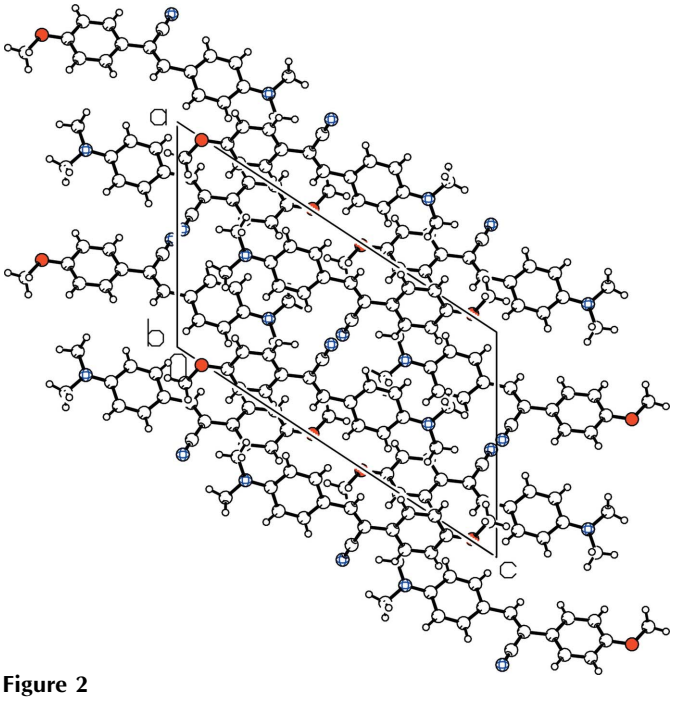


Figure 2
The crystal packing in (I), viewed down the *b* axis.

the double bond linking the two ring systems. The olefinic double bond bearing the three conjugated substituents in (I) has a length of 1.3514 (3) Å which is comparable with (*Z*)-2-(3-thienyl)-3-(3,4,5-trimethoxyphenyl)acrylonitrile [1.353 (3) Å; Sonar *et al.*, 2005] and 2-styrylbenzimidazole [1.304 (4) Å; Bacelo *et al.*, 1997], suggesting some delocalization in the unsaturated bridging unit. The C14–C13–C12═C9 torsion angle of 169.55 (2)° indicates the deviation of the (*N*,*N*-dimethylamino)phenyl ring from the plane of the olefinic double bond. The structure exhibits an intramolecular hydrogen bond of the type C—H···N (Table 2).

Experimental

To a well stirred suspension of 4-(*N*,*N*-dimethylamino)benzaldehyde (1 g, 6.8 mmol) in 5% NaOH (10 ml) solution, was added 2-(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 50 min, 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 the crude mass, which on recrystallization from methanol gave (I) as a colourless crystalline solid (m.p 373 K). Single crystals of (I) suitable for the X-ray analysis were obtained by slow evaporation of a methanol solution.

Crystal data

$C_{18}H_{18}N_2O$
M_r = 278.34
Monoclinic, $P2_1/c$
a = 11.736 (9) Å
b = 7.646 (8) Å
c = 19.932 (12) Å
β = 123.379 (5)°
V = 1494 (2) Å^3
Z = 4
D_x = 1.238 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 295 (2) K
Block, colourless
0.30 × 0.25 × 0.25 mm

Data collection

MacScience DIPLabo 32001 diffractometer
ω scans
Absorption correction: none
3743 measured reflections
2514 independent reflections
2080 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.146
S = 1.05
2514 reflections
194 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0771P)^2 + 0.2878P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ <0.001
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O7—C5	1.366 (3)	N19—C16	1.368 (3)
O7—C8	1.422 (3)	N19—C20	1.444 (3)
N11—C10	1.148 (3)	N19—C21	1.447 (3)
C5—O7—C8	118.19 (17)	O7—C5—C6	124.86 (17)
C16—N19—C20	120.66 (16)	N11—C10—C9	176.2 (2)
C16—N19—C21	121.10 (18)	N19—C16—C15	122.18 (18)
C20—N19—C21	117.63 (15)	N19—C16—C17	121.27 (17)
O7—C5—C4	116.07 (17)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C18—H18···N11	0.93	2.61	3.419 (5)	146

H atoms were placed at idealized positions and allowed to ride on their parent atoms, with C—H = 0.93—0.96 Å and $U_{iso}(H) = xU_{eq}(C)$, where x = 1.5 for methyl H atoms and x = 1.2 for other H atoms.

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*.

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