Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

(*Z*)-3-(1-Methyl-1*H*-indol-3-yl)-2-(thiophen-3-yl)acrylonitrile

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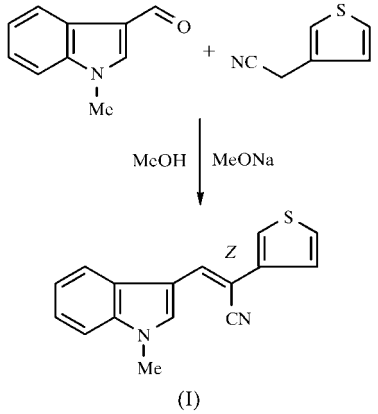
Received 21 January 2004
Accepted 2 February 2004
Online 19 February 2004

The title compound, $C_{16}H_{12}N_2S$, has been synthesized by base-catalyzed condensation of 1-methylindole-3-carboxaldehyde with thiophene-3-acetonitrile. The product assumes an approximately planar *Z* configuration. The molecule has a thienyl-ring flip disorder.

Comment

There has been an increasing demand for new antituberculosis agents, as the fast development of mycobacterial resistance to conventional drugs is one of the major difficulties in the treatment of tuberculosis. Encouraged by the antitubercular activity of 3-aryl-substituted 2-[1*H*(2*H*)-benzotriazol-1(2)-yl]-acrylonitriles (Sanna *et al.*, 2000), we have synthesized a series of substituted aryl/heteroaryl 2-(thiophen-3-yl)acrylonitriles and evaluated them for antitubercular activity against *Mycobacterium tuberculosis* $H37R_v$. In the present paper, we present the results of an X-ray structural investigation of the title



(I)

compound, (I), which was prepared by base-catalyzed condensation of 1-methylindole-3-carboxaldehyde with thiophene-3-acetonitrile. The compound was initially identified by NMR spectroscopy. In order to confirm the geometry, and to obtain detailed information on the structural conformation of the molecule, its X-ray structure determination has been carried out and the results are presented here.

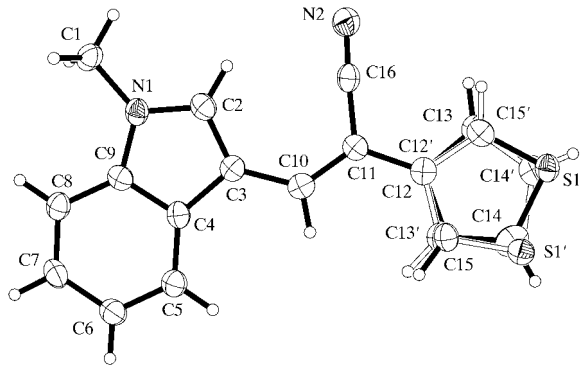


Figure 1
A view of (I), with displacement ellipsoids drawn at the 50% probability level.

Fig. 1 shows an ellipsoid plot of (I), and selected geometric parameters are presented in Table 1. In (I), the indole ring is in a *trans* disposition with respect to the thiophene across the double bond linking the two rings. Deviations from the ideal bond-angle geometry around the Csp^2 atoms of the double bonds are observed. The C10═C11—C16 and C13═C12—C11 bond angles are 120.3 (2) and 121.1 (4)°, respectively, close to the ideal geometry (120°); however, the C2═C3—C10, C11═C10—C3, C10═C11—C12 and C16—C11—C12 angles [129.8 (2), 129.8 (2), 125.3 (2) and 114.4 (2)°, respectively] are distorted because of steric hindrance of the double bond linking the two ring systems. The C3—C10, C11—C12 and C11—C16 bond lengths [1.436 (3), 1.472 (3) and 1.435 (4) Å, respectively] are slightly shorter than C_{ar}—Csp^2 single bonds, providing evidence of some delocalization in the bridging units of this molecule (*International Tables for*

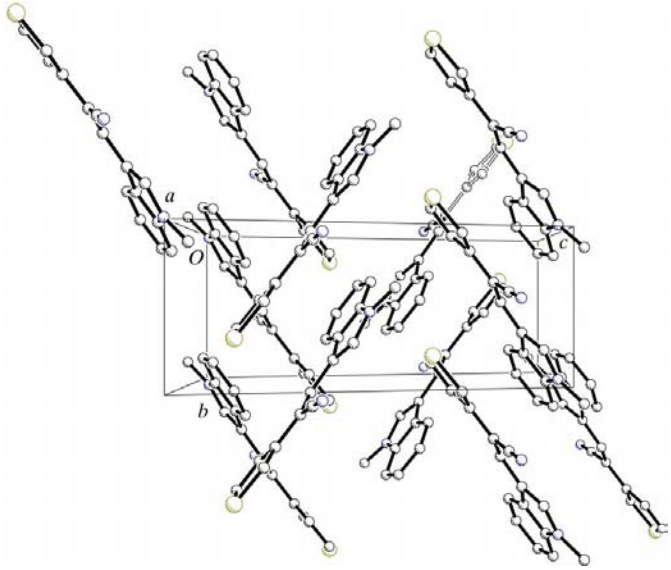


Figure 2
The crystal packing of (I), viewed along the *a* direction.

 DOI: 10.1107/S0108270104002550

Crystallography, 1992, Vol. C, Table 9.5.1.1). The C2═C3—C10═C11, C10═C11—C12═C13, C3—C10═C11—C16 and C16—C11—C12═C13 torsion angles [−5.7 (4), 176.4 (9), 0.0 (4) and −1.3 (10)°] indicate that the indole and thiophene ring planes deviate little from the plane of the double bond. Furthermore, the conformation is stabilized by intramolecular C—H···N and C—H···π interactions (Table 2). The molecule has a thienyl-ring flip disorder, the thiophene ring being disordered over two conformations about the C11—C12 bond, with a major–minor ratio of about 75:25.

The mode of packing of (I) along the *a* direction is illustrated in Fig. 2; van der Waals forces contribute to the stabilization of the crystal structure.

Experimental

A mixture of 1-methylindole-3-carboxaldehyde (0.796 g, 5 mmol) and thiophene-3-acetonitrile (0.616 g, 5 mmol) was dissolved in 5% methanol sodium methoxide (15 ml) and the solution was refluxed for 2 h. The cooled reaction mixture was poured on to crushed ice (100 g) and the yellow solid that separated was collected by filtration and dried. Crystallization from methanol afforded yellow needles suitable for X-ray analysis. ^{1}H NMR ($CDCl_3$, p.p.m.): 3.87 (*s*, 3H), 7.25–7.39 (*m*, 5H), 7.45 (*t*, 1H), 7.73 (*s*, 1H), 7.75 (*s*, 1H), 8.24 (*s*, 1H); ^{13}C NMR ($CDCl_3$, p.p.m.): 33.8, 99.8, 110.2, 110.4, 118.2, 120.2, 121.1, 121.2, 123.2, 124.2, 127.1, 128.1, 130.4, 132.0, 136.7, 137.1.

Crystal data

$C_{16}H_{12}N_2S$
M_r = 264.34
Monoclinic, $P2_1/c$
a = 15.4031 (8) Å
b = 5.8621 (4) Å
c = 15.4902 (9) Å
β = 113.237 (3)°
V = 1285.22 (13) Å^3
Z = 4
D_x = 1.366 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2486 reflections
θ = 1.0–25.4°
μ = 0.24 mm^{-1}
T = 90.0 (2) K
Splintered needle, yellow
0.40 × 0.10 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
ω scans at fixed χ = 55°
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.911, T_{max} = 0.988
4061 measured reflections
2253 independent reflections
1594 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 25.0°
$h = -18 \rightarrow 18$
$k = -6 \rightarrow 6$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.126
S = 1.57
2253 reflections
210 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0537P)^2 + 0.4997P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.023
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

The geometric and displacement parameters of the atoms of the disordered thienyl ring were restrained to be similar, approximately isotropic, and to obey the rigid-bond criterion. As such, the geometry should not be considered definitive.

Table 1
Selected geometric parameters (Å, °).

N1—C2	1.359 (3)	C11—C12	1.472 (3)
N1—C9	1.380 (3)	C13—S1	1.712 (7)
C3—C10	1.436 (3)	S1—C14	1.682 (10)
C11—C16	1.435 (4)	C16—N2	1.152 (3)
C2—N1—C1	126.3 (2)	C10—C11—C16	120.3 (2)
N1—C2—C3	111.0 (2)	C10—C11—C12	125.3 (2)
C2—C3—C10	129.8 (2)	C16—C11—C12	114.4 (2)
C11—C10—C3	129.8 (2)	N2—C16—C11	176.6 (3)
C2—C3—C10—C11	−5.7 (4)	C10—C11—C16—N2	164 (4)
C10—C11—C12—C13	176.4 (9)	C12—C11—C16—N2	−18 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···C16	0.95	2.49	3.014 (4)	115
C2—H2···N2	0.95	2.62	3.403 (3)	140
C13—H13···C16	0.95	2.49	2.780 (8)	98

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 1995); software used to prepare material for publication: *SHELXL*97 (Sheldrick, 1997) and local procedures.

This investigation was supported by the National Institute of Alcohol Abuse and Alcoholism (grant No. AA12600). The *in vitro* antituberculosis activity was carried out in the Tuberculosis Antimicrobial Acquisition and Coordinating Facility (TAACF) at the National Institute of Allergy and Infectious Disease, Southern Research Institute, GWL Hansen's Disease Center, and Colorado State University, USA.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1640). Services for accessing these data are described at the back of the journal.

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