

Three substituted (*E*)-3-aryl-2-(thienyl)-acrylonitriles: isolated molecules, simple hydrogen-bonded chains and hydrogen-bonded sheets

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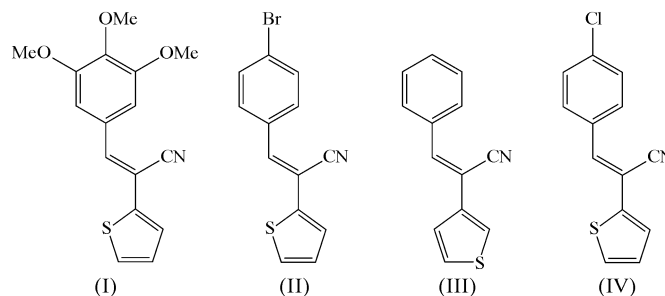
The structure of (*E*)-2-(2-thienyl)-3-(3,4,5-trimethoxyphenyl)-acrylonitrile, C₁₆H₁₅NO₃S, contains no direction-specific intermolecular interactions. The molecules of (*E*)-3-(4-bromophenyl)-2-(2-thienyl)acrylonitrile, C₁₃H₈BrNS, exhibit orientational disorder of the thienyl fragment, and the molecules are linked into simple *C*(5) chains by a single C—H···N hydrogen bond. In (*E*)-3-phenyl-2-(3-thienyl)acrylonitrile, C₁₃H₉NS, the molecules are linked into sheets by a combination of one C—H···N hydrogen bond and one C—H··· π (arene) hydrogen bond.

Comment

We report here the structures of three substituted (*E*)-3-aryl-2-(thienyl)acrylonitriles, namely (*E*)-2-(2-thienyl)-3-(3,4,5-trimethoxyphenyl)acrylonitrile, (I) (Fig. 1), (*E*)-3-(4-bromophenyl)-2-(2-thienyl)acrylonitrile, (II) (Fig. 2), and (*E*)-3-phenyl-2-(3-thienyl)acrylonitrile, (III) (Fig. 3), which have been synthesized for use as potential intermediates in the synthesis of new fused heterocyclic systems. The structure of the analogous (*E*)-3-(4-chlorophenyl)-2-(2-thienyl)acrylonitrile, (IV), was reported recently (Cobo *et al.*, 2005).

For compound (I), the key torsion angles (Table 1) show that the non-H atoms are very nearly coplanar, with the sole exception of atom C141 of the 4-methoxy group. The exocyclic angles at the methoxy substituents in (I) show the usual patterns of behaviour, with markedly different C—C—O angles for the 3- and 5-methoxy substituents, which are effectively coplanar with the aryl ring, and rather similar angles for the 4-methoxy substituent, where the methyl C atom is displaced from the plane of the aryl ring by 1.261 (2) Å.

Compound (II) is isomorphous and isostructural with the chloro analogue, *viz.* (IV) (Cobo *et al.*, 2005). In (II), there is a significant rotation of the aryl group around the



C11—C17 bond, so that this fragment is not coplanar with the rest of the molecule (Table 2). There is no obvious reason for this conformational difference between compounds (I) and (II), as the aryl ring in (II) is not involved in any hydrogen bonding. In the isostructural pair (II) and (IV), although not in compound (I), the 2-thienyl group exhibits orientational disorder over two sets of sites corresponding to a 180° rotation about the C2—C27 bond. The populations of the major and minor conformers in compounds (II) and (IV) are experimentally indistinguishable: 0.798 (3) and 0.202 (3) in (II), and 0.802 (3) and 0.198 (3) in (IV).

The molecules of compound (III) are almost planar, as shown by the key torsion angles (Table 4). In (I)–(III), the nitrile components exhibit quite long C—C bonds and very short C—N bonds. The remaining bond distances in (I)–(III) show no unusual features.

There are no direction-specific intermolecular interactions in the structure of compound (I). In particular, C—H···N, C—H···O and C—H··· π (arene) hydrogen bonds and aromatic π — π stacking interactions are all absent, so that the structure consists of effectively isolated molecules.

By contrast, the molecules of compound (II) are linked by a single C—H···N hydrogen bond (Table 3), exactly as in compound (IV). Alkene atom C17 in the molecule at (*x*, *y*, *z*)

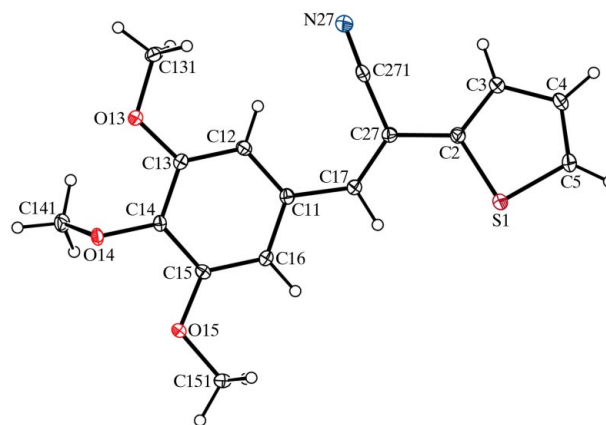


Figure 1

The molecule of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

acts as hydrogen-bond donor to atom N27 in the molecule at $(-\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z)$, so forming a $C(5)$ chain (Bernstein *et al.*, 1995) running parallel to the $[101]$ direction and generated by

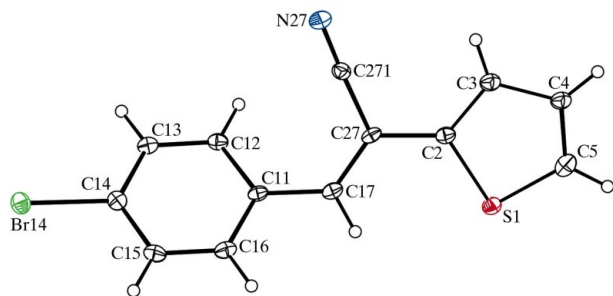


Figure 2
The molecule of compound (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

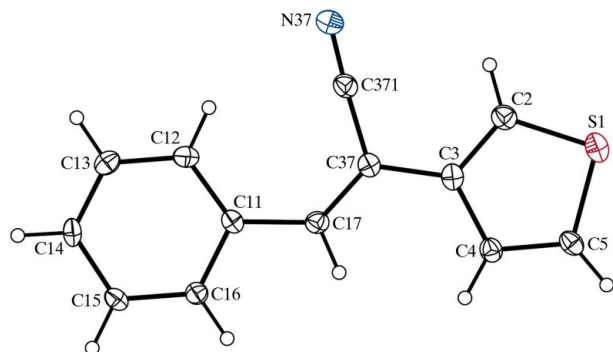


Figure 3
The molecule of compound (III), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. For the sake of clarity, only the major orientation of the disordered thieryl ring is shown.

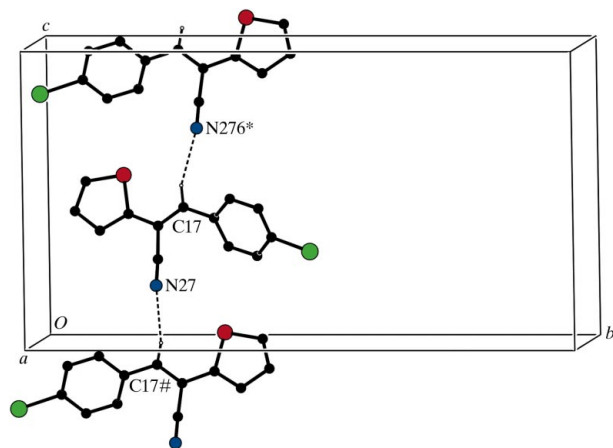


Figure 4
Part of the crystal structure of compound (II), showing the formation of a $C(5)$ chain along $[101]$. For the sake of clarity, only the major orientation of the disordered thieryl ring is shown, and H atoms not involved in the motif shown have been omitted. Atoms marked with an asterisk (*) or a hash symbol (#) are at the symmetry positions $(-\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z)$ and $(\frac{1}{2} + x, \frac{1}{2} - y, -\frac{1}{2} + z)$, respectively.

the n -glide plane at $y = \frac{1}{4}$ (Fig. 4). Two chains of this type, which are related to one another by inversion and hence are anti-parallel, and generated by the n -glide planes at $y = \frac{1}{4}$ and $y = \frac{3}{4}$, pass through each unit cell, but there are no direction-specific interactions between adjacent chains.

In the structure of compound (III), the molecules are linked into sheets by a combination of $C-H \cdots N$ and $C-H \cdots \pi(\text{arene})$ hydrogen bonds (Table 5). Atom C2 in the molecule at (x, y, z) acts as hydrogen-bond donor to atom N37 in the molecule at $(1 - x, -y, 1 - z)$, so generating by inversion an $R_2^2(12)$ dimer centred at $(\frac{1}{2}, 0, \frac{1}{2})$ (Fig. 5). In addition, atoms C13 in the molecules at (x, y, z) and $(1 - x, -y, 1 - z)$, which form a dimer centred at $(\frac{1}{2}, 0, \frac{1}{2})$, act as hydrogen-bond donors to the aryl rings of the molecules at $(2 - x, -\frac{1}{2} + y, \frac{3}{2} - z)$ and $(-1 + x, \frac{1}{2} - y, -\frac{1}{2} + z)$, respectively, which themselves are components of dimers centred at $(\frac{3}{2}, -\frac{1}{2}, 1)$ and $(-\frac{1}{2}, \frac{1}{2}, 0)$, respectively. Similarly, the aryl rings at (x, y, z) and $(1 - x, -y, 1 - z)$ accept hydrogen bonds from atom C13 in the molecules at $(2 - x, \frac{1}{2} + y, \frac{3}{2} - z)$ and $(-1 + x, -\frac{1}{2} - y, -\frac{1}{2} + z)$, themselves parts of dimers centred at $(\frac{3}{2}, \frac{1}{2}, 1)$

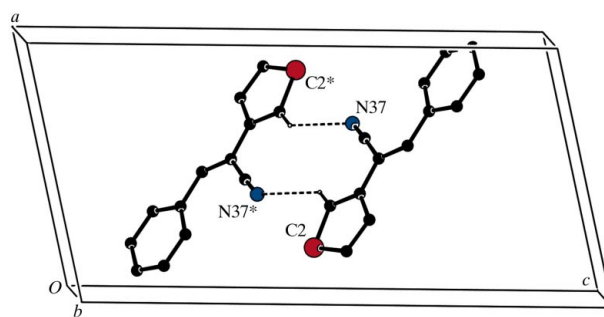


Figure 5
Part of the crystal structure of compound (III), showing the formation of an $R_2^2(12)$ dimer. For the sake of clarity, H atoms not involved in the motif shown have been omitted. Atoms marked with an asterisk (*) are at the symmetry position $(1 - x, -y, 1 - z)$.

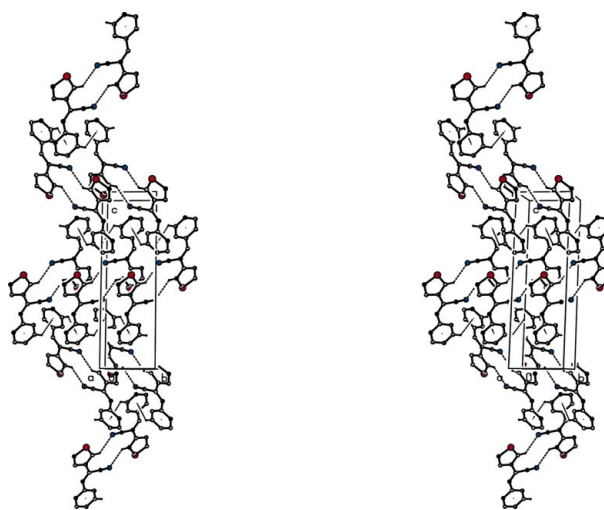


Figure 6
A stereoview of part of the crystal structure of compound (III), showing the formation of a hydrogen-bonded sheet parallel to $(10\bar{2})$. For the sake of clarity, H atoms not involved in the motifs shown have been omitted.

and $(-\frac{1}{2}, -\frac{1}{2}, 0)$, respectively. Propagation of this interaction then links the $R_2^2(12)$ dimers into a sheet parallel to $(10\bar{2})$ (Fig. 6).

Experimental

Compounds (I)–(III) were prepared using procedures similar to that employed for the synthesis of compound (IV) (Cobo *et al.*, 2005). A solution of 2-thiopheneacetonitrile [for (I) and (II)] or 3-thiopheneacetonitrile [for (III)] (1 mmol) and potassium *tert*-butoxide (1 mmol) in anhydrous ethanol (3 ml) was stirred at room temperature for 15 min. A solution of the appropriate benzaldehyde (1 mmol) in anhydrous ethanol (3 ml) was then added, and the overall mixtures were then heated under reflux for 2–3 h. The resulting solid products were collected by filtration, washed with ethanol, dried, and finally crystallized from dimethylformamide to give yellow crystals suitable for single-crystal X-ray diffraction. Compound (I): m.p. 391–392 K, yield 70%; MS EI (30 eV) *m/z* (%): 302 (21), 301 (100, M^+), 286 (39 $M^+ - \text{CH}_3$), 226 (16). Compound (II): m.p. 368–370 K, yield 68%; MS EI (30 eV) *m/z* (%): 292 (17), 291/289 (100/98, M^+), 290 (22), 211 (16), 210 (81), 209 (89), 208 (91), 183 (16), 177 (35), 166 (19), 139 (16), 154 (17), 127 (10), 45 (14). Compound (III): m.p. 348–349 K [literature m.p. 348 K (Stuart *et al.*, 1986)], yield 60%.

Compound (I)

Crystal data

$\text{C}_{16}\text{H}_{15}\text{NO}_3\text{S}$ $Z = 4$
 $M_r = 301.36$ $D_x = 1.413 \text{ Mg m}^{-3}$
 Monoclinic, $P2_1/c$ Mo $K\alpha$ radiation
 $a = 22.5423 (6) \text{ \AA}$ $\mu = 0.24 \text{ mm}^{-1}$
 $b = 8.4647 (3) \text{ \AA}$ $T = 120 (2) \text{ K}$
 $c = 7.4243 (2) \text{ \AA}$ Block, colourless
 $\beta = 91.510 (2)^\circ$ $0.30 \times 0.20 \times 0.10 \text{ mm}$
 $V = 1416.17 (7) \text{ \AA}^3$

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer 17457 measured reflections
 φ and ω scans 3228 independent reflections
 Absorption correction: multi-scan 2538 reflections with $I > 2\sigma(I)$
 (SADABS; Sheldrick, 2003) $R_{\text{int}} = 0.041$
 $T_{\text{min}} = 0.920, T_{\text{max}} = 0.977$ $\theta_{\text{max}} = 27.5^\circ$

Refinement

Refinement on F^2 $w = 1/[\sigma^2(F_o^2) + (0.0648P)^2 + 0.0444P]$
 $R[F^2 > 2\sigma(F^2)] = 0.038$ where $P = (F_o^2 + 2F_c^2)/3$
 $wR(F^2) = 0.113$ $(\Delta/\sigma)_{\text{max}} = 0.001$
 $S = 1.12$ $\Delta\rho_{\text{max}} = 0.40 \text{ e \AA}^{-3}$
 3228 reflections $\Delta\rho_{\text{min}} = -0.43 \text{ e \AA}^{-3}$
 193 parameters
 H-atom parameters constrained

Table 1

Selected geometric parameters ($\text{\AA}, ^\circ$) for (I).

C27–C271	1.443 (2)	C271–N27	1.145 (2)
O13–C13–C12	124.05 (14)	O14–C14–C15	119.86 (13)
O13–C13–C14	115.33 (13)	O15–C15–C14	115.80 (12)
O14–C14–C13	120.47 (14)	O15–C15–C16	124.47 (14)
S1–C2–C27–C17	–9.0 (2)	C12–C13–O13–C131	7.9 (2)
C2–C27–C17–C11	174.72 (15)	C13–C14–O14–C141	75.54 (17)
C27–C17–C11–C12	–5.5 (3)	C16–C15–O15–C151	2.2 (2)

Compound (II)

Crystal data

$\text{C}_{13}\text{H}_8\text{BrNS}$ $Z = 4$
 $M_r = 290.17$ $D_x = 1.669 \text{ Mg m}^{-3}$
 Monoclinic, $P2_1/n$ Mo $K\alpha$ radiation
 $a = 3.8557 (2) \text{ \AA}$ $\mu = 3.71 \text{ mm}^{-1}$
 $b = 24.0484 (7) \text{ \AA}$ $T = 120 (2) \text{ K}$
 $c = 12.5466 (4) \text{ \AA}$ Needle, colourless
 $\beta = 96.877 (2)^\circ$ $0.38 \times 0.04 \times 0.03 \text{ mm}$
 $V = 1154.99 (8) \text{ \AA}^3$

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer 12766 measured reflections
 φ and ω scans 2598 independent reflections
 Absorption correction: multi-scan 1983 reflections with $I > 2\sigma(I)$
 (SADABS; Sheldrick, 2003) $R_{\text{int}} = 0.059$
 $T_{\text{min}} = 0.333, T_{\text{max}} = 0.897$ $\theta_{\text{max}} = 27.5^\circ$

Refinement

Refinement on F^2 $w = 1/[\sigma^2(F_o^2) + (0.032P)^2 + 0.6935P]$
 $R[F^2 > 2\sigma(F^2)] = 0.040$ where $P = (F_o^2 + 2F_c^2)/3$
 $wR(F^2) = 0.081$ $(\Delta/\sigma)_{\text{max}} = 0.001$
 $S = 1.09$ $\Delta\rho_{\text{max}} = 0.54 \text{ e \AA}^{-3}$
 2598 reflections $\Delta\rho_{\text{min}} = -0.76 \text{ e \AA}^{-3}$
 147 parameters Extinction correction: SHELXL97
 H-atom parameters constrained (Sheldrick, 1997)
 Extinction coefficient: 0.0211 (12)

Table 2

Selected geometric parameters ($\text{\AA}, ^\circ$) for (II).

C27–C271	1.446 (4)	C271–N27	1.143 (4)
S1–C2–C27–C17	5.2 (4)	C27–C17–C11–C12	38.7 (4)
C2–C27–C17–C11	–178.1 (3)		

Table 3

Hydrogen-bond geometry ($\text{\AA}, ^\circ$) for (II).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
C17–H17 \cdots N27 ⁱ	0.95	2.55	3.450 (4)	159

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

Compound (III)

Crystal data

$\text{C}_{13}\text{H}_9\text{NS}$ $Z = 4$
 $M_r = 211.27$ $D_x = 1.360 \text{ Mg m}^{-3}$
 Monoclinic, $P2_1/c$ Mo $K\alpha$ radiation
 $a = 9.6280 (11) \text{ \AA}$ $\mu = 0.27 \text{ mm}^{-1}$
 $b = 5.7190 (3) \text{ \AA}$ $T = 120 (2) \text{ K}$
 $c = 19.247 (2) \text{ \AA}$ Block, yellow
 $\beta = 103.129 (7)^\circ$ $0.49 \times 0.31 \times 0.20 \text{ mm}$
 $V = 1032.09 (17) \text{ \AA}^3$

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer 24963 measured reflections
 φ and ω scans 2368 independent reflections
 Absorption correction: multi-scan 1788 reflections with $I > 2\sigma(I)$
 [SADABS (Sheldrick, 2003) and EVALCCD (Duisenberg *et al.*, 2003)] $R_{\text{int}} = 0.036$
 $T_{\text{min}} = 0.878, T_{\text{max}} = 0.947$ $\theta_{\text{max}} = 27.5^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1146P)^2 + 1.443P]$
$R[F^2 > 2\sigma(F^2)] = 0.067$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.206$	$(\Delta/\sigma)_{\max} < 0.001$
$S = 1.07$	$\Delta\rho_{\max} = 0.57 \text{ e } \text{\AA}^{-3}$
2368 reflections	$\Delta\rho_{\min} = -0.67 \text{ e } \text{\AA}^{-3}$
136 parameters	
H-atom parameters constrained	

Table 4

Selected geometric parameters (\AA , $^\circ$) for (III).

C37—C371	1.444 (4)	C371—N37	1.146 (4)
C2—C3—C37—C17	179.0 (3)	C37—C17—C11—C12	-1.7 (5)
C3—C37—C17—C11	-178.1 (2)		

Table 5

Hydrogen-bond geometry (\AA , $^\circ$) for (III).

Cg is the centroid of the C11—C16 ring.

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
C2—H2 \cdots N37 ⁱ	0.95	2.59	3.324 (5)	135
C13—H13 \cdots Cg ⁱⁱ	0.95	2.86	3.566 (4)	132

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

The space groups $P2_1/c$, $P2_1/n$ and $P2_1/c$ for compounds (I), (II) and (III), respectively, were uniquely assigned from the systematic absences. All H atoms were located in difference maps and then treated as riding atoms, with C—H distances of 0.95 \AA and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$, or C—H = 0.98 \AA and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for the methyl groups. In compound (II), the disorder of the thienyl group was modelled using a common set of sites for atoms C2, C4 and C5 in the two orientations and individual sites for the remaining atoms of this unit, denoted S1 and C3 for the major orientation, and S3 and C1 for the minor orientation. The refined site occupancies for the two orientations were 0.798 (3) and 0.202 (3).

For all compounds, data collection: *COLLECT* (Nonius, 1999). Cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* for (I) and (II); *DIRAX/LSQ* (Duisenberg *et al.*, 2000) for (III). Data reduction: *DENZO* and *COLLECT* for (I) and (II); *EVALCCD* (Duisenberg *et al.*, 2003) for (III). For all compounds,

program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *OSCAIL* (McArdle, 2003) and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

X-ray data were collected at the EPSRC National X-ray Crystallography Service, University of Southampton, England. JC and JT thank the Consejería de Innovación, Ciencia y Empresa (Junta de Andalucía, Spain), and the Universidad de Jaén for financial support. JT also thanks the Universidad de Jaén for a research scholarship supporting a short stay at the EPSRC National X-ray Crystallography Service. JQ and DC thank COLCIENCIAS and UNIVALLE (Universidad del Valle, Colombia) for financial support.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK3037). Services for accessing these data are described at the back of the journal.

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supporting information

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Three substituted (*E*)-3-aryl-2-(thienyl)acrylonitriles: isolated molecules, simple hydrogen-bonded chains and hydrogen-bonded sheets

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Computing details

For all compounds, data collection: *COLLECT* (Nonius, 1999). Cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* for (I), (II); *DIRAX/LSQ* (Duisenberg *et al.*, 2000) for (III). Data reduction: *DENZO* and *COLLECT* for (I), (II); *EVALCCD* (Duisenberg *et al.*, 2003) for (III). For all compounds, program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *OSCAIL* (McArdle, 2003) and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

(I) (*E*)-2-(2-thienyl)-3-(3,4,5-trimethoxyphenyl)acrylonitrile

Crystal data

C₁₆H₁₅NO₃S

M_r = 301.36

Monoclinic *P*2₁/*c*

Hall symbol: -*P* 2ybc

a = 22.5423 (6) Å

b = 8.4647 (3) Å

c = 7.4243 (2) Å

β = 91.510 (2)°

V = 1416.17 (7) Å³

Z = 4

F(000) = 632

D_x = 1.413 Mg m⁻³

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 3228 reflections

θ = 3.6–27.5°

μ = 0.24 mm⁻¹

T = 120 K

Block, colourless

0.30 × 0.20 × 0.10 mm

Data collection

Bruker Nonius KappaCCD area-detector diffractometer

Radiation source: Bruker Nonius FR591 rotating anode

Graphite monochromator

Detector resolution: 9.091 pixels mm⁻¹

φ and ω scans

Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)

T_{min} = 0.920, *T_{max}* = 0.977

17457 measured reflections

3228 independent reflections

2538 reflections with *I* > 2σ(*I*)

R_{int} = 0.041

θ_{\max} = 27.5°, θ_{\min} = 3.6°

h = -29→29

k = -10→10

l = -9→9

*Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.038$ $wR(F^2) = 0.113$ $S = 1.12$

3228 reflections

193 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0648P)^2 + 0.0444P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 0.40 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.43 \text{ e } \text{\AA}^{-3}$ *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.365337 (18)	-0.03283 (5)	0.35486 (6)	0.02416 (15)
C3	0.43017 (7)	0.1966 (2)	0.2500 (2)	0.0224 (4)
C4	0.46269 (7)	0.0567 (2)	0.2213 (2)	0.0273 (4)
C5	0.43338 (7)	-0.0758 (2)	0.2709 (2)	0.0275 (4)
C2	0.37542 (7)	0.16707 (19)	0.3227 (2)	0.0191 (3)
C27	0.33118 (7)	0.28406 (18)	0.3742 (2)	0.0182 (3)
C271	0.35150 (7)	0.4454 (2)	0.3625 (2)	0.0205 (4)
N27	0.36949 (6)	0.57108 (17)	0.3475 (2)	0.0285 (4)
C17	0.27596 (7)	0.24530 (19)	0.4285 (2)	0.0184 (3)
C11	0.22779 (6)	0.34124 (19)	0.49727 (19)	0.0174 (3)
C12	0.23237 (7)	0.5027 (2)	0.5352 (2)	0.0187 (3)
C13	0.18419 (7)	0.58327 (18)	0.6037 (2)	0.0176 (3)
O13	0.18495 (5)	0.73973 (13)	0.64863 (15)	0.0215 (3)
C131	0.23604 (7)	0.8279 (2)	0.6012 (2)	0.0233 (4)
C14	0.13091 (7)	0.50422 (19)	0.6354 (2)	0.0166 (3)
O14	0.08350 (5)	0.58386 (12)	0.70614 (14)	0.0195 (3)
C141	0.05347 (7)	0.6835 (2)	0.5761 (2)	0.0245 (4)
C15	0.12678 (6)	0.34192 (19)	0.6026 (2)	0.0168 (3)
O15	0.07431 (4)	0.27269 (13)	0.64579 (15)	0.0206 (3)
C151	0.07015 (7)	0.10541 (19)	0.6219 (2)	0.0236 (4)
C16	0.17474 (7)	0.26185 (18)	0.5325 (2)	0.0173 (3)
H3	0.4443	0.2993	0.2226	0.027*
H4	0.5012	0.0554	0.1722	0.033*
H5	0.4488	-0.1797	0.2602	0.033*
H17	0.2671	0.1358	0.4201	0.022*
H12	0.2684	0.5573	0.5141	0.022*
H13A	0.2715	0.7802	0.6577	0.035*
H13B	0.2320	0.9370	0.6431	0.035*
H13C	0.2398	0.8271	0.4699	0.035*
H14A	0.0824	0.7522	0.5186	0.037*
H14C	0.0240	0.7484	0.6367	0.037*
H14B	0.0335	0.6177	0.4844	0.037*
H15A	0.0755	0.0793	0.4947	0.035*
H15B	0.0310	0.0690	0.6588	0.035*

H15C	0.1010	0.0532	0.6957	0.035*
H16	0.1716	0.1519	0.5082	0.021*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0187 (2)	0.0200 (2)	0.0340 (3)	0.00169 (16)	0.00617 (18)	0.00149 (17)
C3	0.0173 (8)	0.0215 (9)	0.0285 (9)	-0.0005 (6)	0.0027 (7)	-0.0024 (7)
C4	0.0155 (9)	0.0281 (10)	0.0385 (11)	0.0004 (7)	0.0062 (7)	-0.0056 (8)
C5	0.0191 (9)	0.0234 (9)	0.0402 (11)	0.0049 (7)	0.0037 (7)	-0.0030 (8)
C2	0.0166 (8)	0.0193 (8)	0.0213 (8)	0.0007 (6)	-0.0011 (6)	-0.0017 (6)
C27	0.0179 (8)	0.0178 (8)	0.0190 (8)	-0.0003 (6)	0.0001 (6)	-0.0005 (6)
C271	0.0155 (8)	0.0248 (10)	0.0214 (9)	0.0035 (6)	0.0045 (6)	0.0006 (7)
N27	0.0247 (8)	0.0226 (9)	0.0386 (9)	-0.0001 (6)	0.0077 (7)	0.0028 (6)
C17	0.0180 (8)	0.0170 (8)	0.0202 (8)	-0.0001 (6)	0.0011 (6)	-0.0016 (6)
C11	0.0178 (8)	0.0188 (8)	0.0158 (8)	0.0018 (6)	0.0010 (6)	0.0005 (6)
C12	0.0164 (8)	0.0200 (8)	0.0199 (8)	-0.0014 (6)	0.0021 (6)	-0.0003 (6)
C13	0.0202 (8)	0.0156 (8)	0.0169 (8)	0.0016 (6)	0.0001 (6)	-0.0003 (6)
O13	0.0200 (6)	0.0166 (6)	0.0282 (6)	-0.0010 (4)	0.0056 (5)	-0.0038 (5)
C131	0.0267 (9)	0.0172 (8)	0.0262 (9)	-0.0046 (7)	0.0028 (7)	-0.0019 (7)
C14	0.0165 (8)	0.0176 (8)	0.0156 (8)	0.0033 (6)	0.0020 (6)	-0.0002 (6)
O14	0.0185 (6)	0.0186 (6)	0.0218 (6)	0.0062 (4)	0.0066 (5)	0.0021 (5)
C141	0.0213 (8)	0.0228 (9)	0.0296 (9)	0.0059 (7)	0.0023 (7)	0.0050 (7)
C15	0.0149 (7)	0.0197 (8)	0.0158 (7)	-0.0009 (6)	0.0008 (6)	0.0016 (6)
O15	0.0153 (6)	0.0181 (6)	0.0287 (6)	-0.0016 (4)	0.0057 (5)	-0.0018 (5)
C151	0.0211 (9)	0.0197 (9)	0.0303 (9)	-0.0049 (7)	0.0054 (7)	-0.0016 (7)
C16	0.0195 (8)	0.0150 (8)	0.0174 (8)	0.0011 (6)	0.0004 (6)	-0.0015 (6)

Geometric parameters (Å, °)

S1—C5	1.7100 (17)	C13—C14	1.400 (2)
S1—C2	1.7248 (17)	O13—C131	1.4243 (17)
C3—C2	1.383 (2)	C131—H13A	0.98
C3—C4	1.412 (2)	C131—H13B	0.98
C3—H3	0.95	C131—H13C	0.98
C4—C5	1.357 (2)	C14—O14	1.3792 (17)
C4—H4	0.95	C14—C15	1.398 (2)
C5—H5	0.95	O14—C141	1.4377 (19)
C2—C27	1.463 (2)	C141—H14A	0.98
C27—C17	1.359 (2)	C141—H14C	0.98
C27—C271	1.443 (2)	C141—H14B	0.98
C271—N27	1.145 (2)	C15—O15	1.3655 (17)
C17—C11	1.459 (2)	C15—C16	1.389 (2)
C17—H17	0.95	O15—C151	1.4299 (19)
C11—C12	1.399 (2)	C151—H15A	0.98
C11—C16	1.402 (2)	C151—H15B	0.98
C12—C13	1.390 (2)	C151—H15C	0.98
C12—H12	0.95	C16—H16	0.95

C13—O13	1.3658 (18)		
C5—S1—C2	92.03 (8)	O13—C131—H13A	109.5
C2—C3—C4	112.32 (15)	O13—C131—H13B	109.5
C2—C3—H3	123.8	H13A—C131—H13B	109.5
C4—C3—H3	123.8	O13—C131—H13C	109.5
C5—C4—C3	113.16 (15)	H13A—C131—H13C	109.5
C5—C4—H4	123.4	H13B—C131—H13C	109.5
C3—C4—H4	123.4	O14—C14—C13	120.47 (14)
C4—C5—S1	111.79 (13)	O14—C14—C15	119.86 (13)
C4—C5—H5	124.1	C15—C14—C13	119.60 (13)
S1—C5—H5	124.1	C14—O14—C141	112.76 (11)
C3—C2—C27	126.95 (15)	O14—C141—H14A	109.5
C3—C2—S1	110.71 (12)	O14—C141—H14C	109.5
C27—C2—S1	122.31 (11)	H14A—C141—H14C	109.5
C17—C27—C271	122.75 (14)	O14—C141—H14B	109.5
C17—C27—C2	123.37 (15)	H14A—C141—H14B	109.5
C271—C27—C2	113.88 (13)	H14C—C141—H14B	109.5
N27—C271—C27	176.82 (17)	O15—C15—C14	115.80 (12)
C27—C17—C11	131.72 (15)	O15—C15—C16	124.47 (14)
C27—C17—H17	114.1	C16—C15—C14	119.72 (13)
C11—C17—H17	114.1	C15—O15—C151	116.76 (12)
C12—C11—C16	119.31 (13)	O15—C151—H15A	109.5
C12—C11—C17	124.24 (14)	O15—C151—H15B	109.5
C16—C11—C17	116.41 (14)	H15A—C151—H15B	109.5
C13—C12—C11	119.90 (14)	O15—C151—H15C	109.5
C13—C12—H12	120.1	H15A—C151—H15C	109.5
C11—C12—H12	120.1	H15B—C151—H15C	109.5
O13—C13—C12	124.05 (14)	C15—C16—C11	120.81 (15)
O13—C13—C14	115.33 (13)	C15—C16—H16	119.6
C12—C13—C14	120.61 (15)	C11—C16—H16	119.6
C13—O13—C131	116.92 (12)		
C2—C3—C4—C5	0.0 (2)	C12—C13—O13—C131	7.9 (2)
C3—C4—C5—S1	-0.2 (2)	C14—C13—O13—C131	-173.66 (13)
C2—S1—C5—C4	0.32 (15)	O13—C13—C14—O14	0.4 (2)
C4—C3—C2—C27	178.10 (15)	C12—C13—C14—O14	178.93 (13)
C4—C3—C2—S1	0.26 (18)	O13—C13—C14—C15	-176.66 (13)
C5—S1—C2—C3	-0.33 (13)	C12—C13—C14—C15	1.8 (2)
C5—S1—C2—C27	-178.29 (14)	C15—C14—O14—C141	-107.39 (16)
C3—C2—C27—C17	173.35 (16)	C13—C14—O14—C141	75.54 (17)
S1—C2—C27—C17	-9.0 (2)	O14—C14—C15—O15	-0.3 (2)
C3—C2—C27—C271	-6.7 (2)	C13—C14—C15—O15	176.77 (13)
S1—C2—C27—C271	170.92 (12)	O14—C14—C15—C16	-179.56 (13)
C271—C27—C17—C11	-5.2 (3)	C13—C14—C15—C16	-2.5 (2)
C2—C27—C17—C11	174.72 (15)	C16—C15—O15—C151	2.2 (2)
C27—C17—C11—C12	-5.5 (3)	C14—C15—O15—C151	-176.98 (13)
C27—C17—C11—C16	176.90 (16)	O15—C15—C16—C11	-177.93 (13)

C16—C11—C12—C13	-1.3 (2)	C14—C15—C16—C11	1.2 (2)
C17—C11—C12—C13	-178.78 (14)	C12—C11—C16—C15	0.6 (2)
C11—C12—C13—O13	178.39 (14)	C17—C11—C16—C15	178.35 (13)
C11—C12—C13—C14	0.0 (2)		

(II) (E)-3-(4-bromophenyl)-2-(2-thienyl)acrylonitrile

Crystal data

C₁₃H₈BrNS

M_r = 290.17

Monoclinic *P*2₁/*n*

Hall symbol: -*P* 2₁ *yn*

a = 3.8557 (2) Å

b = 24.0484 (7) Å

c = 12.5466 (4) Å

β = 96.877 (2)°

V = 1154.99 (8) Å³

Z = 4

F(000) = 576

D_x = 1.669 Mg m⁻³

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 2598 reflections

θ = 2.4–27.5°

μ = 3.71 mm⁻¹

T = 120 K

Needle, colourless

0.38 × 0.04 × 0.03 mm

Data collection

Bruker Nonius KappaCCD area-detector diffractometer

Radiation source: Bruker Nonius FR591 rotating anode

Graphite monochromator

Detector resolution: 9.091 pixels mm⁻¹

φ and ω scans

Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)

T_{min} = 0.333, *T_{max}* = 0.897

12766 measured reflections

2598 independent reflections

1983 reflections with *I* > 2σ(*I*)

R_{int} = 0.059

θ_{\max} = 27.5°, θ_{\min} = 2.4°

h = -4→5

k = -31→30

l = -16→16

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2σ(*F*²)] = 0.040

wR(*F*²) = 0.081

S = 1.09

2598 reflections

147 parameters

4 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

w = 1/[σ²(*F_o*²) + (0.032*P*)² + 0.6935*P*]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} = 0.001

Δρ_{max} = 0.54 e Å⁻³

Δρ_{min} = -0.76 e Å⁻³

Extinction correction: *SHELXL97* (Sheldrick, 1997), *F_c** = *kF_c*[1 + 0.001*xF_c*²λ³/sin(2θ)]^{-1/4}

Extinction coefficient: 0.0211 (12)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{iso}</i> */ <i>U_{eq}</i>	Occ. (<1)
S1	0.2321 (3)	0.14823 (4)	0.54665 (9)	0.0221 (3)	0.798 (3)
C1	0.264 (6)	0.13968 (19)	0.5189 (9)	0.025*	0.202 (3)
C2	0.3661 (7)	0.16218 (11)	0.4236 (2)	0.0207 (6)	
C3	0.4157 (13)	0.11210 (18)	0.3703 (4)	0.0246 (6)	0.798 (3)
S3	0.4109 (14)	0.10914 (14)	0.3378 (3)	0.025*	0.202 (3)
C4	0.3362 (8)	0.06369 (12)	0.4311 (2)	0.0246 (6)	
C5	0.2416 (8)	0.07953 (13)	0.5257 (3)	0.0287 (7)	

C27	0.4262 (7)	0.21857 (12)	0.3872 (2)	0.0190 (6)	
C271	0.5318 (7)	0.22230 (12)	0.2807 (2)	0.0220 (7)	
N27	0.6257 (7)	0.22288 (11)	0.1977 (2)	0.0321 (7)	
C17	0.4092 (7)	0.26435 (12)	0.4483 (2)	0.0217 (6)	
C11	0.4573 (7)	0.32203 (12)	0.4150 (2)	0.0196 (6)	
C12	0.3298 (7)	0.34148 (12)	0.3129 (2)	0.0213 (6)	
C13	0.3837 (7)	0.39590 (12)	0.2839 (2)	0.0211 (6)	
C14	0.5699 (7)	0.43113 (12)	0.3564 (2)	0.0219 (7)	
Br14	0.66118 (7)	0.504938 (12)	0.31339 (3)	0.02715 (14)	
C15	0.6937 (7)	0.41383 (12)	0.4589 (3)	0.0251 (7)	
C16	0.6312 (8)	0.35932 (12)	0.4881 (2)	0.0249 (7)	
H1	0.2122	0.1629	0.5762	0.030*	0.202 (3)
H3	0.4935	0.1102	0.3014	0.029*	0.798 (3)
H4	0.3491	0.0263	0.4076	0.029*	
H5	0.1836	0.0535	0.5776	0.034*	
H17	0.3613	0.2587	0.5200	0.026*	
H12	0.2046	0.3170	0.2628	0.026*	
H13	0.2933	0.4090	0.2147	0.025*	
H15	0.8187	0.4387	0.5084	0.030*	
H16	0.7083	0.3472	0.5591	0.030*	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0225 (5)	0.0252 (5)	0.0195 (6)	0.0007 (4)	0.0058 (4)	0.0004 (4)
C2	0.0172 (14)	0.0268 (15)	0.0179 (16)	-0.0008 (11)	0.0013 (12)	-0.0009 (13)
C3	0.0257 (13)	0.0286 (13)	0.0185 (15)	0.0005 (10)	-0.0009 (11)	-0.0006 (11)
C4	0.0257 (13)	0.0286 (13)	0.0185 (15)	0.0005 (10)	-0.0009 (11)	-0.0006 (11)
C5	0.0211 (15)	0.0374 (18)	0.0268 (19)	-0.0053 (13)	-0.0005 (13)	0.0072 (15)
C27	0.0148 (13)	0.0295 (15)	0.0126 (15)	-0.0007 (11)	0.0009 (11)	0.0026 (13)
C271	0.0231 (15)	0.0223 (15)	0.0202 (18)	-0.0027 (12)	0.0012 (13)	-0.0028 (13)
N27	0.0411 (16)	0.0348 (15)	0.0222 (16)	-0.0014 (13)	0.0107 (13)	-0.0009 (13)
C17	0.0188 (14)	0.0299 (16)	0.0167 (16)	0.0002 (12)	0.0035 (12)	0.0009 (13)
C11	0.0168 (14)	0.0272 (15)	0.0157 (16)	0.0003 (12)	0.0055 (12)	-0.0010 (13)
C12	0.0176 (14)	0.0268 (16)	0.0195 (17)	-0.0016 (12)	0.0024 (12)	-0.0034 (13)
C13	0.0167 (14)	0.0280 (15)	0.0188 (17)	0.0019 (12)	0.0024 (12)	-0.0012 (13)
C14	0.0158 (14)	0.0244 (15)	0.0263 (18)	0.0000 (11)	0.0068 (12)	-0.0014 (14)
Br14	0.02422 (18)	0.02505 (19)	0.0323 (2)	-0.00274 (12)	0.00387 (13)	-0.00046 (14)
C15	0.0217 (15)	0.0301 (16)	0.0232 (18)	-0.0013 (13)	0.0016 (13)	-0.0073 (14)
C16	0.0243 (15)	0.0325 (17)	0.0177 (17)	0.0040 (13)	0.0014 (12)	-0.0003 (14)

Geometric parameters (\AA , $^\circ$)

S1—C5	1.674 (3)	C27—C271	1.446 (4)
S1—C2	1.718 (3)	C271—N27	1.143 (4)
C1—C2	1.411 (3)	C17—C11	1.467 (4)
C1—C5	1.452 (3)	C17—H17	0.95
C1—H1	0.95	C11—C16	1.396 (4)

C2—C3	1.402 (5)	C11—C12	1.397 (4)
C2—C27	1.458 (4)	C12—C13	1.381 (4)
C2—S3	1.691 (3)	C12—H12	0.95
C3—C4	1.445 (5)	C13—C14	1.380 (4)
C3—H3	0.95	C13—H13	0.95
S3—C4	1.652 (3)	C14—C15	1.382 (4)
C4—C5	1.338 (4)	C14—Br14	1.900 (3)
C4—H4	0.95	C15—C16	1.390 (4)
C5—H5	0.95	C15—H15	0.95
C27—C17	1.347 (4)	C16—H16	0.95
C5—S1—C2	92.13 (15)	C17—C27—C271	120.9 (3)
C2—C1—C5	117.2 (4)	C17—C27—C2	124.2 (3)
C2—C1—H1	121.4	C271—C27—C2	114.8 (2)
C5—C1—H1	121.4	N27—C271—C27	176.5 (3)
C3—C2—C1	98.2 (4)	C27—C17—C11	126.5 (3)
C3—C2—C27	127.9 (3)	C27—C17—H17	116.7
C1—C2—C27	133.9 (3)	C11—C17—H17	116.7
C1—C2—S3	108.1 (3)	C16—C11—C12	118.4 (3)
C27—C2—S3	118.0 (2)	C16—C11—C17	119.2 (3)
C3—C2—S1	109.5 (3)	C12—C11—C17	122.4 (3)
C27—C2—S1	122.6 (2)	C13—C12—C11	120.8 (3)
S3—C2—S1	119.3 (2)	C13—C12—H12	119.6
C2—C3—C4	113.0 (4)	C11—C12—H12	119.6
C2—C3—H3	123.5	C14—C13—C12	119.4 (3)
C4—C3—H3	123.5	C14—C13—H13	120.3
C4—S3—C2	90.5 (2)	C12—C13—H13	120.3
C5—C4—C3	109.7 (3)	C13—C14—C15	121.5 (3)
C5—C4—S3	122.0 (3)	C13—C14—Br14	119.1 (2)
C5—C4—H4	125.2	C15—C14—Br14	119.4 (2)
C3—C4—H4	125.2	C14—C15—C16	118.5 (3)
S3—C4—H4	112.7	C14—C15—H15	120.7
C4—C5—C1	102.0 (3)	C16—C15—H15	120.7
C4—C5—S1	115.7 (2)	C15—C16—C11	121.2 (3)
C4—C5—H5	122.1	C15—C16—H16	119.4
C1—C5—H5	135.9	C11—C16—H16	119.4
S1—C5—H5	122.1		
C5—C1—C2—C3	-1.3 (18)	C2—C1—C5—S1	-177 (6)
C5—C1—C2—C27	-178.3 (7)	C2—S1—C5—C4	-0.6 (3)
C5—C1—C2—S3	4 (2)	C2—S1—C5—C1	2 (4)
C5—C1—C2—S1	176 (7)	C3—C2—C27—C17	-172.3 (4)
C5—S1—C2—C3	-0.3 (3)	C1—C2—C27—C17	4.0 (15)
C5—S1—C2—C1	-3 (5)	S1—C2—C27—C17	5.2 (4)
C5—S1—C2—C27	-178.2 (2)	S3—C2—C27—C17	-178.0 (3)
C5—S1—C2—S3	5.0 (3)	C3—C2—C27—C271	3.6 (5)
C1—C2—C3—C4	1.6 (11)	C1—C2—C27—C271	179.8 (15)
C27—C2—C3—C4	178.9 (3)	S3—C2—C27—C271	-2.1 (4)

S3—C2—C3—C4	-154 (2)	S1—C2—C27—C271	-178.9 (2)
S1—C2—C3—C4	1.1 (4)	C271—C27—C17—C11	6.2 (5)
C3—C2—S3—C4	21.0 (16)	C2—C27—C17—C11	-178.1 (3)
C1—C2—S3—C4	-5.0 (11)	C27—C17—C11—C16	-142.4 (3)
C27—C2—S3—C4	176.5 (2)	C27—C17—C11—C12	38.7 (4)
S1—C2—S3—C4	-6.6 (4)	C16—C11—C12—C13	1.9 (4)
C2—C3—C4—C5	-1.5 (5)	C17—C11—C12—C13	-179.2 (2)
C2—C3—C4—S3	157.8 (18)	C11—C12—C13—C14	0.9 (4)
C2—S3—C4—C5	6.4 (4)	C12—C13—C14—C15	-2.4 (4)
C2—S3—C4—C3	-16.7 (13)	C12—C13—C14—Br14	177.1 (2)
C3—C4—C5—C1	0.6 (11)	C13—C14—C15—C16	0.9 (4)
S3—C4—C5—C1	-4.9 (11)	Br14—C14—C15—C16	-178.5 (2)
C3—C4—C5—S1	1.2 (4)	C14—C15—C16—C11	2.0 (4)
S3—C4—C5—S1	-4.2 (4)	C12—C11—C16—C15	-3.4 (4)
C2—C1—C5—C4	0.5 (19)	C17—C11—C16—C15	177.7 (3)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C17—H17...N27 ⁱ	0.95	2.55	3.450 (4)	159

Symmetry code: (i) $x-1/2, -y+1/2, z+1/2$.**(III) (*E*)-3-phenyl-2-(3-thienyl)acrylonitrile***Crystal data*C₁₃H₉NS $M_r = 211.27$ Monoclinic $P2_1/c$

Hall symbol: -P 2ybc

 $a = 9.6280$ (11) Å $b = 5.7190$ (3) Å $c = 19.247$ (2) Å $\beta = 103.129$ (7)° $V = 1032.09$ (17) Å³ $Z = 4$ $F(000) = 440$ $D_x = 1.360$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2368 reflections

 $\theta = 5.3$ – 27.5 ° $\mu = 0.27$ mm⁻¹ $T = 120$ K

Block, yellow

 $0.49 \times 0.31 \times 0.20$ mm*Data collection*Bruker Nonius KappaCCD area-detector
diffractometerRadiation source: Bruker Nonius FR591
rotating anode φ and ω scans

Absorption correction: multi-scan

[*SADABS* (Sheldrick, 2003) and *EVALCCD*
(Duisenberg *et al.*, 2003)] $T_{\min} = 0.878$, $T_{\max} = 0.947$

24963 measured reflections

2368 independent reflections

1788 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.036$ $\theta_{\max} = 27.5$ °, $\theta_{\min} = 5.3$ ° $h = -12 \rightarrow 12$ $k = -7 \rightarrow 7$ $l = -24 \rightarrow 25$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.067$

$wR(F^2) = 0.206$

$S = 1.07$

2368 reflections

136 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1146P)^2 + 1.443P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.57 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.67 \text{ e } \text{\AA}^{-3}$

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.18654 (8)	0.46218 (15)	0.47177 (4)	0.0376 (3)
C2	0.3419 (3)	0.3547 (5)	0.52137 (15)	0.0319 (6)
C3	0.3987 (3)	0.4916 (5)	0.57933 (14)	0.0269 (6)
C4	0.3108 (3)	0.6931 (5)	0.58268 (14)	0.0283 (6)
C5	0.1886 (3)	0.7005 (5)	0.52663 (13)	0.0285 (6)
C37	0.5316 (3)	0.4330 (5)	0.63047 (14)	0.0259 (6)
C371	0.5959 (3)	0.2150 (5)	0.61653 (14)	0.0302 (6)
N37	0.6417 (3)	0.0398 (4)	0.60331 (14)	0.0381 (6)
C17	0.5907 (3)	0.5662 (5)	0.68726 (14)	0.0270 (6)
C11	0.7234 (3)	0.5391 (4)	0.74236 (13)	0.0243 (5)
C12	0.8195 (3)	0.3550 (5)	0.74831 (15)	0.0302 (6)
C13	0.9417 (3)	0.3504 (5)	0.80161 (15)	0.0314 (6)
C14	0.9721 (3)	0.5248 (5)	0.85060 (13)	0.0278 (6)
C15	0.8791 (3)	0.7136 (5)	0.84729 (14)	0.0279 (6)
C16	0.7549 (3)	0.7200 (5)	0.79274 (14)	0.0271 (6)
H2	0.3849	0.2144	0.5102	0.038*
H4	0.3330	0.8087	0.6189	0.034*
H5	0.1177	0.8192	0.5200	0.034*
H17	0.5372	0.7018	0.6927	0.032*
H12	0.8004	0.2301	0.7150	0.036*
H13	1.0059	0.2230	0.8042	0.038*
H14	1.0569	0.5182	0.8872	0.033*
H15	0.8996	0.8359	0.8815	0.033*
H16	0.6912	0.8484	0.7898	0.032*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0342 (4)	0.0468 (5)	0.0295 (4)	-0.0054 (3)	0.0021 (3)	-0.0012 (3)
C2	0.0313 (13)	0.0355 (15)	0.0304 (13)	-0.0022 (12)	0.0101 (11)	-0.0026 (11)
C3	0.0229 (12)	0.0362 (14)	0.0229 (12)	-0.0037 (10)	0.0078 (10)	0.0043 (10)
C4	0.0282 (13)	0.0327 (14)	0.0240 (12)	-0.0011 (10)	0.0057 (10)	-0.0002 (10)
C5	0.0311 (13)	0.0344 (14)	0.0215 (12)	-0.0061 (11)	0.0090 (10)	-0.0027 (10)
C37	0.0246 (12)	0.0292 (13)	0.0247 (12)	-0.0032 (10)	0.0075 (10)	0.0007 (10)

C371	0.0295 (13)	0.0306 (14)	0.0304 (13)	-0.0038 (11)	0.0068 (11)	-0.0034 (11)
N37	0.0415 (14)	0.0319 (13)	0.0397 (14)	-0.0010 (11)	0.0065 (11)	-0.0073 (11)
C17	0.0254 (12)	0.0290 (13)	0.0283 (13)	-0.0003 (10)	0.0098 (10)	-0.0022 (10)
C11	0.0243 (12)	0.0283 (13)	0.0212 (12)	-0.0050 (9)	0.0070 (9)	-0.0007 (9)
C12	0.0332 (14)	0.0268 (13)	0.0320 (13)	0.0003 (11)	0.0103 (11)	-0.0051 (11)
C13	0.0295 (13)	0.0318 (14)	0.0352 (14)	0.0048 (11)	0.0121 (11)	0.0027 (11)
C14	0.0192 (11)	0.0433 (16)	0.0195 (11)	-0.0022 (10)	0.0016 (9)	0.0050 (10)
C15	0.0304 (13)	0.0304 (13)	0.0231 (12)	-0.0042 (10)	0.0067 (10)	-0.0059 (10)
C16	0.0245 (12)	0.0298 (13)	0.0273 (12)	0.0005 (10)	0.0066 (10)	-0.0031 (10)

Geometric parameters (Å, °)

S1—C2	1.696 (3)	C17—H17	0.95
S1—C5	1.722 (3)	C11—C12	1.389 (4)
C2—C3	1.371 (4)	C11—C16	1.403 (4)
C2—H2	0.95	C12—C13	1.374 (4)
C3—C4	1.440 (4)	C12—H12	0.95
C3—C37	1.464 (4)	C13—C14	1.358 (4)
C4—C5	1.405 (4)	C13—H13	0.95
C4—H4	0.95	C14—C15	1.395 (4)
C5—H5	0.95	C14—H14	0.95
C37—C17	1.348 (4)	C15—C16	1.401 (4)
C37—C371	1.444 (4)	C15—H15	0.95
C371—N37	1.146 (4)	C16—H16	0.95
C17—C11	1.471 (4)		
C2—S1—C5	93.22 (14)	C11—C17—H17	114.2
C3—C2—S1	112.9 (2)	C12—C11—C16	117.8 (2)
C3—C2—H2	123.6	C12—C11—C17	126.3 (2)
S1—C2—H2	123.6	C16—C11—C17	115.9 (2)
C2—C3—C4	111.3 (2)	C13—C12—C11	121.2 (2)
C2—C3—C37	122.9 (3)	C13—C12—H12	119.4
C4—C3—C37	125.8 (2)	C11—C12—H12	119.4
C5—C4—C3	112.9 (2)	C14—C13—C12	121.2 (3)
C5—C4—H4	123.6	C14—C13—H13	119.4
C3—C4—H4	123.6	C12—C13—H13	119.4
C4—C5—S1	109.7 (2)	C13—C14—C15	120.1 (2)
C4—C5—H5	125.1	C13—C14—H14	119.9
S1—C5—H5	125.1	C15—C14—H14	119.9
C17—C37—C371	121.2 (3)	C14—C15—C16	119.0 (2)
C17—C37—C3	124.0 (3)	C14—C15—H15	120.5
C371—C37—C3	114.7 (2)	C16—C15—H15	120.5
N37—C371—C37	177.1 (3)	C15—C16—C11	120.8 (2)
C37—C17—C11	131.5 (3)	C15—C16—H16	119.6
C37—C17—H17	114.2	C11—C16—H16	119.6
C5—S1—C2—C3	0.0 (2)	C3—C37—C17—C11	-178.1 (2)
S1—C2—C3—C4	-0.2 (3)	C37—C17—C11—C12	-1.7 (5)

S1—C2—C3—C37	179.25 (19)	C37—C17—C11—C16	177.4 (3)
C2—C3—C4—C5	0.4 (3)	C16—C11—C12—C13	-0.2 (4)
C37—C3—C4—C5	-179.1 (2)	C17—C11—C12—C13	178.9 (2)
C3—C4—C5—S1	-0.4 (3)	C11—C12—C13—C14	0.5 (4)
C2—S1—C5—C4	0.2 (2)	C12—C13—C14—C15	-0.3 (4)
C2—C3—C37—C17	179.0 (3)	C13—C14—C15—C16	-0.2 (4)
C4—C3—C37—C17	-1.6 (4)	C14—C15—C16—C11	0.5 (4)
C2—C3—C37—C371	-2.0 (4)	C12—C11—C16—C15	-0.3 (4)
C4—C3—C37—C371	177.3 (2)	C17—C11—C16—C15	-179.5 (2)
C371—C37—C17—C11	2.9 (4)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C2—H2...N37 ⁱ	0.95	2.59	3.324 (5)	135
C13—H13...Cg ⁱⁱ	0.95	2.86	3.566 (4)	132

Symmetry codes: (i) $-x+1, -y, -z+1$; (ii) $-x+2, y-1/2, -z+3/2$.