(*E*)-3-(4-Chlorophenyl)-2-(2-thienyl)acrylonitrile: chains built from C—H···N hydrogen bonds

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Key indicators

In the title compound, $C_{13}H_8ClNS$, the thiophene ring is disordered over two orientations. The molecules are linked into $C(5)$ chains by a single C—H···N hydrogen bond.
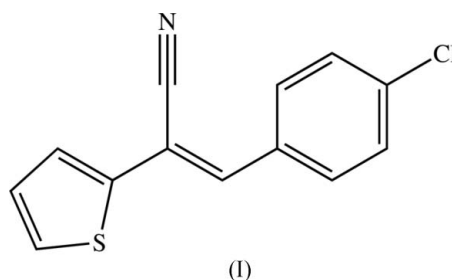
Comment

The title compound, (I), was prepared for use as an intermediate in the synthesis of new fused heterocyclic systems.

(I)

The thiophene rings are disordered over two orientations corresponding to a 180° rotation about the C2—C22 bond (Fig. 1); the two conformers have unequal occupancy, with refined site-occupancy factors for the major and minor conformers of 0.802 (3) and 0.198 (3), respectively. The bond distances show no unexpected values; in particular, the C—N and C—C distances for the nitrile unit (Table 1) are typical, where the mean values (Allen *et al.*, 1987) are 1.136 and 1.427 Å, respectively. The bond angles at C2 deviate significantly from regular trigonal geometry, while the C—C—N fragment is not quite linear. While both orientations of the thiophene unit are almost coplanar with the central C=C double bond, the 4-chlorophenyl ring is twisted well out of this plane. Thus, the dihedral angles between the plane of the central space unit C22—C2=C3—C31 and the planes of the adjacent rings are as follows: thiophene ring (major orientation) 5.7 (2)°, thiophene ring (minor orientation) 4.9 (5)° and aryl ring 38.2 (2)°.

The molecules are linked by a single C—H···N hydrogen bond (Table 2) into $C(5)$ (Bernstein *et al.*, 1995) chains generated by the *n*-glide planes and running parallel to the [10$\bar{1}$] direction (Fig. 2). Two such chains, related to one another by inversion, pass through each unit cell, but there are no direction-specific interactions between adjacent chains: in particular π–π stacking interactions, and C—H···π(arene) and C—H···π(thiophene) hydrogen bonds are all absent.

Experimental

A solution of 2-thiopheneacetonitrile (2.87 mmol) and potassium *tert*-butoxide (0.53 mmol) in anhydrous ethanol (3 ml) was stirred at

room temperature for 15 min; a solution of 4-chlorobenzaldehyde (2.87 mmol) in anhydrous ethanol (3 ml) was then added. The resulting precipitate was collected by filtration, washed with ethanol, dried and finally crystallized from dimethylformamide to give yellow crystals suitable for single-crystal X-ray diffraction.

Crystal data

$C_{13}H_8ClNS$	$D_x = 1.453$ Mg m^{-3}
$M_r = 245.72$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 2552
$a = 3.8142$ (2) Å	reflections
$b = 23.6852$ (8) Å	$\theta = 3.4$–27.5°
$c = 12.5319$ (5) Å	$\mu = 0.49$ mm^{-1}
$\beta = 97.2160$ (16)°	$T = 120$ (2) K
$V = 1123.17$ (8) Å^3	Block, yellow
$Z = 4$	$0.30 \times 0.20 \times 0.12$ mm

Data collection

Bruker–Nonius KappaCCD diffractometer	2552 independent reflections
	2132 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.033$
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	$\theta_{max} = 27.5°$
	$h = -4 \rightarrow 4$
$T_{min} = 0.866$, $T_{max} = 0.943$	$k = -30 \rightarrow 25$
11691 measured reflections	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0351P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.039$	$+ 0.9116P]$
$wR(F^2) = 0.094$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} = 0.001$
2552 reflections	$\Delta\rho_{max} = 0.55$ e Å^{-3}
145 parameters	$\Delta\rho_{min} = -1.02$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.147 (3)	C2—C3	1.346 (3)
C1—C2	1.448 (3)		
N1—C1—C2	176.2 (2)	C1—C2—C22	114.88 (15)
C1—C2—C3	120.69 (17)	C3—C2—C22	124.33 (15)
C3—C2—C22—S21	4.2 (3)	C2—C3—C31—C32	37.9 (3)
C3—C2—C22—S21A	−177.4 (2)		

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C3—H3$\cdots$N1^i	0.95	2.53	3.438 (2)	161

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

All H atoms were located in difference maps and then treated as riding atoms, with C—H = 0.95 Å and U_{iso}(H) = 1.2U_{eq}(C). It was apparent at an early stage that the thiophene unit was disordered over two sets of sites related by a rotation of 180° about the exocyclic C—C bond; the geometry of the minor orientation was restrained to match that of the major orientation, and the anisotropic displacement parameters for corresponding atoms in the two orientations were constrained to be equal. The deepest hole in the difference map is located 0.33 Å from the minor-occupancy atom S21A.

Data collection: COLLECT (Hooft, 1999); cell refinement: DENZO (Otwinowski & Minor, 1997) and COLLECT; data reduction: DENZO and COLLECT; program(s) used to solve structure:
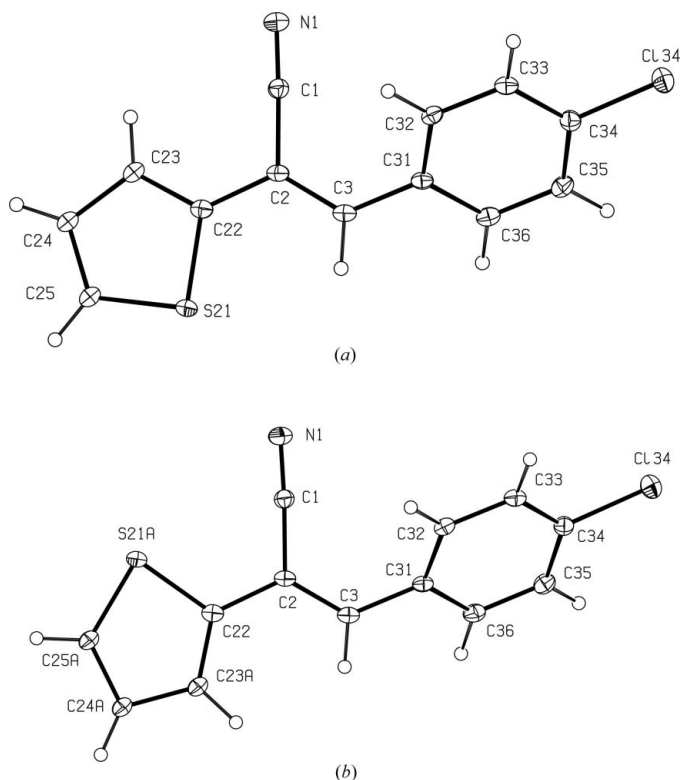

Figure 1
The molecule of compound (I), showing the atom-labelling scheme for (a) the major conformer and (b) the minor conformer. Displacement ellipsoids are drawn at the 30% probability level.
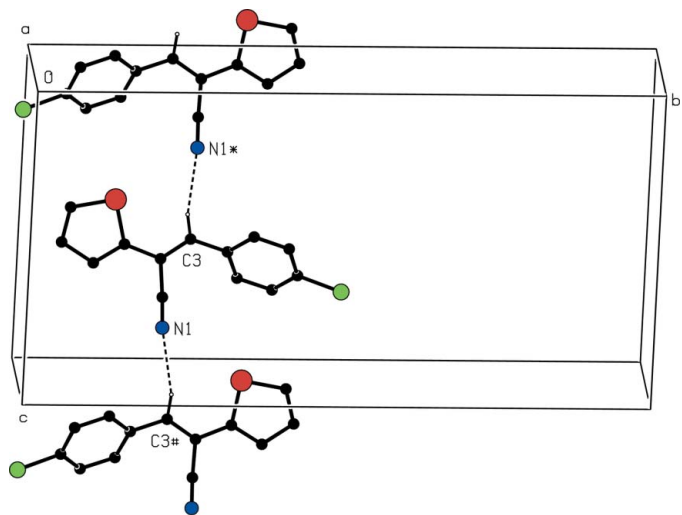

Figure 2
Part of the crystal structure of compound (I), showing the formation of a $C(5)$ chain along $[10\bar{1}]$ generated by the n-glide plane at $y = 0.25$. For the sake of clarity, H atoms not involved in the motif shown have been omitted, and only the major conformer is shown. Atoms marked with an asterisk (*) or a hash (#) 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.

OSCAIL (McArdle, 2003) and SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: OSCAIL and SHELXL97 (Sheldrick, 1997); molecular graphics: PLATON (Spek, 2003); software used to prepare material for publication: SHELXL97 and PRPKAPPA (Ferguson, 1999).

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