Rodier, N., Gillo, M.-P., Piessard, S. & Le Baut, G. (1986). *Acta Cryst.* C**42**, 1397–1402.
Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures.* University of Göttingen, Germany.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.

*Acta Cryst.* (1998). C**54**, 787–788

# *trans*-2-(2-Thienyl)-3-(5-phenyl-2-furyl)-propenenitrile

JÁN LOKAJ,[a] JARMILA ŠTETINOVÁ,[a] RUDOLF KADA,[a] VIKTOR KETTMANN[b] AND SALAM A. WARDA[c]

[a]*Faculty of Chemical Technology, Slovak Technical University, Radlinskeho 9, Bratislava, Slovakia 81237,* [b]*Faculty of Pharmacy, Comenius University, Odbojarov 10, Bratislava, Slovakia 83232, and* [c]*Department of Chemistry, Philipps University, Marburg, Germany 35032. E-mail: sivy@fpharm.uniba.sk*

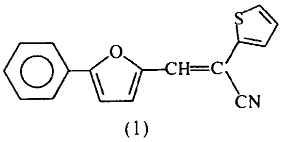
(*Received 6 February 1997; accepted 25 July 1997*)

## Abstract

The title compound, $C_{17}H_{11}NOS$, belongs to a series of extensively conjugated molecules which have potential as organic dyes. As indicated by the planarity of the molecule and bond-length patterns, the $\pi$-electron delocalization extends over the whole molecule. As expected, the arrangement of the phenylfuran and thiophene moieties at the double bond is *trans*.

## Comment

As part of our study on the relationships between molecular and electronic structures and absorption properties of extensively delocalized molecules as potential organic dyes, we have prepared a series of 1-(2-furyl)-2-cyanoethylenes. These molecules absorb in the near ultraviolet and visible regions of the spectrum, and exhibit pronounced colour changes depending on the nature of the substituents attached to the ethylene C atom and the furan ring. Consequently, it is of interest to examine the extent of conjugation in these molecules by the combined use of theoretical and experimental methods. In this communication, we report on the crystal structure of the title phenyl–thienyl derivative, (1).



(1)

As shown in Fig. 1, the furyl and thienyl functions are *trans*-positioned at the C5=C7 double bond. This bond [1.346 (3) Å] is considerably longer than the bond length of 1.314 (6) Å in unsubstituted ethylene (van Nes & Vos, 1977), but falls into a range of values generally observed for polarized ethylenes (Banerjee *et al.*, 1985). Bond lengths and angles within the furan and thiophene rings also agree well with those found in other compounds containing these molecular fragments (Sanni *et al.*, 1987; Gilmore *et al.*, 1983; Bak *et al.*, 1961; Pavelčík *et al.*, 1989).

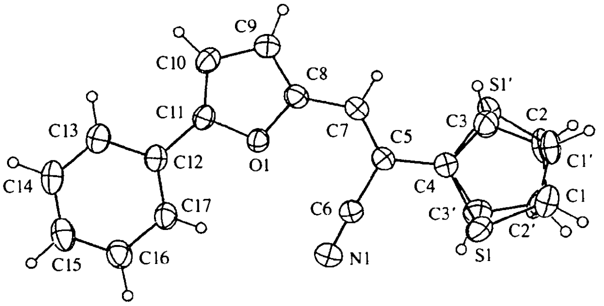


Fig. 1. *ORTEP*II (Johnson, 1976) view of the title molecule showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 50% probability level and H atoms are drawn as small circles of arbitrary radii. Both orientations of the disordered thiophene ring are shown.

The thiophene ring shows disorder; it adopts two different orientations related by a rotation of 172.3 (8)° about the C4—C5 bond. An unrestrained model of the disorder gave a reasonable fit to the experimental data but the resulting bond lengths and angles were unsatisfactory, particularly for the sites of lower occupancy. Therefore, restraints on both geometrical and displacement parameters as implemented in *SHELXL*93 (Sheldrick, 1993) were used. This resulted in a model with occupancies of 0.63 and 0.37 for the major and minor sites and a reasonable geometry for both rotamers.

The molecule as a whole is approximately planar. As expected, the furyl and phenyl rings are planar (r.m.s. deviations 0.000 and 0.003 Å, respectively), and the dihedral angle between these two planes is 2.8 (2)°. The twist about the ethylenic bond, as measured by the angle between the planes through C4, C5, C6 and H7, C7, C8 is 4.6 (9)°. While the furan ring lies in the plane of this double bond [dihedral angles 3.8 (4)°], the thiophene ring is substantially rotated out of the mean plane of the ethylenic bond [dihedral angle 13.2 (8) and 7.9 (9)° for the major and minor rotamers, respectively]. Thus, the planarity of the whole molecule is perturbed mainly by the thiophene moiety.

The extensive conjugation in the molecule is also observed in the C4—C5, C7—C8 and C11—C12 bond distances, which are all substantially shorter than the normal value of 1.487 Å reported for the $C_{sp^2}$—$C_{sp^2}$ single bond (Shmueli *et al.*, 1973).

## Experimental

Full details of the synthetic procedure will be published elsewhere (Štetinová *et al.*, 1998). Briefly, to a cooled ethanolic solution of 2-thienylacetonitrile was added an equimolar amount of 5-phenyl-2-furancarbaldehyde and 3–5 drops of 10% aqueous NaOH. After stirring the reaction mixture at ambient temperature for 2 h and pouring onto ice, the crude product was isolated and crystallized from methanol–water (2:1).

*Crystal data*

$C_{17}H_{11}NOS$
$M_r = 277.33$
Monoclinic
$P2_1/n$
$a = 10.456\,(2)$ Å
$b = 11.277\,(2)$ Å
$c = 12.676\,(3)$ Å
$\beta = 112.40\,(3)°$
$V = 1381.9\,(5)$ Å$^3$
$Z = 4$
$D_x = 1.333$ Mg m$^{-3}$
$D_m = 1.34\,(1)$ Mg m$^{-3}$
$D_m$ measured by flotation in bromoform–hexane

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 15 reflections
$\theta = 25–38°$
$\mu = 2.021$ mm$^{-1}$
$T = 293\,(2)$ K
Prism
0.40 × 0.25 × 0.20 mm
Pale yellow

*Data collection*

Syntex $P2_1$ diffractometer
$\theta/2\theta$ scans
Absorption correction: none
2214 measured reflections
2056 independent reflections
1878 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.043$

$\theta_{\rm max} = 59.96°$
$h = -11 \rightarrow 11$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 14$
2 standard reflections frequency: 100 min intensity decay: none

*Refinement*

Refinement on $F^2$
$R = 0.061$
$wR = 0.142$
$S = 1.095$
2056 reflections
219 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.1025P)^2 + 0.1198P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.035$
$\Delta\rho_{\rm max} = 0.558$ e Å$^{-3}$
$\Delta\rho_{\rm min} = -0.295$ e Å$^{-3}$
Extinction correction: *SHELXL*93
Extinction coefficient: 0.0090 (15)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| S1—C4 | 1.687 (2) | C6—N1 | 1.144 (3) |
| S1—C1 | 1.684 (13) | C7—C8 | 1.431 (3) |
| C1—C2 | 1.345 (11) | C8—C9 | 1.351 (3) |
| C2—C3 | 1.41 (2) | C8—O1 | 1.373 (2) |
| C3—C4 | 1.364 (11) | C9—C10 | 1.407 (3) |
| C4—C5 | 1.459 (3) | C10—C11 | 1.342 (3) |
| C5—C7 | 1.346 (3) | C11—O1 | 1.374 (2) |
| C5—C6 | 1.438 (3) | C11—C12 | 1.463 (3) |
| C4—S1—C1 | 93.7 (4) | C5—C7—C8 | 129.2 (2) |
| C2—C1—S1 | 110.9 (11) | C9—C8—O1 | 109.5 (2) |
| C1—C2—C3 | 111.8 (12) | C9—C8—C7 | 131.5 (2) |
| C4—C3—C2 | 113.0 (9) | O1—C8—C7 | 118.96 (15) |
| C3—C4—C5 | 128.6 (6) | C8—C9—C10 | 106.9 (2) |
| C3—C4—S1 | 109.5 (5) | C11—C10—C9 | 107.5 (2) |
| C5—C4—S1 | 121.82 (15) | C10—C11—O1 | 109.5 (2) |
| C7—C5—C6 | 121.3 (2) | C10—C11—C12 | 134.9 (2) |
| C7—C5—C4 | 123.5 (2) | O1—C11—C12 | 115.57 (15) |
| C6—C5—C4 | 115.25 (15) | C8—O1—C11 | 106.62 (13) |
| N1—C6—C5 | 176.2 (2) | | |

The H atoms of the disordered thiophene ring were placed in calculated positions and allowed to refine as riding models on their associated C atoms, with $U_{\rm iso}$ set to 1.2 times the $U_{\rm eq}$ of the parent atom. All other H atoms were located in a difference map and fixed at these positions with $U_{\rm iso}$ set to 1.2 times the $U_{\rm eq}$ of the associated atom. The disorder of the thiophene ring was modelled by resolving the positions of the S1, C1, C2 and C3 atoms into two components, and using a total of 54 restraints on equivalent bond distances and anisotropic displacement parameters [a combination of *SAME*, *DELU* and *SIMU* options in *SHELXL*93 (Sheldrick, 1993)]. The refined occupancies of the major (unprimed) and minor (primed) sites were 62.6 (4) and 37.4 (4)%, respectively.

Data collection: Syntex $P2_1$ diffractometer software. Cell refinement: Syntex $P2_1$ diffractometer software. Data reduction: *XP*21 (Pavelčík, 1987). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93. Molecular graphics: *ORTEP*II (Johnson, 1976) . Software used to prepare material for publication: *SHELXL*93.

---

Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1199). Services for accessing these data are described at the back of the journal.

---

## References

Bak, B., Christensen, D., Hansen-Nygaard, L. & Rastrap-Anderson, J. (1961). *J. Mol. Spectrosc.* **7**, 58–64.

Banerjee, T., Chaudhuri, S. & Muthiah, P. T. (1985). *Acta Cryst.* C**41**, 269–271.

Gilmore, C. J., Mallinson, P. R. & Speakman, J. C. (1983). *Acta Cryst.* C**39**, 1111–1113.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Nes, G. J. K. van & Vos, A. (1977). *Acta Cryst.* B**33**, 1653–1654.

Pavelčík, F. (1987). *XP*21. *Program for Syntex $P2_1$ Data Reduction.* Comenius University, Bratislava, Slovakia.

Pavelčík, F., Sivý, J., Havránek, E., Sivý, P., Komanová, E. & Nevýdal, J. (1989). *Acta Cryst.* C**45**, 836–837.

Sanni, S. B., Behm, H., Beurskens, P. T. & Adesogan, E. K. (1987). *Acta Cryst.* C**43**, 555–558.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.

Shmueli, U., Shanan-Atidi, H., Horwitz, H. & Shvo, Y. (1973). *J. Chem. Soc. Perkin Trans.* 2, pp. 657–652.

Štetinová, J., Kada, R. & Leško, J. (1998). *Collect. Czech. Chem. Commun.* In preparation.