

4-(2-Thienyl)-1H-pyrrole-2-carbaldehyde

Rohan A. Davis,^a Anthony R. Carroll,^a Ronald J. Quinn,^a Peter C. Healy^{a*} and Edward R. Tiekink^{b*‡}^aEskitis Institute of Cell and Molecular Therapies, Griffith University, Nathan, Queensland 4111, Australia, and ^bSchool of Science, Griffith University, Nathan, Queensland 4111, Australia

‡ Present address: Department of Chemistry, The University of Texas at San Antonio, 6900 North Loop 1604 West, San Antonio, Texas 78249-0698, USA.

Correspondence e-mail:
p.healy@griffith.edu.au,
Edward.Tiekink@utsa.edu

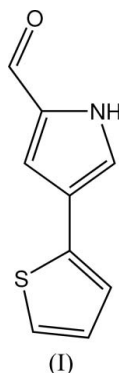
Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
Disorder in main residue
 R factor = 0.036
 wR factor = 0.112
Data-to-parameter ratio = 12.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The thienyl and pyrrole rings in the molecule of the title compound, $\text{C}_9\text{H}_7\text{NOS}$, are not coplanar, their planes forming a dihedral angle of $14.7(3)^\circ$; the C—C bond linking the rings almost coincides with the line of intersection of the planes of the rings. The molecules in the crystal structure form centrosymmetric dimeric aggregates, held together by means of $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds.

Comment

The title compound has been utilized as a template in the synthesis of combinatorial libraries (Davis *et al.*, 2002). The molecular structure (Fig. 1) shows that while both pyrrole and thienyl rings are essentially planar (r.m.s. deviations = 0.002 Å for each), there is a twist in the molecule about the C4—C41 bond, as evidenced by the C3—C4—C41—S42 torsion angle of $-13.8(4)^\circ$; the dihedral angle formed by the planes of the two rings is $14.7(3)^\circ$. The molecules are linked into centrosymmetric pairs *via* $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds [$\text{H1}\cdots\text{O21}^i = 2.03$ Å, $\text{N1}\cdots\text{O21}^i = 2.861(3)$ Å and $\text{N1}-\text{H1}\cdots\text{O21}^i = 163^\circ$; symmetry code: (i) $-x, -y, 2 - z$; Fig. 1].

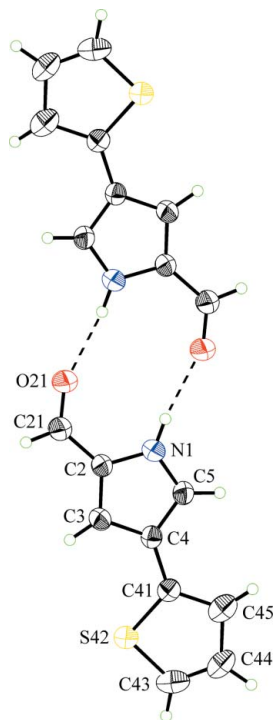


Experimental

The compound was prepared by the Suzuki–Miyaura coupling reaction as reported by Davis *et al.* (2002). Crystals suitable for X-ray diffraction studies were obtained by the slow evaporation of a dichloromethane solution of the compound; m.p. 456–458 K.

Crystal data

$\text{C}_9\text{H}_7\text{NOS}$	$D_x = 1.393$ Mg m ⁻³
$M_r = 177.22$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 25 reflections
$a = 6.303(2)$ Å	$\theta = 11.4\text{--}18.5^\circ$
$b = 7.745(3)$ Å	$\mu = 0.33$ mm ⁻¹
$c = 17.424(1)$ Å	$T = 293(2)$ K
$\beta = 96.64(1)^\circ$	Block, orange
$V = 844.9(4)$ Å ³	$0.50 \times 0.20 \times 0.10$ mm
$Z = 4$	


Figure 1

The structure of the centrosymmetric dimer formed *via* N—H...O hydrogen bonds, showing the crystallographic numbering scheme. Displacement ellipsoids are drawn at the 35% probability level. Minor components of the disordered atoms have been omitted. The symmetry-related molecule is derived using the $(-x, -y, 2 - z)$ transformation.

Data collection

Rigaku AFC-7R diffractometer
 ω - 2θ scans
 Absorption correction: none
 1641 measured reflections
 1496 independent reflections
 883 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$

$\theta_{\text{max}} = 25.0^\circ$
 $h = 0 \rightarrow 7$
 $k = 0 \rightarrow 9$
 $l = -20 \rightarrow 20$
 3 standard reflections
 every 150 reflections
 intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.112$
 $S = 1.01$
 1496 reflections
 117 parameters
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0512P)^2 + 0.0355P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.14 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.23 \text{ e } \text{\AA}^{-3}$

The H atoms were included in the riding-model approximation, with distances N—H = 0.86 Å and C—H = 0.93 Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N,C})$. The thienyl ring is disordered over two positions related by a 180° rotation around the C4—C41 bond. This disorder gives rise to two positions for each of the S42 and C45 atoms; the refinement of their occupancies showed that one of these positions is predominant, with an occupancy of 0.795 (3). The positions of C43 and C44 are effectively not affected by the disorder.

Data collection: *MSC/AFC Diffractometer Control* (Molecular Structure Corporation, 1996); cell refinement: *MSC/AFC Diffractometer Control*; data reduction: *TEXSAN for Windows* (Molecular Structure Corporation, 1999); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *TEXSAN for Windows*.

The Queensland Government is thanked for the award of a Smart Returns Fellowship (ERTT).

References

- Altomare, A., Cascarano, M., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
 Davis, R. A., Carroll, A. R. & Quinn, R. J. (2002). *Aust. J. Chem.* **55**, 789–794.
 Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
 Molecular Structure Corporation (1996). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
 Molecular Structure Corporation (1999). *TEXSAN for Windows*. Version 1.05. MSC, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.
 Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.