

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

(E)-2-[2-(2-Thienyl)vinyl]-1H-1,3-benzimidazole

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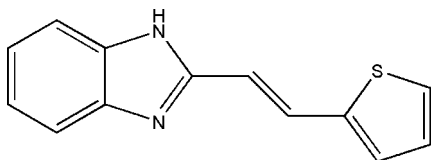
Received 27 January 2008; accepted 7 March 2008

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; R factor = 0.085; wR factor = 0.214; data-to-parameter ratio = 18.2.

In the title compound, $\text{C}_{13}\text{H}_{10}\text{N}_2\text{S}$, the dihedral angle between the imidazole and thiophene rings is 16.89 (19)°, and the double bond adopts an *E* configuration. In the crystal structure, $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds link the molecules into rows along *b*. There is also evidence of weak $\text{C}-\text{H}\cdots\text{S}$ interactions.

Related literature

For general background, see: Huang *et al.* (2003); Wang *et al.* (2005); Ye *et al.* (2006, 2007). For the crystal structures of related compounds, see: Ozbey *et al.* (1998); Li & Clarkson (2007).



Experimental

Crystal data

$\text{C}_{13}\text{H}_{10}\text{N}_2\text{S}$
 $M_r = 226.06$
 Orthorhombic, *Pnma*
 $a = 12.239$ (2) Å
 $b = 16.389$ (3) Å
 $c = 11.487$ (2) Å

$V = 2304.1$ (7) Å³
 $Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 0.25$ mm⁻¹
 $T = 293$ (2) K
 $0.15 \times 0.10 \times 0.07$ mm

Data collection

Rigaku Mercury2 diffractometer
 Absorption correction: multi-scan
 (*CrystalClear*; Rigaku, 2005)
 $T_{\text{min}} = 0.796$, $T_{\text{max}} = 1.000$
 (expected range = 0.782–0.983)

21849 measured reflections
 2637 independent reflections
 1360 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.145$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.084$
 $wR(F^2) = 0.214$
 $S = 1.07$
 2637 reflections
 145 parameters

1 restraint
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.22$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.28$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| <i>D</i> — <i>H</i> ··· <i>A</i> | <i>D</i> — <i>H</i> | <i>H</i> ··· <i>A</i> | <i>D</i> ··· <i>A</i> | <i>D</i> — <i>H</i> ··· <i>A</i> |
|----------------------------------|---------------------|-----------------------|-----------------------|----------------------------------|
| C11—H11A···S1 | 0.93 | 2.76 | 3.161 (4) | 107 |
| N1—H1B···N1 ¹ | 0.86 | 2.01 | 2.865 (6) | 170 |
| N2—H2B···N2 ⁱⁱ | 0.86 | 2.11 | 2.906 (5) | 154 |

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

This work was supported by a Start-up Grant from Southeast University (to YQ).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2460).

References

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supplementary materials

Acta Cryst. (2008). E64, o964 [doi:10.1107/S1600536808006405]

(*E*)-2-[2-(2-Thienyl)vinyl]-1*H*-1,3-benzimidazole

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Comment

It has been generally accepted that imidazole groups play an important role in coordination chemistry (Huang *et al.*, 2003). A flexible ligand readily induces coordination compounds to crystallize in non-centrosymmetric space groups, which makes it possible to investigate their interesting physical properties such as second harmonic generation, ferroelectric and piezoelectric properties (Wang *et al.*, 2005). As a continuation of our work in this field, (Ye *et al.*, 2006, 2007), we have synthesized the title compound, 1, Fig 1.

The title compound, C₁₂H₁₀N₂S, was successfully prepared through the reaction between 2-methyl-1*H*-benzo[*d*]imidazole and thiophene-2-carbaldehyde. It adopts a *trans* configuration about the C9=C11 bond and the dihedral angle between the mean plane of the imidazole ring and thiophenyl ring is 16.89 (19)°. The crystal packing is dominated by N—H⋯N interactions linking the molecules into rows along *b*, Fig 2. There is also evidence of weak C—H⋯S interactions.

Experimental

2-methyl-1*H*-benzo[*d*]imidazole (10 mmol, 1.32 g) and thiophene-2-carbaldehyde (45 mmol, 5.04 g) were reacted as a melt at 180°C with stirring for 18 h. Then 20 ml 2-propanol and 1.5 g oxalic acid were added to the reaction mixture, the solution filtered and the precipitate washed with copious quantities of boiling water. The pH was adjusted to 8–9 with ammonia to afford the title compound as a pale-yellow solid powder. Crystals suitable for single-crystal X-ray diffraction studies were obtained by slow evaporation of a solution in ethanol at room temperature over several days.

Refinement

All carbon-bound H atoms were positioned geometrically, with C—H = 0.93 Å and included in the refinement as riding, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}$. The H atoms attached to N were found in the difference Fourier map and were subsequently treated as riding atoms, with N—H = 0.86 Å, and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$.

Figures

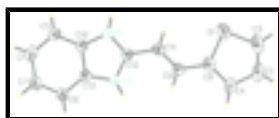


Fig. 1. The molecular structure of the compound with the atomic numbering scheme. Displacement ellipsoids were drawn at the 30% probability level.

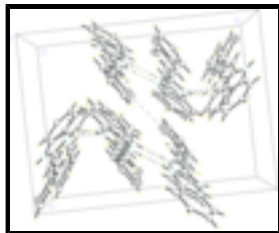


Fig. 2. The crystal packing of 1 with hydrogen bonds drawn as dashed lines.

(E)-2-[2-(2-Thienyl)vinyl]-1H-1,3-benzimidazole

Crystal data

$C_{13}H_{10}N_2S$

$M_r = 226.06$

Orthorhombic, *Pnna*

Hall symbol: -P 2a 2bc

$a = 12.239 (2) \text{ \AA}$

$b = 16.389 (3) \text{ \AA}$

$c = 11.487 (2) \text{ \AA}$

$V = 2304.1 (7) \text{ \AA}^3$

$Z = 8$

$F_{000} = 944$

$D_x = 1.305 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 14820 reflections

$\theta = 3.0\text{--}29.2^\circ$

$\mu = 0.25 \text{ mm}^{-1}$

$T = 293 (2) \text{ K}$

Block, colorless

$0.15 \times 0.10 \times 0.07 \text{ mm}$

Data collection

Mercury2 (2x2 bin mode)
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: $13.6612 \text{ pixels mm}^{-1}$

$T = 293(2) \text{ K}$

CCD profile fitting scans

Absorption correction: multi-scan
(CrystalClear; Rigaku, 2005)

$T_{\min} = 0.796$, $T_{\max} = 1.000$

21849 measured reflections

2637 independent reflections

1360 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.145$

$\theta_{\text{max}} = 27.5^\circ$

$\theta_{\text{min}} = 3.3^\circ$

$h = -15 \rightarrow 15$

$k = -21 \rightarrow 21$

$l = -14 \rightarrow 14$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.084$

$wR(F^2) = 0.214$

$S = 1.07$

2637 reflections

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.081P)^2 + 0.7151P]$

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

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

$\Delta\rho_{\text{max}} = 0.22 \text{ e \AA}^{-3}$

145 parameters

$$\Delta\rho_{\min} = -0.28 \text{ e } \text{\AA}^{-3}$$

1 restraint

Extinction correction: none

Primary atom site location: structure-invariant direct methods

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

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

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|------|--------------|--------------|--------------|----------------------------------|-----------|
| S1 | 0.88515 (10) | 0.06908 (7) | 0.51647 (11) | 0.0801 (5) | |
| N1 | 0.6821 (2) | 0.07121 (18) | 0.1000 (3) | 0.0577 (9) | |
| H1B | 0.7264 | 0.0309 | 0.0924 | 0.069* | 0.50 |
| N2 | 0.5945 (2) | 0.17665 (16) | 0.1790 (2) | 0.0452 (7) | |
| H2B | 0.5751 | 0.2134 | 0.2283 | 0.054* | 0.50 |
| C1 | 0.5111 (4) | 0.1178 (3) | -0.1555 (4) | 0.0824 (14) | |
| H1A | 0.4956 | 0.1028 | -0.2319 | 0.099* | |
| C2 | 0.8179 (4) | 0.1317 (3) | 0.7051 (4) | 0.0898 (15) | |
| H2A | 0.8136 | 0.1453 | 0.7836 | 0.108* | |
| C3 | 0.8987 (5) | 0.0855 (3) | 0.6610 (4) | 0.0890 (16) | |
| H3A | 0.9559 | 0.0647 | 0.7053 | 0.107* | |
| C4 | 0.7391 (3) | 0.1583 (3) | 0.6209 (3) | 0.0653 (11) | |
| H4A | 0.6790 | 0.1913 | 0.6364 | 0.078* | |
| C5 | 0.5901 (4) | 0.0750 (3) | -0.0942 (4) | 0.0753 (13) | |
| H5A | 0.6278 | 0.0316 | -0.1277 | 0.090* | |
| C6 | 0.4751 (3) | 0.2076 (2) | 0.0054 (3) | 0.0604 (11) | |
| H6A | 0.4376 | 0.2514 | 0.0381 | 0.073* | |
| C7 | 0.4546 (4) | 0.1825 (3) | -0.1057 (4) | 0.0720 (12) | |
| H7A | 0.4015 | 0.2094 | -0.1492 | 0.086* | |
| C8 | 0.7672 (3) | 0.1264 (2) | 0.5105 (3) | 0.0581 (10) | |
| C9 | 0.7079 (3) | 0.1380 (2) | 0.4039 (3) | 0.0532 (10) | |
| H9A | 0.6461 | 0.1709 | 0.4074 | 0.064* | |
| C10 | 0.6101 (3) | 0.0995 (2) | 0.0186 (3) | 0.0515 (9) | |
| C11 | 0.7328 (3) | 0.1064 (2) | 0.3007 (3) | 0.0526 (10) | |
| H11A | 0.7950 | 0.0740 | 0.2961 | 0.063* | |
| C12 | 0.6700 (3) | 0.1188 (2) | 0.1941 (3) | 0.0479 (9) | |
| C13 | 0.5538 (3) | 0.1655 (2) | 0.0678 (3) | 0.0461 (9) | |

supplementary materials

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| S1 | 0.0830 (9) | 0.0654 (8) | 0.0918 (10) | -0.0012 (6) | -0.0304 (7) | 0.0032 (6) |
| N1 | 0.0607 (19) | 0.0583 (19) | 0.054 (2) | 0.0158 (16) | -0.0072 (15) | -0.0140 (16) |
| N2 | 0.0532 (17) | 0.0401 (16) | 0.0422 (17) | 0.0063 (13) | 0.0015 (13) | -0.0027 (13) |
| C1 | 0.097 (3) | 0.096 (4) | 0.054 (3) | 0.004 (3) | -0.017 (3) | -0.015 (3) |
| C2 | 0.113 (4) | 0.106 (4) | 0.050 (3) | -0.026 (3) | -0.023 (2) | 0.013 (3) |
| C3 | 0.112 (4) | 0.076 (3) | 0.079 (4) | -0.023 (3) | -0.043 (3) | 0.023 (3) |
| C4 | 0.070 (3) | 0.081 (3) | 0.045 (2) | -0.018 (2) | -0.0037 (18) | 0.001 (2) |
| C5 | 0.083 (3) | 0.079 (3) | 0.064 (3) | 0.018 (2) | -0.011 (2) | -0.028 (2) |
| C6 | 0.072 (3) | 0.054 (2) | 0.055 (2) | 0.009 (2) | -0.009 (2) | 0.001 (2) |
| C7 | 0.081 (3) | 0.071 (3) | 0.064 (3) | 0.009 (2) | -0.021 (2) | 0.007 (2) |
| C8 | 0.063 (2) | 0.051 (2) | 0.061 (3) | -0.0153 (19) | -0.014 (2) | 0.0087 (19) |
| C9 | 0.055 (2) | 0.049 (2) | 0.056 (3) | -0.0009 (18) | -0.0023 (18) | 0.0054 (18) |
| C10 | 0.053 (2) | 0.051 (2) | 0.051 (2) | 0.0034 (18) | -0.0051 (18) | -0.0070 (18) |
| C11 | 0.051 (2) | 0.048 (2) | 0.059 (3) | 0.0005 (17) | -0.0004 (19) | -0.0009 (18) |
| C12 | 0.048 (2) | 0.049 (2) | 0.047 (2) | -0.0011 (17) | 0.0016 (17) | 0.0022 (17) |
| C13 | 0.052 (2) | 0.0418 (19) | 0.044 (2) | -0.0023 (17) | 0.0020 (17) | 0.0001 (16) |

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

| | | | |
|------------|-----------|------------|-----------|
| S1—C3 | 1.690 (5) | C4—C8 | 1.415 (5) |
| S1—C8 | 1.723 (4) | C4—H4A | 0.9300 |
| N1—C12 | 1.341 (4) | C5—C10 | 1.379 (5) |
| N1—C10 | 1.366 (4) | C5—H5A | 0.9300 |
| N1—H1B | 0.8600 | C6—C7 | 1.365 (5) |
| N2—C12 | 1.335 (4) | C6—C13 | 1.385 (5) |
| N2—C13 | 1.383 (4) | C6—H6A | 0.9300 |
| N2—H2B | 0.8600 | C7—H7A | 0.9300 |
| C1—C5 | 1.386 (6) | C8—C9 | 1.436 (5) |
| C1—C7 | 1.390 (6) | C9—C11 | 1.329 (5) |
| C1—H1A | 0.9300 | C9—H9A | 0.9300 |
| C2—C3 | 1.344 (6) | C10—C13 | 1.401 (5) |
| C2—C4 | 1.435 (6) | C11—C12 | 1.461 (4) |
| C2—H2A | 0.9300 | C11—H11A | 0.9300 |
| C3—H3A | 0.9300 | | |
| C3—S1—C8 | 92.0 (3) | C7—C6—H6A | 121.3 |
| C12—N1—C10 | 106.4 (3) | C13—C6—H6A | 121.3 |
| C12—N1—H1B | 126.8 | C6—C7—C1 | 121.6 (4) |
| C10—N1—H1B | 126.8 | C6—C7—H7A | 119.2 |
| C12—N2—C13 | 106.0 (3) | C1—C7—H7A | 119.2 |
| C12—N2—H2B | 127.0 | C4—C8—C9 | 126.3 (4) |
| C13—N2—H2B | 127.0 | C4—C8—S1 | 111.7 (3) |
| C5—C1—C7 | 121.6 (4) | C9—C8—S1 | 122.0 (3) |
| C5—C1—H1A | 119.2 | C11—C9—C8 | 126.3 (4) |
| C7—C1—H1A | 119.2 | C11—C9—H9A | 116.8 |

| | | | |
|------------|-----------|--------------|-----------|
| C3—C2—C4 | 114.3 (4) | C8—C9—H9A | 116.8 |
| C3—C2—H2A | 122.8 | N1—C10—C5 | 131.2 (4) |
| C4—C2—H2A | 122.8 | N1—C10—C13 | 107.7 (3) |
| C2—C3—S1 | 112.8 (4) | C5—C10—C13 | 121.1 (4) |
| C2—C3—H3A | 123.6 | C9—C11—C12 | 125.0 (3) |
| S1—C3—H3A | 123.6 | C9—C11—H11A | 117.5 |
| C8—C4—C2 | 109.2 (4) | C12—C11—H11A | 117.5 |
| C8—C4—H4A | 125.4 | N1—C12—N2 | 112.7 (3) |
| C2—C4—H4A | 125.4 | N1—C12—C11 | 122.5 (3) |
| C1—C5—C10 | 117.0 (4) | N2—C12—C11 | 124.9 (3) |
| C1—C5—H5A | 121.5 | N2—C13—C6 | 131.5 (3) |
| C10—C5—H5A | 121.5 | N2—C13—C10 | 107.3 (3) |
| C7—C6—C13 | 117.5 (4) | C6—C13—C10 | 121.2 (3) |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|----------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| C11—H11A \cdots S1 | 0.93 | 2.76 | 3.161 (4) | 107 |
| N1—H1B \cdots N1 ⁱ | 0.86 | 2.01 | 2.865 (6) | 170 |
| N2—H2B \cdots N2 ⁱⁱ | 0.86 | 2.11 | 2.906 (5) | 154 |

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

Fig. 1

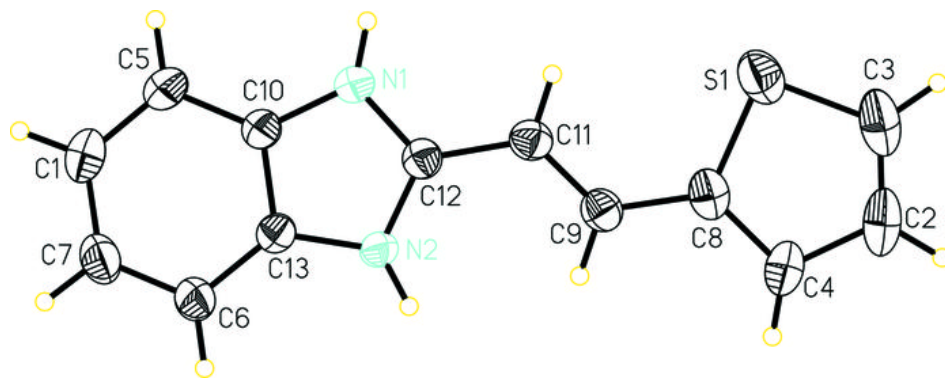


Fig. 2

