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2-(2-Pyrrolyl)-1,3-benzothiazole

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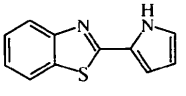
Abstract

The title compound, $C_{11}H_8N_2S$, already known from the literature, was prepared and studied by X-ray diffraction. The five-membered heterocyclic ring is coplanar with its fused benzene ring. However, the molecule itself is non-planar, with the dihedral angle between the planes of the 2-benzothiazolyl and pyrrolyl groups being 17.3 (7)°. The molecules related by the twofold rotation axis are connected in pairs by N—H···N hydrogen bonds of 3.032 (9) Å.

Comment

Organic luminescent materials are widely used in the basic as well as applied research fields and various kinds of luminescent compounds are now known. Among this group of compounds are benzothiazole derivatives, whose importance as substances used as fluorescent brighteners (Allen, 1971) and for fluorometric measurements (Akiyama *et al.*, 1987) has led us to study the spectral characteristics of some aryl- and heteroaryl-substituted benzothiazoles (Tralić-Kulenović *et al.*, 1993). 2-(2-Pyrrolyl)benzothiazole (Brown, 1962) exhibited a relatively large fluorescence quantum efficiency and it was therefore of interest to find out more about the structure of this compound, (I).



(I)

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The bond distances in both five-membered heterocyclic rings are consistent with those usually found in the literature (Potenza & Mastropaolo, 1974; Rudd & Barany, 1984). The benzene ring and its fused thiazolyl ring are coplanar. The bond S—C11 [1.739 (3) Å] is shorter than S—C5 [1.754 (3) Å], due to the fact that C5 is sp^2 hybridized, whereas C11 is part of the aromatic ring (Yeap *et al.*, 1991). Therefore, the molecular structure is not planar; the dihedral angle between the planes of the 2-benzothiazolyl and pyrrolyl groups is 17.3 (7)°. The deviation from coplanarity is significant; the torsion angles N1—C1—C5—N2, N1—C1—C5—S, C4—C1—C5—N2 and C4—C1—C5—S are 11.1 (4), −172.4 (2), −159.6 (3) and 16.9 (5)°, respectively.

The molecules, related by the twofold rotation axis, are connected in pairs by two hydrogen bonds [NH···N 3.032 (9), H···N 2.169 (7) Å and N—H···N 166.0 (7)°].

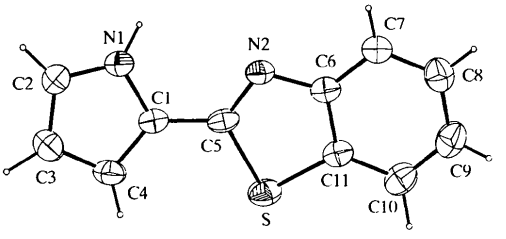

Fig. 1. View of the title molecule with the atom-labelling scheme for the non-H atoms. Displacement ellipsoids are drawn at the 50% probability level.

Experimental

The title compound was prepared from aminothiophenole and the required heteroaldehyde according to the method of Bogert & Stull (1935). Good quality crystals for X-ray diffraction analysis were obtained by slow evaporation from a dimethylformamide solution.

Crystal data

$C_{11}H_8N_2S$
M_r = 200.25
Tetragonal
$P4_32_12$
a = 8.0916 (9) Å
c = 30.078 (4) Å
V = 1969.3 (4) Å^3
Z = 8
D_x = 1.351 Mg m^{-3}
D_m = 1.3 Mg m^{-3}
D_m measured by flotation in benzene/CCl_4
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 32 reflections
θ = 7.8–13.1°
μ = 0.285 mm^{-1}
T = 293 (2) K
Bipyramid
0.135 × 0.120 × 0.113 mm
Bronze

Data collection

Philips PW1100 updated by Stoe diffractometer
R_{int} = 0.050
θ_{max} = 28.99°

ω–2θ scans
Absorption correction: none
5310 measured reflections
2791 independent reflections
1857 reflections with $I > 2\sigma(I)$
$h = -2 \rightarrow 11$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 41$
4 standard reflections
frequency: 120 min
intensity decay: 3.5%

Refinement

Refinement on F^2
$R(F) = 0.055$
$wR(F^2) = 0.087$
$S = 0.899$
2590 reflections
159 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0247P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.131$ e Å^{-3}
$\Delta\rho_{min} = -0.152$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: Flack (1983)
Flack parameter = 0.12 (12)

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

S—C11	1.739 (3)	C2—C3	1.354 (5)
S—C5	1.754 (3)	C3—C4	1.396 (5)
N1—C2	1.346 (4)	C6—C7	1.383 (4)
N1—C1	1.374 (3)	C6—C11	1.390 (4)
N2—C5	1.299 (3)	C7—C8	1.375 (5)
N2—C6	1.398 (3)	C8—C9	1.395 (5)
C1—C4	1.370 (4)	C9—C10	1.366 (5)
C1—C5	1.443 (4)	C10—C11	1.387 (4)
C11—S—C5	89.1 (2)	C1—C5—S	119.5 (2)
C2—N1—C1	109.0 (3)	C7—C6—C11	119.7 (3)
C5—N2—C6	110.6 (3)	C7—C6—N2	124.9 (3)
C4—C1—N1	107.1 (3)	C11—C6—N2	115.3 (3)
C4—C1—C5	131.5 (3)	C8—C7—C6	119.2 (4)
N1—C1—C5	120.9 (3)	C7—C8—C9	120.5 (4)
N1—C2—C3	109.0 (3)	C10—C9—C8	120.8 (4)
C2—C3—C4	107.2 (4)	C9—C10—C11	118.6 (4)
C1—C4—C3	107.7 (4)	C10—C11—C6	121.1 (3)
N2—C5—C1	124.9 (3)	C10—C11—S	129.4 (3)
N2—C5—S	115.5 (2)	C6—C11—S	109.4 (3)
C4—C1—C5—N2	−159.6 (3)	C4—C1—C5—S	16.9 (5)
N1—C1—C5—N2	11.1 (4)	N1—C1—C5—S	−172.4 (2)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···N2^i	0.88 (2)	2.169 (7)	3.032 (9)	166.0 (7)

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

Intensities were corrected for Lorentz and polarization effects. The structure was solved by direct methods. The H atoms were found in the difference Fourier map and were refined isotropically. Final full-matrix least-squares refinement of the coordinates, anisotropic displacement parameters for non-H atoms and isotropic displacement parameters for H atoms reduced R to 0.055. The absolute structure of the title compound was unequivocally established by refining the Flack chirality parameter (Flack, 1983).

Data collection: *STADI*4 (Stoe & Cie, 1995). Cell refinement: *STADI*4. Data reduction: *X-RED* (Stoe & Cie, 1995). Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*a*). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*b*). Molecular graphics: *PLATON*97 (Spek, 1990) and *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*97 and *PLATON*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1289). Services for accessing these data are described at the back of the journal.

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Diastereomeric complexes of 1,1′-binaphthyl-2,2′-diol and (*R*,*R*)-1,2-cyclohexanediamine

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Abstract

The structures of the molecular complexes (*R*)-1,1′-binaphthyl-2,2′-diol–(*R*,*R*)-1,2-cyclohexanediamine–toluene (1/1/1), $C_{20}H_{14}O_2 \cdot C_6H_{14}N_2 \cdot C_7H_8$, (I), and (*S*)-