Crystal structure of *N*-(2-pyridyl)methyl-*N*-(3-thienyl)methyl-5,7-dihydrodibenzo[*c,e*]azepinium bromide, C₂₅H₂₅N₂SBr

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Abstract

C₂₅H₂₅BrN₂S, monoclinic, C12/c1 (No. 15), *a* = 12.187(5) Å, *b* = 17.399(4) Å, *c* = 10.105(2) Å, β = 96.43(3)°, *V* = 2129.2 Å³, *Z* = 4, *R*_{gt}(*F*) = 0.093, *R*_w(*F*) = 0.175, *T* = 294 K.

Source of material

The title compound was prepared in 60% yield by reaction of 2,2'-bis(bromomethyl)biphenyle and *N*'-(2-pyridyl)methyl-(3-thienyl)methylamine in ethylacetate solvent at room temperature, in presence of *N,N*'-diisopropylethylamine for five days. Crystals were prepared in a 1:1 mixture CH₃CH₂OH/CH₂Cl₂ at room temperature.

Discussion

In the crystal structure the N atom is located on the two-fold axis and therefore the molecule presents a two-fold symmetry. As a consequence, the mixed atom N/C is common to:

- the pyridyl ring C9/C10/C11/C12/C13/(N/C);
- the thienyl ring C9/C10/C11/S/(N/C); and the C12, C13 and S atoms have an occupancy of one half.

Table 1. Data collection and handling.

Crystal:	colorless, prismatic, size 0.30 x 0.40 x 0.40 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	20.18 cm ⁻¹
Diffractometer, scan mode:	Enraf-Nonius CAD4, $\theta/2\theta$
2 $\theta_{\max}$:	47.96°
<i>N(hkl)</i> _{measured} , <i>N(hkl)</i> _{unique} :	3677, 3677
Criterion for <i>I</i> _{obs} , <i>N(hkl)</i> _{gt} :	<i>I</i> _{obs} > 2 σ (<i>I</i> _{obs}), 1117
<i>N(param)</i> _{refined} :	132
Programs:	MOLLEN [1], SDP [2], ORTEP [3], MULTAN80 [4]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	Occ.	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
S	8f	0.5	0.452(1)	0.3681(9)	0.416(2)	0.066(4)
C(12)	8f	0.5	0.415(5)	0.362(3)	0.411(6)	0.07(2)
C(13)	8f	0.5	0.481(4)	0.381(3)	0.513(5)	0.05(1)
H(1)	8f		0.7477	0.0956	0.6118	0.0724
H(2)	8f		0.8734	-0.0133	0.6835	0.1005
H(3)	8f		0.7865	-0.1241	0.7917	0.0781
H(4)	8f		0.5964	-0.1261	0.8250	0.0807
H(5)	8f		0.5714	0.1470	0.5689	0.0606
H(6)	8f		0.4607	0.0909	0.5818	0.0606
H(7)	8f		0.3802	0.2480	0.7762	0.0573
H(8)	8f		0.3329	0.1761	0.6741	0.0573
H(9)	8f		0.3447	0.1846	0.4349	0.0726
H(10)	8f		0.3794	0.2746	0.2514	0.0937
H(11)	8f		0.3947	0.4016	0.3238	0.0888
H(12)	8f		0.5353	0.4265	0.5363	0.0794
H(13)	8f		0.4789	0.3498	0.7014	0.1018

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Br	4c	3/4	1/4	1/2	0.066(2)	0.029(1)	0.045(1)	−0.007(2)	0.016(1)	0.001(2)
N	4e	1/2	0.164(1)	3/4	0.04(1)	0.04(1)	0.03(1)	0	0.01(1)	0
N/C	8f	0.461(2)	0.333(1)	0.600(3)	0.12(2)	0.06(1)	0.11(2)	0.01(1)	0.04(2)	−0.01(1)
C(1)	8f	0.716(2)	0.048(1)	0.659(3)	0.08(2)	0.03(1)	0.06(2)	0.01(1)	0.02(1)	0.00(1)
C(2)	8f	0.788(2)	−0.014(1)	0.699(3)	0.09(2)	0.04(1)	0.08(2)	0.02(1)	0.00(2)	−0.02(1)
C(3)	8f	0.738(2)	−0.077(1)	0.760(3)	0.08(2)	0.03(1)	0.08(2)	0.02(1)	0.01(1)	−0.01(1)
C(4)	8f	0.630(2)	−0.078(1)	0.778(3)	0.09(2)	0.04(1)	0.06(2)	0.01(1)	0.02(1)	−0.01(1)
C(5)	8f	0.560(2)	−0.017(1)	0.739(3)	0.08(2)	0.03(1)	0.05(1)	0.00(1)	0.02(1)	0.00(1)
C(6)	8f	0.607(2)	0.047(1)	0.681(2)	0.06(1)	0.02(1)	0.04(1)	−0.01(1)	0.01(1)	0.00(1)
C(7)	8f	0.532(2)	0.112(1)	0.635(2)	0.06(1)	0.03(1)	0.05(1)	0.00(1)	0.02(1)	0.00(1)
C(8)	8f	0.400(2)	0.213(1)	0.700(2)	0.04(1)	0.04(1)	0.05(1)	0.02(1)	0.01(1)	0.00(1)
C(9)	8f	0.417(2)	0.261(1)	0.581(2)	0.06(1)	0.02(1)	0.05(1)	0.01(1)	0.01(1)	−0.01(1)
C(10)	8f	0.381(2)	0.240(1)	0.454(2)	0.07(1)	0.05(1)	0.06(1)	0.01(1)	0.01(1)	0.00(1)
C(11)	8f	0.393(2)	0.292(2)	0.352(3)	0.07(2)	0.10(2)	0.04(1)	0.01(2)	0.01(1)	0.01(2)

References

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