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Crystal structure of bis(pyridin-2-yl)disulfide hydrotriiodide, $[C_{10}H_9N_2S_2][I_3]$

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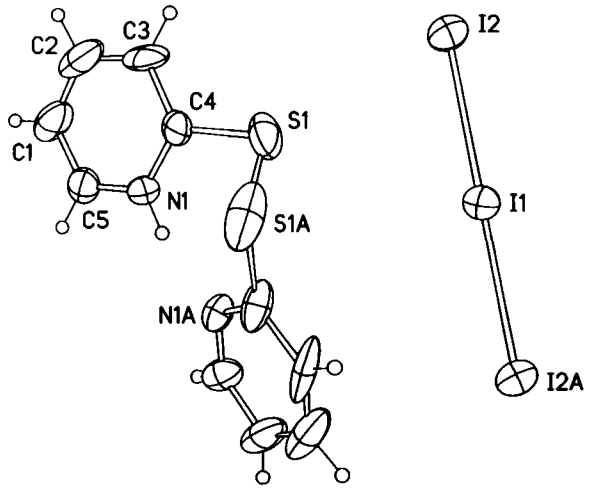

Abstract

$C_{10}H_9I_3N_2S_2$, monoclinic, *C*12/*c*1 (no. 15), a = 16.4841(9) Å, b = 9.6845(7) Å, c = 13.0227(9) Å, β = 127.309(2)°, V = 1653.6 Å^3, Z = 4, $R_{gt}(F)$ = 0.068, $wR_{ref}(F^2)$ = 0.179, T = 153 K.

Source of material

The title compound was prepared by the mixture of pyridine-2-thiol (PySH, 1 mmol) and nickel iodide (0.5 mmol) was dissolved in hot ethanol-water (25 ml; 3:2, *v*/*v*) and stirred at 353 K for 30 min and then filtered. After allowing the solution to stand for about two weeks, brown block-like crystals of the title compound were formed unexpectedly.

Discussion

The title crystal structure consists of $[C_{10}H_9N_2S_2]^+$ cations and $[I_3]^-$ anions. Two PySH ligands couple together through the S atoms with S—S distance of 2.04(2) Å and the angle of two pyridine-ring planes is 29.7°. $[I_3]^-$ anions are regularly arranged around the cations. In addition, there are relatively short distances (centroid separation of 3.648(4) Å, interplanar spacing of 3.542(4) Å) between the pyridine-ring plane and the symmetry-related plane at (½–x,½–y,1–z) in an adjacent cation, implying weak π-π interactions in this structure.

The structure of the title compound represents a second modification to a known phase crystallizing in $P2_1$ [1], which was synthesized by oxidation/dimerization of pyridine-2-thiol with diiodine in dichloromethane. The reason for the two modifications is a different crystal packing caused by different intermolecular bonds. In the title structure, each terminal I atom of the $[I_3]^-$ anion forms two hydrogen bonds with the 4-site and 6-site pyridyl C atoms with C···I distances of 3.952(2) Å and 4.008(2) Å, respectively. In the already published structure [1], the 6-site C atoms of the four independent $[C_{10}H_9N_2S_2]^+$ cations form H bonds to the central I atoms with C···I distances of 3.697 Å, 3.878 Å, 3.902 Å and 3.934 Å.

Table 1. Data collection and handling.

Crystal:	brown block, size 0.10 × 0.16 × 0.26 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	59.06 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50.1°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2510, 1464
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1114
$N(param)_{refined}$:	77
Programs:	SHELXS-97 [2], SHELXL-97 [3]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{iso}
H(1N)	8*f*	0.50	0.5352	0.7925	0.2669	0.075
H(1)	8*f*		0.8029	0.9412	0.4090	0.125
H(2)	8*f*		0.8586	0.7282	0.4040	0.152
H(3)	8*f*		0.7426	0.5467	0.3024	0.141
H(5)	8*f*		0.6426	0.9662	0.3512	0.087

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
I(1)	4*c*	¼	¼	0	0.0606(9)	0.061(1)	0.075(1)	0.0032(7)	0.0401(8)	–0.0041(7)
I(2)	8*f*	0.43132(8)	0.1560(1)	0.0277(1)	0.0592(8)	0.090(1)	0.090(1)	–0.0015(6)	0.0487(7)	–0.0064(7)
S(1)	8*f*	0.5297(5)	0.5230(6)	0.2005(6)	0.112(5)	0.075(3)	0.102(4)	0.028(3)	–0.001(3)	–0.036(3)
N(1)	8*f*	0.5958(9)	0.779(1)	0.290(1)	0.051(7)	0.065(8)	0.060(8)	0.013(6)	0.027(6)	–0.003(6)
C(1)	8*f*	0.759(1)	0.866(3)	0.378(1)	0.06(1)	0.19(3)	0.08(1)	–0.01(1)	0.05(1)	–0.01(2)
C(2)	8*f*	0.791(1)	0.741(3)	0.371(1)	0.05(1)	0.26(4)	0.06(1)	0.04(2)	0.03(1)	0.01(2)
C(3)	8*f*	0.723(2)	0.632(3)	0.315(2)	0.08(1)	0.19(3)	0.05(1)	0.10(2)	0.02(1)	0.02(1)
C(4)	8*f*	0.623(1)	0.654(2)	0.276(1)	0.07(1)	0.09(1)	0.039(8)	0.02(1)	0.008(8)	–0.014(8)
C(5)	8*f*	0.662(1)	0.881(2)	0.341(2)	0.07(1)	0.10(1)	0.06(1)	–0.02(1)	0.046(9)	–0.02(1)

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