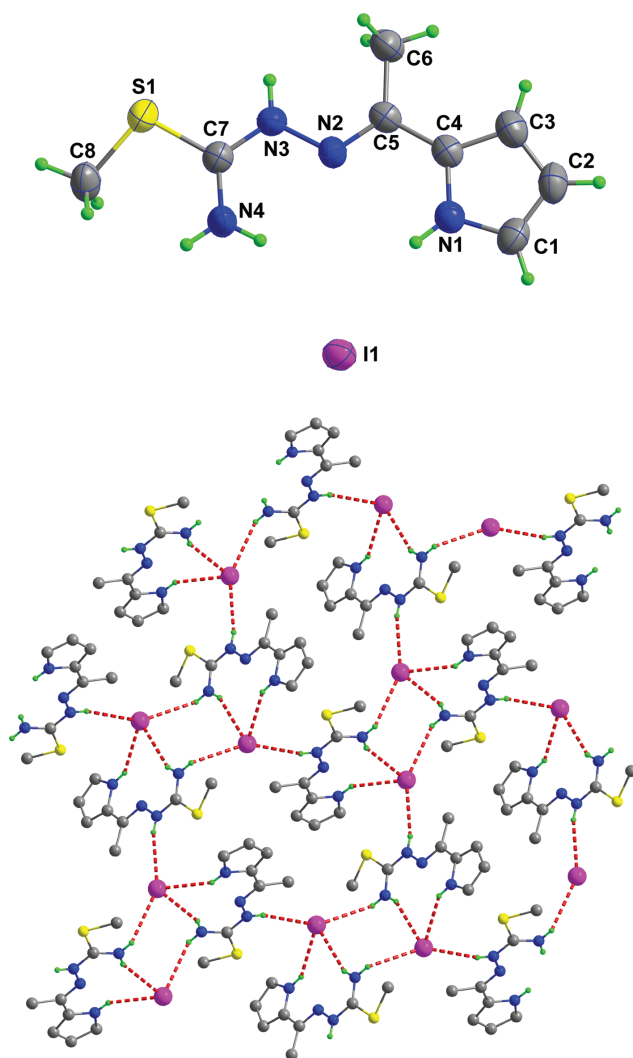


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Crystal structure of 2-acetylpyrrole *S*-methylthiosemicarbazonium hydroiodide, C₈H₁₃IN₄S



Abstract

C₈H₁₃IN₄S, monoclinic, $P2_1/n$ (no. 14), $a = 5.9563(4)$ Å, $b = 12.8592(8)$ Å, $c = 16.1795(10)$ Å, $\beta = 99.450(1)^\circ$, $V = 1222.42(14)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0228$, $wR_{\text{ref}}(F^2) = 0.0535$, $T = 296$ K.

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The asymmetric unit of the title crystal structure is shown in the upper part of the figure, whereas the hydrogen bonding scheme is shown in the lower part. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.15 × 0.15 × 0.10 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	2.76 m ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
$2\theta_{\text{max}}$, completeness:	25°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	6139, 2146, 0.020
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1880
$N(\text{param})_{\text{refined}}$:	127
Programs:	Bruker programs [1], SHELX [2]

Source of material

Thiosemicarbazide (0.091 g, 1 mmol) and iodomethane (0.142 g, 1 mmol) were dissolved in an ethanol solution (10 mL). The mixture was stirred for 1 h under refluxing, and then 2-acetylpyrrole (0.109 g, 1 mmol) was added. The resulting solution was stirred for another 2 h under the same conditions, and then left in air for a few days, yielding yellow block-shaped crystals.

Experimental details

The hydrogen atoms were placed at calculated positions and refined as riding atoms with isotropic displacement parameters.

Discussion

In the past few decades, thiosemicarbazones (TSCs) and their metal complexes have been of interest to chemists and

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
I1	−0.08624(4)	0.340072(15)	0.093888(12)	0.04865(9)
S1	−0.04359(14)	0.22772(6)	0.32909(6)	0.0537(2)
N1	0.8020(4)	−0.04470(18)	0.23107(15)	0.0439(6)
H1	0.7559	−0.0484	0.2786	0.053*
N2	0.4354(4)	0.07985(18)	0.23997(14)	0.0386(5)
N3	0.2459(4)	0.14077(17)	0.24625(15)	0.0398(5)
H3	0.1815	0.1779	0.2049	0.048*
N4	0.2524(4)	0.0743(2)	0.37718(15)	0.0484(6)
H4A	0.3587	0.0320	0.3695	0.058*
H4B	0.2011	0.0739	0.4239	0.058*
C1	0.9810(5)	−0.0976(2)	0.2104(2)	0.0526(8)
H1A	1.0730	−0.1439	0.2448	0.063*
C2	1.0037(5)	−0.0716(3)	0.1311(2)	0.0566(9)
H2	1.1131	−0.0966	0.1012	0.068*
C3	0.8312(5)	0.0002(3)	0.1024(2)	0.0531(8)
H3A	0.8064	0.0319	0.0501	0.064*
C4	0.7054(5)	0.0154(2)	0.16527(17)	0.0393(6)
C5	0.5086(5)	0.0802(2)	0.16959(17)	0.0371(6)
C6	0.4087(5)	0.1408(3)	0.09352(19)	0.0527(8)
H6A	0.4944	0.1276	0.0493	0.079*
H6B	0.4142	0.2137	0.1066	0.079*
H6C	0.2534	0.1200	0.0759	0.079*
C7	0.1689(5)	0.1393(2)	0.31804(18)	0.0379(6)
C8	−0.1612(6)	0.1736(3)	0.4149(2)	0.0559(9)
H8A	−0.2831	0.2171	0.4268	0.084*
H8B	−0.0450	0.1698	0.4635	0.084*
H8C	−0.2185	0.1051	0.4003	0.084*

biologists because of their wide range of pharmacological effects [3, 4]. Our previous work shows that several TSC bearing pyrrole units have potential antitumor activity [5]. As part of our ongoing studies on TSCs, the title compound was synthesized and characterized by X-ray diffraction.

In the title structure, there are one protonated thiosemicarbazone molecule and one iodide anion in the asymmetric

unit. The bond length of C7–N3 of the thiosemicarbazone moiety is 1.317(4) Å, which is quite shorter than that of the normal C–N bond, while slightly longer than that of the common C=N bond. This fact indicates that the C7–N3 should be a double bond and the N3 atoms may be protonated. In the crystal, intermolecular N–H···I hydrogen bonds between all terminal N–H donors of the title cation and the iodide anions link them into a 2D supermolecular network. The geometric parameters of the title cation are in the expected ranges [6]. The same is true for the N–H···I hydrogen bonds of a cation with NH₂ and NH functionality both connected to the iodide counter anions [7].

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