Redetermination of the crystal structure of 5-methyl-2-thiocyto-sine hemihydrate, $C_5H_7N_3S \cdot 0.5H_2O$, at 213 K

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Source of material: Crystals were obtained from the slow evaporation of an aqueous solution of the compound (Sigma) containing a 10 molar excess of NaOH (pH = 12.7); the pH of the solution was 10.0 when crystals deposited. NMR (heteronuclear COSY) in d_6 -DMSO were recorded on a Varian Gemini 2000. 1H (300 Mz): δ 11.97 (H1), 7.79 (H4a), 7.33 (H6), 7.19 (H4b), 1.88 ppm (H5). ^{13}C (75.47 Mz): δ 178.8 (C2), 163.0 (C5), 140.5 (C6), 104.9 (C4), 13.4 ppm (Me).

The analysis is in essential agreement with an earlier study (see ref. 1). The $d(C-S)$, $d(C2-N1)$, $d(C2-N3)$, $d(N3-C4)$ and $d(C4-N4)$ distances of 1.709(2) Å, 1.367(3) Å, 1.340(3) Å, 1.360(3) Å and 1.327(3) Å, respectively indicate significant resonance in the structure with notable additional π -electron density in the C4–N4 bond. The NMR results indicate that this is substantially the case in solution.

$C_5H_7N_3O_{0.5}S$, orthorhombic, $P2_12_12$ (No. 18), $a = 9.914(1)$ Å, $b = 12.875(2)$ Å, $c = 5.399(2)$ Å, $V = 689.1$ Å³, $Z = 4$, $R(F) = 0.032$, $R_w(F) = 0.035$.

Table 3. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(2)	4c	0.23341(5)	0.14322(4)	−0.1753(1)	0.0301(2)	0.0327(3)	0.0329(3)	−0.0033(2)	0.0009(2)	−0.0065(3)
O(1)	2a	−0.5	1/2	−0.1056(4)	0.041(1)	0.036(1)	0.028(1)	0.005(1)	0	0
N(1)	4c	0.1005(2)	0.2723(1)	0.1218(4)	0.0288(8)	0.031(1)	0.031(1)	−0.0024(7)	0.0013(8)	−0.0021(9)
N(3)	4c	0.3356(2)	0.2717(1)	0.1618(4)	0.0292(8)	0.0277(9)	0.0262(9)	−0.0022(7)	0.0004(9)	0.0004(9)
N(4)	4c	0.4360(2)	0.3708(2)	0.4611(4)	0.0291(8)	0.040(1)	0.030(1)	0.0008(8)	−0.0013(8)	−0.007(1)
C(2)	4c	0.2240(2)	0.2355(2)	0.0516(4)	0.0302(9)	0.0247(9)	0.030(1)	−0.0021(8)	0.002(1)	0.0034(9)
C(4)	4c	0.3228(2)	0.3392(2)	0.3540(4)	0.0300(9)	0.024(1)	0.024(1)	−0.0029(8)	−0.0005(9)	0.0045(9)
C(5)	4c	0.1947(2)	0.3757(2)	0.4386(4)	0.0290(9)	0.028(1)	0.028(1)	0.0027(8)	0.0015(9)	0.001(1)
C(5')	4c	0.1852(2)	0.4487(2)	0.6564(6)	0.036(1)	0.037(1)	0.037(1)	0.007(1)	−0.002(1)	−0.006(1)
C(6)	4c	0.0864(2)	0.3411(2)	0.3132(5)	0.030(1)	0.032(1)	0.035(1)	0.0030(8)	0.001(1)	0.000(1)

Table 1. Parameters used for the X-ray data collection

Crystal:	colorless block, size 0.29 x 0.29 x 0.29 mm
Wavelength:	Mo K_{α} radiation (0.7107 Å)
μ :	3.88 cm ^{−1}
Diffraction:	Rigaku AFC6R
Scan mode:	$\omega/2\theta$
$T_{\text{measurement}}$:	213 K
$2\theta_{\text{max}}$:	55°
$N(hkl)_{\text{unique}}$:	955
Criterion for F_o :	$F_o > 0 \sigma(F_o)$
$N(\text{param})_{\text{refined}}$:	119
Program:	TEXSAN

Table 2. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U_{iso}
H(1)	4c	0.019(4)	0.253(3)	0.014(9)	0.11(1)
H(1o)	4c	−0.441(3)	0.467(3)	0.003(7)	0.08(1)
H(4a)	4c	0.522(2)	0.357(2)	0.387(5)	0.043(7)
H(4b)	4c	0.434(3)	0.418(2)	0.608(6)	0.052(9)
H(5a)	4c	0.238(3)	0.423(2)	0.809(5)	0.047(8)
H(5b)	4c	0.215(3)	0.514(2)	0.614(5)	0.052(8)
H(5c)	4c	0.086(4)	0.460(2)	0.697(6)	0.08(1)
H(6)	4c	−0.010(2)	0.362(2)	0.360(4)	0.027(6)

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References

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