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Refinement of the Crystal Structure of Cu(py)$_2$(NCS)$_2$

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To obtain more quantitative evaluations of the mutal influence of ligand consequences on the interatomic distances, degree of a tetragonal distortion [1], and bond modes of coordinated thiocyanate ligands [2] in coordination polyheder of Cu(II) with derivatives of pyridine as ligands, an X-ray structure analysis of Cu(py)$_2$(NCS)$_2$ was required since this compound served as a starting comparing substance. The crystal structure of Co(py)$_2$(NCS)$_2$ was described in [3]. Since Co(py)$_2$(NCS)$_2$ was isostructural with Cu(py)$_2$(NCS)$_2$, the authors on this basis made some conclusions concerning the structure of the Cu(II) compound.

Crystals of Cu(py)$_2$(NCS)$_2$ were prepared according to [4]. These were monoclinic with the following unit cell parameters: $a = 0.9211(2)$, $b = 1.4519(7)$, $c = 0.5626(1)$ nm, $\beta = 108.66(2)°$, $V = 0.7127(3)$ nm^3, space group $C2/m$, $Z = 2$. Density was measured by a flotation method in a mixture with bromophorm and ethanol, $\rho_{\mathrm{obsd}} = 1.55$ g cm^{-3}, $\rho_{\mathrm{calcd}} = 1.57$ g cm^{-3}. All data are in a good agreement with those given in ref. [3].

Integral intensities of 424 symmetrically independent reflections were measured with an automatic diffractometer SYNTEX P2$_1$, using θ--2θ method in an angle interval $0 < 2\theta \leqslant 100°$, CuK$_\alpha$ radiation ($\lambda = 0.154178$ nm), linear absorption coefficient $\mu = 4.74$ mm^{-1}. All computing was performed on a minicomputer NOVA 1200 with the SYNTEX XTL structure determination system.

The crystal structure was solved by a heavy-atom method and refinement by a least-squares method gave $R = 0.083$ for 350 observed reflections and for anisotropic thermal parameters for non-hydrogen atoms. The maximum residual electron density in the differential Fourier synthesis was 0.73×10^{30} e.m^{-3}. The central atom is coordinated octahedrally by two

TABLE I. Final Atomic Coordinates ($\times 10^4$) and Coefficients of Anisotropic Temperature Factors. E.s.d.s Are Given in Parentheses. Temperature Factor is of the Form: $T = \exp[-1/4(B_{11}h^2a^{*2} + B_{22}k^2b^{*2} + B_{33}l^2c^{*2} + 2B_{12}hka^*b^* + 2B_{13}hla^*c^* + 2B_{23}klb^*c^*)]$.

Atom	x	y	z
Cu	0(0)	0(0)	0(0)
N(1)	−1328(17)	0(0)	2077(28)
C(1)	−1970(20)	0(0)	3478(32)
S(1)	−2910(6)	0(0)	5498(9)
N(2)	0(0)	−1366(12)	0(0)
C(2)	0(0)	−3372(20)	0(0)
C(3)	−327(38)	−1909(18)	−2140(53)
C(4)	−327(56)	−2832(21)	−2206(59)
C(5)	−1068(57)	−1803(26)	−79(82)
C(6)	−1164(48)	−2863(26)	−87(84)

Atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Cu	3.66(21)	5.13(23)	3.58(19)	−0.03(0)	2.41(18)	0.03(0)
N(1)	1.79(81)	10.59(143)	2.72(85)	−0.04(0)	3.31(75)	0.04(0)
C(1)	0.59(83)	4.79(117)	1.57(84)	−0.02(0)	0.03(72)	0.02(0)
S(1)	2.25(28)	5.44(34)	3.38(30)	−0.03(0)	2.56(25)	0.03(0)
N(2)	3.14(91)	4.92(107)	4.22(92)	−0.04(0)	−0.07(80)	0.04(0)
C(2)	13.14(273)	6.20(185)	9.20(203)	−0.09(0)	−6.39(205)	0.09(0)
C(3)	8.01(210)	3.10(140)	5.57(162)	1.84(148)	−2.20(167)	−1.28(12)
C(4)	17.94(384)	4.04(177)	6.69(191)	1.22(231)	−6.68(248)	−1.39(15)
C(5)	10.21(360)	4.24(225)	3.75(176)	5.11(229)	0.65(255)	0.54(20)
C(6)	4.87(240)	5.98(254)	5.88(217)	−4.20(204)	2.01(210)	0.01(22)

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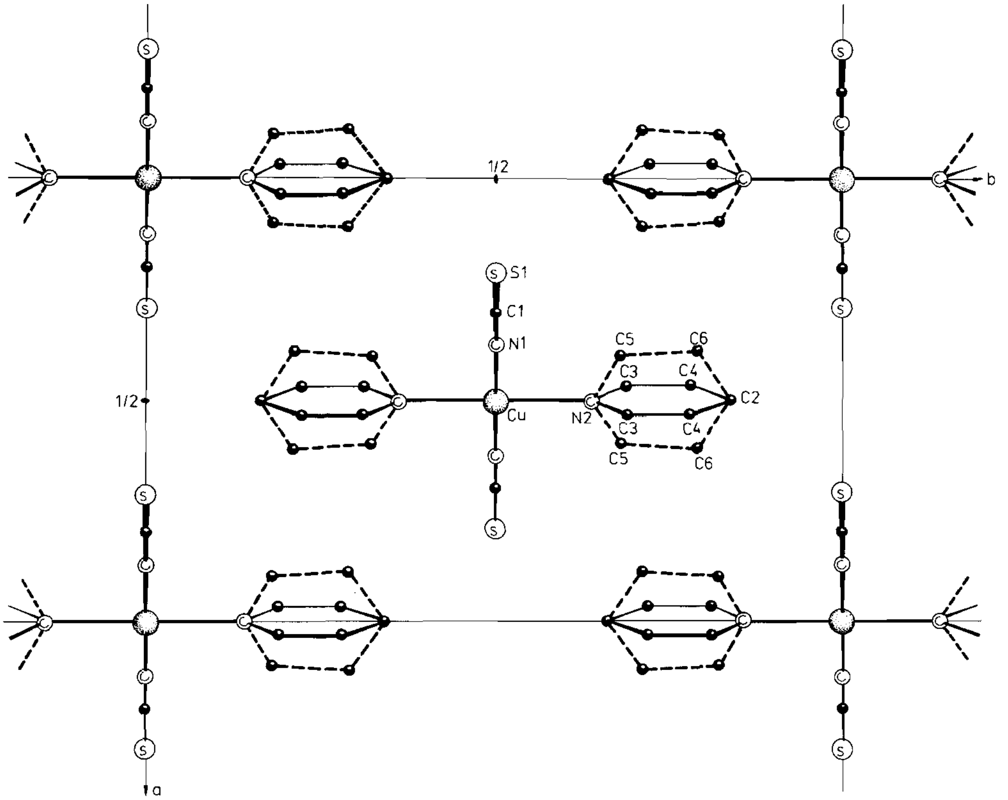

Fig. 1. The projection of the structure in the direction of the *c* axis and the numbering scheme of the atoms.

TABLE II. Interatomic Distances (nm) and Angles (°) in the Complex $Cu(Py)_2(NCS)_2$, with their Estimated Standard Deviations in Parentheses.

Cu–N(1)	0.194(2)	C(1)–S(1)	0.163(2)
Cu–N(2)	0.198(2)	C(2)–C(6)	0.130(5)
N(1)–C(1)	0.113(2)	C(2)–C(4)	0.141(3)
N(2)–C(5)	0.117(5)	C(3)–C(4)	0.134(4)
N(2)–C(3)	0.138(3)	C(5)–C(6)	0.154(5)
Cu–S(1)	0.304(2)		
N(1)–Cu–N(2)	89.0(5)	C(2)–C(4)–C(3)	122.5(32)
Cu–N(1)–C(1)	173.2(13)	C(2)–C(6)–C(5)	121.2(36)
Cu–N(2)–C(5)	123.7(24)	C(3)–N(2)–C(3)	110.9(19)
Cu–N(2)–C(3)	124.1(14)	C(4)–C(2)–C(4)	112.8(25)
N(1)–C(1)–S(1)	179.7(14)	C(5)–N(2)–C(5)	113.7(33)
N(2)–C(5)–C(6)	126.9(39)	C(6)–C(2)–C(6)	110.2(30)
N(2)–C(3)–C(4)	125.7(29)		

nitrogen atoms of thiocyanate groups, two nitrogen atoms of pyridine circles, and two sulphur atoms of bridging thiocyanate groups. Contrary to the assumption in ref. [3], the pyridine circles in the elementary cell can be found in two possible positions which, when considering the high symmetry of the elementary cell, cannot be differentiated. Consequently, a statistical treatment was used in our paper. Initial input for occupancy factors of atoms C_3,C_4,C_5 and C_6 was equal to 0.5 after refinement; however, values of 0.6 and 0.4 were obtained for atoms C_3,C_4 and C_5,C_6 respectively. Both possible modes of orientation of circles are planar and the angle between them is 88.08°.

Coordinates for non-hydrogen atoms are listed in Table I, and bond distances and angles in Table II. The packing of structural units in the elementary cell with the atom numbering scheme is shown in Fig. 1.

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