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Structure of Bis(2-furaldehyde thiosemicarbazonato)nickel(II)*

BY VEDAVATI G. PURANIK, S. S. TAVALE AND T. N. GURU ROW†

Physical and Structural Chemistry Unit, National Chemical Laboratory, Pune 411008, *India*

AND P. UMAPATHY AND A. P. BUDHKAR

Inorganic Chemistry Division, National Chemical Laboratory, Pune 411008, *India*

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Abstract. $[Ni(C_6H_6N_3OS)_2]$, $M_r = 395·1$, rhombohedral, $R\bar{3}$, $a = 10·689\ (2)$ Å, $\alpha = 82·04\ (2)°$, $V = 1188·9\ (4)$ Å^3, $Z = 3$, $D_m = 1·65\ (7)$, $D_x = 1·655$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0·7107$ Å, $\mu = 1·492$ mm^{-1}, $F(000) = 606$, $T = 293$ K, $R = 0·028$ for 929 observed reflections. The Ni^{II} ion is in a distorted square-planar ligand field formed by the N_2S_2 chromophore. The planar furan rings are in a symmetric arrangement. The thiosemicarbazonato group is nearly planar.

Introduction. The α-(*N*)-heterocyclic carboxaldehyde thiosemicarbazones constitute a class of agents which possess both antineoplastic and antiviral activity. The correlation between antitumor activity and chelating ability of such compounds has been reported (Michaud & Sartorelli, 1968). Metal chelates of Pt^{II}, Pd^{II} and Ni^{II} with several ligands containing carboxaldehyde thiosemicarbazones have been synthesized in order to study the nature of the bonding and the stereochemistry. 2-Furaldehyde thiosemicarbazone has already been prepared (Sah & Daniels, 1950) and the title compound was prepared using this ligand.

Experimental. Crystals from alcohol, approximate dimensions 0·15 × 0·25 × 0·4 mm; D_m by flotation; Nonius CAD-4F-11M diffractometer; graphite-monochromated Mo $K\alpha$ radiation; $\omega/2\theta$ scan mode, scan speed 1° min^{-1}; $\theta < 23·5°$, h 0 to 12, k −12 to 12, l −12 to 12. 3472 reflections collected, 929 judged significant ($|F_o| \geq 3\sigma|F_o|$). Lattice parameters from 25 reflections ($12 < 2\theta < 36°$); three standard reflections ($\bar{5}\bar{1}\bar{2}$, $\bar{4}0\bar{2}$ and $\bar{1}1\bar{5}$) every 1000 s, 3% variation in intensity. No correction for absorption. Structure was solved by direct methods, *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) using a modified procedure (Tavale & Guru Row, 1986). Full-matrix least-squares refinement (*LALS*; Gantzel, Sparks & Trueblood, 1961) of scale factor, positional and anisotropic thermal parameters (H atoms fixed geometrically, isotropic thermal parameters, not refined) converged to $R = 0·028$ and $wR = 0·028$; $\sum w(|F_o| - |F_c|)^2$ minimized, $w = (8·5 + 1·0|F_o| + 0·014|F_o|^2)^{-1}$. Max. $(\Delta/\sigma) = 0·1$. Final $\Delta\rho$ excursions $<|0·2|$ e Å^{-3}. No correction for secondary extinction. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).‡

Discussion. The atomic parameters with their e.s.d.'s and equivalent isotropic thermal parameters are given in Table 1. Bond lengths and bond angles involving non-H atoms are given in Table 2. Fig. 1 gives a perspective view of the molecule along with the numbering of atoms. Ni^{II} is in the distorted square-planar ligand field of the N_2S_2 chromophore as in [1,1′-(2,4-butanedione dihydrazono)-di-2,2′-phenylethanethiolato(2−)]nickel(II) (Hansen & Larsen, 1977), [2,5-hexanedione bis(4-phenylthiosemicarbazonato)]nickel(II) (Nandi, Chaudhuri, Mazumdar & Ghosh, 1984) and in bis(diiminosuccinonitrilo)nickel(II) (Peng, Wang & Chiang, 1984). The thiosemicarbazone group is planar with Ni—S = 2·149 (1) Å and Ni—N(2) = 1·921 (2) Å. The coordination around Ni is

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[†] To whom all correspondence should be addressed.

[‡] Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44264 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters for non-*H *atoms with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{4}{3}[(B_{11} + B_{22} + B_{33})a^2 + (B_{12} + B_{13} + B_{23})a^2\cos\alpha].$$

	x	y	z	B_{eq}(Å^2)
Ni	5000	10000	5000	2·51
S	6368 (1)	8333 (1)	5202 (1)	3·62
O	3015 (3)	10740 (2)	809 (2)	4·79
N(1)	5283 (3)	8258 (2)	3101 (3)	3·29
N(2)	4747 (2)	9453 (2)	3424 (2)	3·00
N(3)	6738 (3)	6565 (3)	3677 (3)	4·77
C(1)	6087 (3)	7698 (3)	3884 (3)	3·08
C(2)	4067 (3)	10103 (3)	2597 (3)	3·56
C(3)	3771 (3)	9755 (3)	1428 (3)	3·53
C(4)	3970 (3)	8763 (3)	753 (3)	3·94
C(5)	3300 (4)	9117 (4)	−334 (3)	4·68
C(6)	2753 (4)	10314 (4)	−252 (4)	5·11

Table 2. *Bond distances* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

Ni—N(2)	1·922 (2)	C(3)—O—C(6)	106·5 (3)
S—C(1)	1·724 (3)	N(2)—N(1)—C(1)	111·6 (3)
O—C(3)	1·386 (4)	Ni—N(2)—N(1)	121·0 (2)
O—C(6)	1·357 (5)	Ni—N(2)—C(2)	125·1 (2)
N(1)—N(2)	1·395 (3)	N(1)—N(2)—C(2)	113·9 (2)
N(1)—C(1)	1·310 (4)	S—C(1)—N(1)	123·7 (3)
N(2)—C(2)	1·289 (4)	S—C(1)—N(3)	116·8 (3)
N(3)—C(1)	1·342 (5)	N(1)—C(1)—N(3)	119·6 (3)
C(2)—C(3)	1·439 (4)	N(2)—C(2)—C(3)	129·2 (3)
C(3)—C(4)	1·340 (5)	O—C(3)—C(2)	110·7 (3)
C(4)—C(5)	1·429 (5)	O—C(3)—C(4)	109·1 (3)
C(5)—C(6)	1·341 (6)	C(2)—C(3)—C(4)	140·1 (3)
		C(3)—C(4)—C(5)	107·4 (3)
		C(4)—C(5)—C(6)	106·0 (3)

trans planar with respect to the two S and two N atoms. A similar coordination is found in bis(thiosemicarbazidato)nickel(II) (Cavalca, Nardelli & Fava, 1962). The furan ring plane is at an angle of 3 (1)° to the coordination plane.

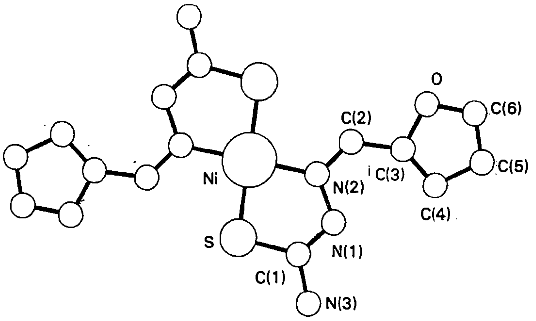

Fig. 1. A perspective view of the molecule.

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The Structures of 1-Methylpyridinium Aqua-di-μ-chloro-trichlorodicuprate(II) and 4-Aminopyridinium Aqua-di-μ-chloro-trichlorodicuprate(II) Hydrate

BY MARCUS R. BOND AND ROGER D. WILLETT

Chemical Physics Program, Washington State University, Pullman, WA 99163, *USA*

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Abstract. $C_6H_8N^+.[Cu_2Cl_5(H_2O)]^-$, $M_r = 416{\cdot}50$, monoclinic, $P2_1/n$; at $T \sim 293$ K: $a = 12{\cdot}051$ (3), $b = 7{\cdot}493$ (2), $c = 15{\cdot}211$ (4) Å, $\beta = 108{\cdot}35$ (2)°, $V = 1303{\cdot}5$ (5) Å^3, $Z = 4$, $D_x = 2{\cdot}12$ g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71069$ Å, $\mu = 42{\cdot}83$ cm^{-1}, $F(000) = 815{\cdot}85$. Least-squares refinement of 1877 independent observed $[|F| > 3\sigma(F)]$ reflections gave $R = 0{\cdot}038$, $wR = 0{\cdot}045$. The structure consists of parallel stacks (parallel to the b axis) of $[Cu_2Cl_5(H_2O)]^-$ planar bibridged dinuclear oligomers separated by parallel stacks of 1-methylpyridinium cations. Adjacent oligomers in the stack are related by centers of inversion.

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