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Acta Cryst. (1978). B**34**, 1999–2000

Bis[(dithionitrito-*S*)amido]nickel(II) – A New (β) Modification

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(*Received* 5 *December* 1977; *accepted* 20 *January* 1978)

Abstract. $Ni(S_2N_2H)_2$, orthorhombic, *Pbca*, a = 8·390 (1), b = 12·410 (3), c = 13·320 (2) Å, V = 1390 Å^3, Z = 8, D_x = 2·34 g cm^{-3}. The structure was refined to R = 0·034 for 1329 independent reflexions, and differs from the old (α) modification only in the molecular packing.

Introduction. Co, Ni, Pd, Pt form the chelate complexes $M(S_2N_2H)_2$. The structures of the last three have been determined from photographic data (Lindqvist & Weiss, 1958; Weiss & Thewalt, 1968).

The structural data were rather inaccurate and did not include H atoms. As these compounds are examples of the rare, purely inorganic chelate complexes, a redetermination of the structure of at least one of them seemed desirable. The Ni complex was prepared by varying the procedure of Goehring, Daum & Weiss (1955): 5 g of $NiCl_2.6H_2O$ and 5 g of S_4N_4 were refluxed for 1 h in 400 ml methanol. The deep-violet solution was filtered and then evaporated to complete dryness (rotator-evaporator). The residue was washed on a G3 glass frit with a large amount of cold water, until the filtrate was free from Cl^-. After drying in a desiccator it was washed with hot cyclohexane to remove S_4N_4, then dissolved in a small volume of acetone, filtered and allowed to evaporate by standing. Instead of growing the crystals from acetone, whereby the monoclinic crystals of the former structure determination were obtained, we used ethyl acetate. Lattice constants corresponded well with the known data, but insertion of the published atomic coordinates did not result in a reasonable R. A detailed inspection of the lattice parameters and the intensities revealed that the crystal was orthorhombic rather than monoclinic. The exact lattice parameters were calculated from the θ values of 25 reflexions, determined on an automatic single-crystal diffractometer, by least squares (Berdesinski & Nuber, 1966). Intensity measurements on the Siemens diffractometer with the five-value method, θ–2θ scans up to $2\theta = 70°$ with Mo $K\alpha$ radiation, were carried out treating the crystal as monoclinic. Corresponding intensities were averaged to give an orthorhombic data set of 1329 independent observed reflexions, classifying reflexions with $I < 2·58\sigma(I)$ as unobserved. The observed intensities were corrected for Lorentz and polarization factors only. The structure was solved with *MULTAN* (Main, Germain & Woolfson, 1971). H atoms were located from a difference map. Mixed refinement (isotropic temperature factors for H, anisotropic for the other atoms) converged at R = 0·034.* Calculations were performed on a IBM 370/168 computer with the XRAY system (Stewart, Kundell & Baldwin, 1970). Scattering factors were taken from *International Tables for X-ray Crystallography* (1974).

Discussion. Atomic coordinates are listed in Table 1. The asymmetric unit is formed by one complex molecule $[Ni(S_2N_2H)_2]$. The two halves of the molecule

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33342 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Final fractional coordinates* ($\times 10^4$)

	x	y	z
Ni	5256 (1)	693 (1)	1245 (1)
S(1)	5855 (2)	2367 (1)	1246 (2)
S(2)	8745 (2)	1298 (1)	1234 (2)
S(3)	2791 (2)	1098 (1)	1118 (1)
S(4)	2833 (2)	8902 (1)	1387 (1)
N(1)	7455 (5)	365 (4)	1223 (5)
N(2)	7846 (5)	2374 (4)	1239 (7)
N(3)	4661 (5)	9268 (4)	1400 (4)
N(4)	1842 (5)	9940 (3)	1261 (5)
H(1)	7863 (100)	9817 (68)	1033 (69)
H(2)	5205 (74)	8704 (49)	1419 (52)

Table 2. *Deviations* (Å) *of atoms from least-squares planes*

Atoms marked with an asterisk were used to calculate the plane.

Ni	0·006*	0·006*	N(1)	−0·008*	−0·148
S(1)	−0·003*	0·256	N(2)	−0·001*	0·182
S(2)	0·006*	−0·027	N(3)	0·220	0·002*
S(3)	−0·181	−0·015*	N(4)	0·013	0·020*
S(4)	0·195	−0·013*	H(1)	−0·254	−0·500
			H(2)	0·253	−0·081

are planar and enclose an angle of 8·2°. Deviations from the best plane are shown in Table 2. The numbering scheme is given in Fig. 1, together with bond lengths and angles. Fig. 1 shows a section through the unit cell at $z = \frac{1}{8}$. The molecules are arranged in sheets at $z = n \times \frac{1}{8}$, $n = 1, 3, 5, 7$. Two adjacent sheets are displaced with respect to each other, so that the shortest separation between two Ni atoms of different sheets is 3·76 Å, whereas the interplanar distance between two molecules, perpendicular to the sheets is $\frac{1}{4}c = 3 \cdot 33$ Å. The Ni–S and Ni–N lengths correspond to the sum of the covalent radii.

This work was supported by the Deutsche Forschungsgemeinschaft and by the Fonds der Chemischen Industrie.

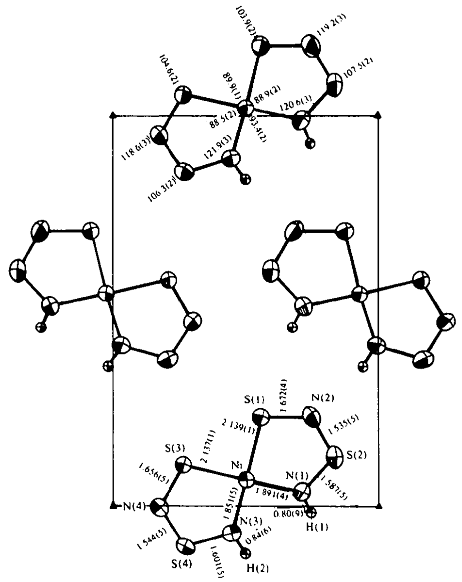

Fig. 1. *ORTEP* plot (Johnson, 1965). View of the plane $x,y,\frac{1}{8}$. Distances are in Å, angles in degrees.

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