both the Mn– and Re–diphenylphosphanido compounds deviate to a slight extent from planarity (dihedral angles 4.1 and 4.6°).

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Redetermination of the Structure of *trans*-Tetraamminebis(thiocyanato-*N*)nickel(II)

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Abstract. [Ni(NCS)$_2$(NH$_3$)$_4$], $M_r = 243.0$, monoclinic, $C2/m$, $a = 11.359\,(9)$, $b = 8.128\,(7)$, $c = 5.608\,(5)$ Å, $\beta = 75.42\,(5)°$, $V = 501.1\,(8)$ Å^3, $Z = 2$, $D_m = 1.594\,(1)$, $D_x = 1.61$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 22.7$ cm^{-1}, $F(000) = 252$, room temperature, final $R = 0.021$ for 491 independent observed reflections. The [Ni(NCS)$_2$(NH$_3$)$_4$] complex has $2/m$ crystallographic symmetry and contains an NiN$_2$N$_4$ chromophore with nearly regular tetragonal bipyramidal geometry. The Ni—NCS and Ni—NH$_3$ distances are 2.079 (3) and 2.103 (2) Å, respectively.

Experimental. The title compound was prepared by the reaction of Ni(NCS)$_2$ (0.5 M water–ethanol solution) and NH$_3$ (concentrated water solution) with Ni:NH$_3$ molar ratio of 1:4. Prism-shaped blue crystals were separated after 2 d. A crystal of dimensions $0.3 \times 0.3 \times 0.3$ mm was used for diffraction measurements. D_m was determined by flotation in bromoform–acetone. Data were collected on a Syntex $P2_1$ diffractometer, using θ–2θ scans of variable scan speed (4.88–29.30° min^{-1}). Unit-cell parameters were determined from 15 reflections with $3.8 < \theta < 10.5°$. An empirical absorption coefficient, based on intensity measurements at different azimuthal angles, was applied with transmission factors of 0.597–1.000. The maximum value of $(\sin\theta)/\lambda$ was 0.6497 Å^{-1}; hkl ranges were h -12 to 14, $k = 0$ to 10, $l = 0$ to 7. Two check reflections, measured after every 100 reflections, showed no significant systema-tic fluctuation. 1398 reflections were measured, of which 621 were unique. Data reduction used *XP*21 (Pavelčik, 1987); $R_{int} = 0.015$ for 120 duplicate measurements; 130 unobserved reflections with $I < 3\sigma(I)$. The structure was solved by heavy-atom methods, with H-atom positions determined from a difference Fourier map. Non-H atoms were refined anisotropically, H atoms isotropically, by full-matrix least squares on F with *SHELX*76 (Sheldrick, 1976). 44 parameters were refined. After the final cycle, $R = 0.021$, $wR = 0.020$. [$w = 0.5669/\sigma^2(F)$], $S = 0.7810$; $(\Delta/\sigma)_{max} = 0.005$, $\Delta\rho = -0.23$ to 0.19 e Å^{-3}. Final atomic parameters are listed in Table 1,* bond distances and angles in Table 2. Fig. 1 presents the molecular structure with atom numbering. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV) (Ni atom) and *SHELX*76 (Sheldrick, 1976) (S, C, H and N atoms), which was used for all calculations. Geometrical analysis was performed using the program *PARST* (Nardelli, 1983).

Related literature. The structure of the title complex was originally published by Paraj-Kojic, Antzish-

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Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic thermal parameters* (Å^2)

H atoms were refined isotropically. For non-H atoms U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} matrix.

	x	y	z	U_{eq}
Ni	0	0	0	0.0403 (2)
S	0.3578 (1)	0	−0.6817 (2)	0.0579 (3)
C	0.2378 (2)	0	−0.4544 (5)	0.0379 (9)
N	0.1510 (2)	0	−0.3003 (5)	0.0529 (10)
N1	0.0835 (2)	0.1862 (3)	0.1599 (4)	0.0527 (8)
H1	0.123 (2)	0.256 (4)	0.054 (5)	0.10 (1)
H2	0.038 (2)	0.252 (4)	0.259 (5)	0.11 (1)
H3	0.128 (2)	0.152 (4)	0.238 (5)	0.10 (1)

Table 2. *Interatomic distances* (Å) *and bond angles* (°)

Ni—N	2.079 (3)	N—C	1.136 (3)
Ni—N1	2.103 (3)	C—S	1.615 (3)
N1—H1	0.86 (3)	N1—H3	0.80 (3)
N1—H2	0.85 (3)		
N—Ni—N1	88.7 (1)	Ni—N—C	175.8 (3)
N—C—S	177.6 (3)		

Fig. 1. Molecular structure with atom numbering.

kina, Dickareva & Jukhnov (1957), but refined structural data were necessary for a study of structure correlations (Jóna, Valach, Gažo, Fendrych & Šramko, 1983), for reactivity studies of solid nickel-(II) complexes (Jóna, Šramko & Gažo, 1975) and for comparison with related [Ni(NCS)₂Lₓ] complexes [L is an N-donor ligand such as ammonia, piper-idine (pip) or pyridine (py)], e.g. [Ni(NCS)₂(pip)₄] (Koman, Handlovič, Ďurčanská & Gažo, 1983), [Ni(NCS)₂(py)₄] (Valach, Sivý & Koreň, 1984) and [Ni(NCS)₂(3-ethylpy)₄] (Ďurčanská, Jamnický, Koman, Wnęk & Głowiak, 1986), for x = 4.

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Structure of Bis(4-methylpyridine-N)copper(I) Bromide

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Abstract. Bromobis(4-methylpyridine)copper(I), [CuBr(C₆H₇N)₂], M_r = 329.71, triclinic, $P\bar{1}$, a = 9.254 (7), b = 9.736 (3), c = 7.955 (3) Å, α = 102.34 (3), β = 112.33 (3), γ = 95.09 (4)°, V = 636.2 (6) Å³, Z = 2, D_m = 1.8, D_x = 1.721 g cm⁻³, λ(Mo $K\alpha$) = 0.710326 Å, μ = 50.84 cm⁻¹, $F(000)$ =
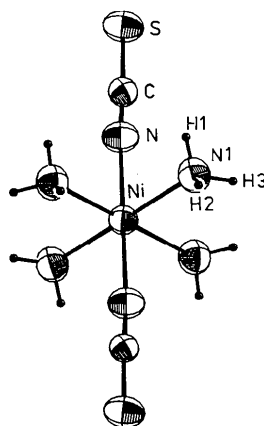
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