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Structure of Bis(diiminosuccinonitrilo)nickel(II), $[Ni(C_4H_2N_4)_2]$

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Abstract. $M_r = 270{\cdot}7$, monoclinic, $P2_1/n$, $a = 3{\cdot}7046\,(8)$, $b = 8{\cdot}773\,(5)$, $c = 15{\cdot}999\,(1)$ Å, $\beta = 93{\cdot}34\,(1)°$, $V = 519{\cdot}1$ Å^3, $Z = 2$, $D_m = 1{\cdot}72$, $D_x = 1{\cdot}73$ Mg m^{-3}, $\lambda = 0{\cdot}7093$ Å, $\mu = 1{\cdot}86$ mm^{-1}, Mo $K\alpha$, $F(000) = 272$, $T = 298$ K, final $R = 0{\cdot}037$ for 873 observed reflections. The complex molecule is nearly planar and has approximate D_{2h} symmetry. The molecules are packed in a parallel fashion along the a axis with a short Ni—Ni distance of 3·705 Å and a close interplanar distance of 3·357 Å.

Introduction. Transition-metal complexes with unsaturated electron-rich ligands are of interest, owing to their unusual spectroscopic, magnetic, redox and structural properties. Examples of this type are metal complexes of dithiene (Teo & Snyder-Robinson, 1979), *O*-benzoquinone diimine anion (Hall & Soderberg, 1968), a 14-membered macrocyclic ligand (Peng & Goedken, 1976) and diiminosuccinonitrilo anion (disn).

The title complex was prepared by the method of Miles, Hursthouse & Robinson (1971), using triethylamine as the base. The structure was determined in order to illustrate the structural properties of the disn ligand.

Experimental. Crystals of $Ni(disn)_2$ obtained by slow diffusion of CCl_4 into a saturated acetonitrile solution. Crystal 0·1 × 0·4 × 0·4 mm. CAD-4 diffractometer. D_m measured by flotation. No absorption correction. $2\theta_{max} = 54°$ ($-4 \le h \le 4, 0 \le k \le 11, 0 \le l \le 20$). Unit cell: least-squares refinement of 16 reflections. Three standard reflections, no significant variation. 1128 unique reflections measured, 873 with $I \ge 3\sigma(I)$. $R = 0{\cdot}037$, $Rw = 0{\cdot}038$, $S = 1{\cdot}64$ based on F. $w = 1/[\sigma^2(F_o) + (0{\cdot}01F_o)^2]$. Structure obtained by Fourier synthesis with Ni atom at $\bar{1}$. H atoms found on difference Fourier synthesis were refined by least-squares process. $(\Delta/\sigma)_{max} = 0{\cdot}03$. Peaks on final $\Delta\rho$ map $\le |0{\cdot}5|$ e Å^{-3}. Secondary-extinction coefficient 0·13 (length in μm). Atomic scattering factors calculated by the analytical form using the coefficients in *International Tables for X-ray Crystallography* (1974). Programs from NRCC *PDP*-8 package (Larson & Gabe, 1978).

Table 1. *Fractional atomic coordinates and* B_{eq} *values*

$$B_{eq} = \tfrac{8}{3}\pi^2(U_{11} + U_{22} + U_{33}).$$

	x	y	z	B_{eq}(Å^2)
Ni	0	0	0	2·21 (3)
N(1)	0·1676 (9)	0·1961 (3)	−0·0025 (2)	2·6 (1)
N(2)	−0·0828 (9)	0·0434 (3)	0·1087 (2)	2·7 (2)
N(5)	0·365 (1)	0·5484 (4)	0·0918 (2)	4·2 (2)
N(6)	−0·123 (1)	0·3057 (4)	0·2744 (2)	4·7 (2)
C(1)	0·1459 (9)	0·2719 (4)	0·0690 (2)	2·4 (2)
C(2)	0·001 (1)	0·1850 (4)	0·1326 (2)	2·4 (2)
C(5)	0·266 (1)	0·4272 (4)	0·0814 (2)	2·9 (2)
C(6)	−0·066 (1)	0·4272 (4)	0·0814 (2)	2·9 (2)
H(1)	0·249 (9)	0·244 (4)	−0·042 (2)	2·4 (8)
H(2)	−0·155 (9)	−0·011 (3)	0·141 (2)	1·8 (7)

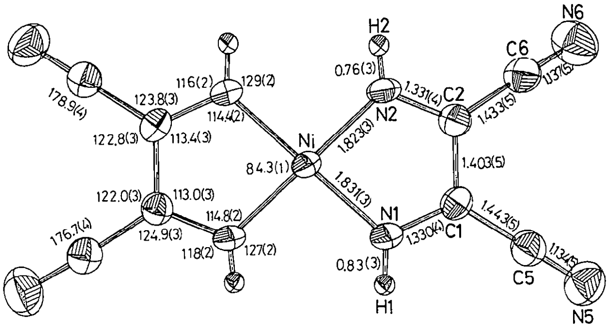

Fig. 1. The molecular structure, labeling scheme, and interatomic distances (Å) and angles (°).

Discussion. Atomic positional parameters and equivalent isotropic temperature factors are listed in Table 1† The bond lengths and angles are given in Fig. 1. The title compound is planar with a maximum deviation of 0·264 (4) Å [N(6)] from the plane formed by the Ni atom and four N atoms. It can be described as having D_{2h} symmetry around the Ni atom within experimental error.

The bond lengths of the disn ligand are comparable to those found in $Pt(disn)_2$ (Lauher & Ibers, 1975) and $Co(disn)_2(CN).2H_2O$ (Peng, Wang, Wang, Chung, Le Page & Gabe, 1981) (Table 2). The significant

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† Tables of anisotropic temperature factors and structure factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39460 (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 2. *Comparison of bond distances* (Å) *and angles* (°) *in metal complexes of disn and free damn*

	$Ni(disn)_2$ (square planar)	$Pt(disn)_2$ (square planar)	$Co(disn)_2(CN)$ (square pyramidal)	damn
M—N(1)	1·827 (4)	1·957 (8)	1·850 (5)	
N(1)—C(1)	1·330 (4)	1·323 (13)	1·325 (8)	1·392 (8)
C(1)—C(2)	1·403 (5)	1·416 (19)	1·428 (8)	1·363 (6)
C(1)—C(5)	1·443 (5)	1·415 (15)	1·454 (9)	1·439 (7)
C(5)—N(5)	1·135 (5)	1·123 (15)	1·122 (10)	1·165 (10)
N(1)—*M*—N(2)	84·3 (1)	79·1 (5)	82·9 (2)	
M—N(1)—C(1)	114·6 (2)	116·4 (7)	115·5 (4)	
N(1)—C(1)—C(2)	113·2 (3)	114·0 (6)	112·7 (6)	124·1 (4)
N(1)—C(1)—C(5)	124·4 (3)	122·6 (9)	124·3 (6)	117·6 (3)
C(2)—C(1)—C(5)	122·4 (3)	123·3 (7)	122·8 (6)	118·1 (8)
C(1)—C(5)—N(5)	177·8 (4)	179·7 (13)	177·2 (9)	179·8 (9)
Reference		Lauher & Ibers (1975)	Peng *et al.* (1981)	Penfold & Lipscomb (1961)

differences between the anion ligand in the complex and the free ligand diaminomaleonitrile (damn) (Penfold & Lipscomb, 1961) involve N(1)—C(1) and C(1)—C(2), with N(1)—C(1) much shorter (1·330 Å) and C(1)—C(2) much longer (1·403 Å) in the complex than in the free ligand (1·392, 1·363 Å). This confirms the delocalization of π electrons over the ligand moiety upon complexation.

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Conformational Aspects of *meso*-Tartaric Acid. VII.* Structure of Calcium *meso*-Tartrate Trihydrate, $Ca^{2+}.C_4H_4O_6^{2-}.3H_2O$

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Abstract. $M_r = 242{\cdot}20$, monoclinic, $P2_1/c$, $a = 8{\cdot}921\,(1)$, $b = 10{\cdot}300\,(3)$, $c = 9{\cdot}881\,(1)$ Å, $\beta = 91{\cdot}78\,(1)$°, $V = 907{\cdot}5\,(3)$ Å^3, $Z = 4$, $D_x = 1{\cdot}773$ Mg m^{-3}, $\lambda(\text{Cu } K\bar{\alpha}) = 1{\cdot}5418$ Å, $\mu = 6{\cdot}17$ mm^{-1}, $F(000) = 496$, $T = 295$ K. Final $R = 0{\cdot}065$ for 1817 observed unique reflections. The *meso*-tartrate dianion adopts a dissymmetric conformation, staggered about the central C—C bond. The non-H skeleton comprises two nearly planar halves consisting of glycolic acid. The Ca^{2+} ion is irregularly surrounded by seven O atoms. Extensive hydrogen bonding contributes to the stabilization of the structure.

Introduction. The crystal structure of the title compound has been determined in the course of a conformational study of the *meso*-tartaric acid molecule.

Experimental. Colourless transparent needle-shaped crystals obtained by single-gel diffusion of $CaCl_2$ in a gel formed upon mixing sodium silicate and *meso*-tartaric acid; crystal size 0·66 × 0·43 × 0·13 mm. Enraf–Nonius CAD-4 diffractometer, Ni-filtered Cu $K\alpha$ radiation; lattice dimensions determined using 14 reflections in θ range 8 to 21°. Systematic absences $h0l$ for $l = 2n+1$ and $0k0$ for $k = 2n+1$; h −10–10, k 0–12, l −12–12; 1858 independent intensities measured up to $2\theta_{\max} = 75$°; 1828 above $2{\cdot}5\sigma(I)$ level considered observed. Three periodically measured standard reflections, 102, $\bar{1}11$, $02\bar{2}$, showed no significant decay in intensity during data collection; severe absorption (up to 60%) was noticed in the ψ scan, but initially no absorption correction applied; Lp correction.

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