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# *cis*-Bis(dicyanamido)bis(1,10-phenanthroline)-nickel(II)

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**Key indicators**

Single-crystal X-ray study
*T* = 293 K
Mean σ(C–C) = 0.003 Å
*R* factor = 0.069
*wR* factor = 0.157
Data-to-parameter ratio = 16.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.



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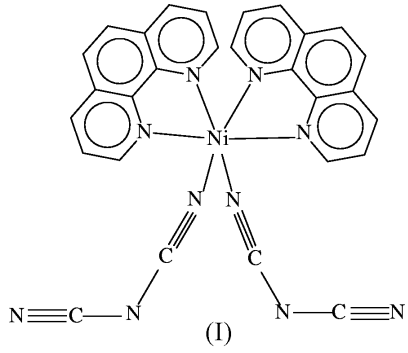
In the title complex, $[Ni(C_2N_3)_2(C_{13}H_8N_2)_2]$, which consists of discrete molecules, the $Ni^{II}$ atom has a slightly distorted octahedral environment, with four N atoms of two phenanthroline ligands and two terminal N atoms of two dicyanamide ligands in a *cis* arrangement. The $\pi$–$\pi$ stacking interactions result in the formation of a one-dimensional chain structure along the [110] direction.

## Comment

Metal dicyanamide {dca, $[N(CN)_2]^-$} coordination chemistry is a fast-growing research field because of the interesting possibilities for coordination and the physical properties of these compounds (Miller & Manson, 2001). Dicyanamide is a versatile ligand for the construction of supramolecular architectures, since it can act in a mono-, bi- or tridentate manner. Compounds formulated as $[M(dca)_2]_n$ ($M$ = Mn, Fe, Co, Ni, Cu, Zn *etc*.) have been previously synthesized (Jensen *et al.*, 1999; Batten *et al.*, 1998). In addition, many ternary compounds have been synthesized by the introduction of monodentate or bidentate co-ligands, such as pyridine, bipyridine, 1,10-phenanthroline and biimidazole, resulting in various interesting structures (Marshall *et al.*, 2000; Manson *et al.*, 1999; Potočnák *et al.*, 1995). As an extension of this research, we have synthesized the title compound, $Ni(dca)_2(1,10\text{-phen})_2$, (I) (Fig. 1).




(I)

The crystal structure of (I) consists of discrete molecules and is similar to those of the reported complexes $M(dca)_2(1,10\text{-phen})_2$ ($M$ = Cu, Mn and Zn; Potocnak *et al.*, 1995; Wang *et al.*, 2000). The coordination environment of the Ni atom can be described as a slightly distorted octahedron. The Ni atom is coordinated by four N atoms of phen ligands (atoms N1, N2, N3 and N4) and two N-terminal atoms (N5 and N8) of dca ligands, the equatorial plane consisting of three N atoms (N1, N3 and N4) of phen ligands and one N atom (N5) of a dca ligand, and the axial positions being occupied by the

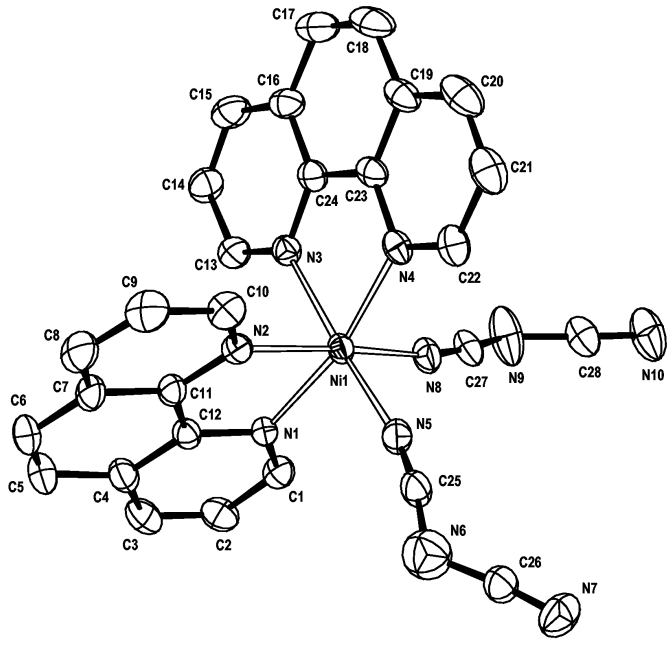



**Figure 1**
The molecular structure of (I), with displacement ellipsoids at the 30% probability level.

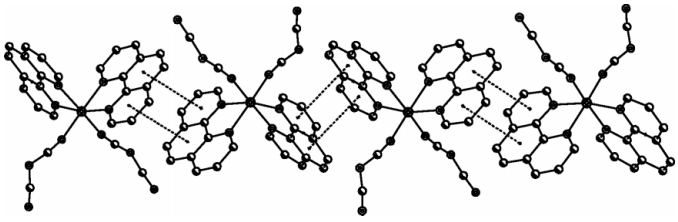


**Figure 2**
The chain structure of (I), constructed from $\pi$–$\pi$ stacking interactions (dotted lines) extending along [110].

two remaining N atoms (N2 and N8). The $Ni^{II}$ atom deviates from the equatorial plane by 0.095 (1) Å. The two crystallographically independent dca ligands coordinated to the Ni atom are in a *cis* arrangement. The mean $Ni-N_{phen}$ bond length [2.096 (2) Å] is longer than the mean $Ni-N_{dca}$ distance [2.050 (2) Å] as a result of the steric hindrance of the larger phen molecules.

The dca ligands are terminally coordinated, with only one of the nitrile N atoms involved in bonding interactions with the $Ni^{II}$ atom. The two crystallographically independent phen molecules are both almost planar, the largest deviation from the mean planes being 0.086 (1) Å for atom N2. The dihedral angle between the two phen mean planes is 81.92 (4)°, which is similar to the corresponding value [77.4 (1)°] for the Cu analogue (Potocnak *et al.*, 1995). There exist $\pi$–$\pi$ stacking interactions in (I), as shown in Fig. 2. Adjacent rings are exactly parallel; the perpendicular spacing of the rings is 3.514 Å, and the ring centroid-to-centroid distance is 3.816 Å. These $\pi$–$\pi$ stacking interactions form an extended chain structure along the [110] direction.

## Experimental

Aqueous solutions of $Ni(CH_3COO)_2\cdot 4H_2O$ (127 mg, 0.51 mmol) and sodium dicyanamide (91 mg, 1.02 mmol, in 4 ml) were mixed thoroughly, and then an ethanol solution (10 ml) of 1,10-phen (100 mg, 0.51 mmol) was added dropwise with stirring. The resulting mixture was filtered and the filtrate was left undisturbed at room temperature. Transparent green prismatic crystals were obtained after a few days. Yield 83.2% (based on Ni).

*Crystal data*

$[Ni(C_2N_3)_2(C_{13}H_8N_2)_2]$
$M_r = 551.22$
Monoclinic, $P2_1/c$
$a = 9.580$ (3) Å
$b = 15.176$ (5) Å
$c = 17.589$ (6) Å
$\beta = 105.021$ (4)°
$V = 2469.9$ (13) Å$^3$
$Z = 4$
$D_x = 1.482$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 4234 reflections
$\theta = 3.1$–27.5°
$\mu = 0.83$ mm$^{-1}$
$T = 293$ (2) K
Prism, green
0.32 × 0.23 × 0.20 mm

*Data collection*

Rigaku Mercury CCD diffractometer
$\omega$ scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku Corporation, 2002)
$T_{min} = 0.955$, $T_{max} = 1.000$
20 640 measured reflections
5668 independent reflections
3542 reflections with $I > 2\sigma(I)$
$R_{int} = 0.056$
$\theta_{max} = 27.5°$
$h = -12 \rightarrow 12$
$k = -19 \rightarrow 18$
$l = -22 \rightarrow 21$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.069$
$wR(F^2) = 0.158$
$S = 1.09$
5668 reflections
352 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.056P)^2 + 0.5P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.004$
$\Delta\rho_{max} = 0.89$ e Å$^{-3}$
$\Delta\rho_{min} = -0.63$ e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Ni1—N5 | 2.0492 (16) | Ni1—N1 | 2.0817 (14) |
| Ni1—N8 | 2.0505 (19) | Ni1—N2 | 2.1064 (17) |
| Ni1—N4 | 2.0802 (16) | Ni1—N3 | 2.1162 (15) |
| C25—N6—C26 | 126.3 (2) | C27—N9—C28 | 123.33 (19) |

H atoms were placed in idealized positions and treated as riding, with C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *CrystalClear* (Rigaku Corporation, 2002); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXTL* (Siemens, 1994); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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