Bis(1,10-phenanthroline)diazidonickel(II)

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In bis(1,10-phenanthroline)diazidonickel(II), [Ni($C_{12}H_8N_2$)$_2$(N_3)$_2$], the nickel atom is chelated by the two heterocyclic ligands in a distorted octahedral geometry; the two azido groups occupy *cis*-positions in the coordination polyhedron.

Comment

The azide anion, which has the ability to bind in several modes (Sheppard *et al.*, 1996), is an excellent unit for bridging divalent transition metal atoms such as copper, manganese and nickel. Such metal–azide complexes serve as models for the investigation of metalloenzymes and ferromagnetic/antiferromagnetic interactions (Aebersold *et al.*, 1998; Baffert *et al.*, 2001). The 1,10-phenanthroline complex of copper diazide exists as a dinuclear compound, which is bridged by the pseudohalide group; adjacent molecules are linked by lattice water molecules into a layer structure (Cheng *et al.*, 2002; Prabakaran, 2002). On the other hand, manganese diazide–bis(1,10-phenanthroline) is a monomeric compound (Shen *et al.*, 1999).

(I)
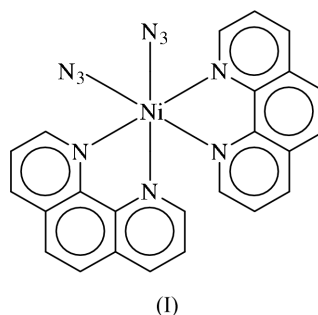
Bis(1,10-phenanthroline)diazidonickel, (I), also exists as a monomeric molecule; the metal atom adopts an NiN$_6$ octahedral geometry, and the two azido groups occupy *cis* positions (Fig. 1). The four dative Ni—N distances are not much different from the two Ni—N$_{azide}$ distances; the distortion of the geometry arises from the small bite of the heterocyclic donor ligands. The bond distances involving the metal atom are not significantly different from those found in the 2,2'-bipyridine analogue, which crystallizes as a monohydrate (Urtiaga *et al.*, 1995).

Adjacent molecules in the title complex are linked by a weak C20—H20$\cdots$N6^i interaction [C20$\cdots$N6^i = 3.405 (3) Å; C20—H20$\cdots$N6^i = 155°; symmetry code (i): $x - 1$, $y + 1$, z] into a linear chain along the diagonal direction in the *ab* plane (Fig. 2). Some evidence for this interaction, which involves the terminal nitrogen end of one the two azido groups, is
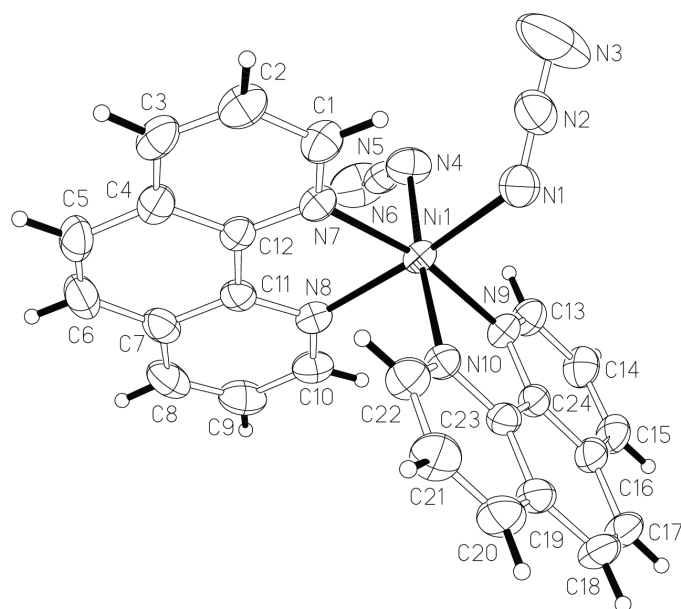
Figure 1
*ORTEP*II plot (Johnson, 1976) of bis(1,10-phenanthroline)diazidonickel with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radii.

suggested by the equivalent isotropic displacement factor of atom N6, which is only about half as large as that of the terminal nitrogen end of the second azido group, N3, which does not participate in any noticeable intermolecular interactions.

Experimental

A mixture of nickel sulfate (1 mmol, 0.23 g), 1,10-phenanthroline (2 mmol, 0.36 g) and sodium azide (2 mmol, 0.13 g) in 50 ml of a water–ethanol mixture (1:2 *v*:*v*) was stirred for several hours at room temperature. The mixture was filtered; dark green crystals separated from the solution after a week.

Crystal data

$C_{24}H_{16}N_{10}Ni$	$Z = 2$
$M_r = 503.18$	$D_x = 1.587$ Mg m^{-3}
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 8.200 (1)$ Å	Cell parameters from 30
$b = 11.002 (2)$ Å	reflections
$c = 12.408 (2)$ Å	$\theta = 3.0$–14.4°
$\alpha = 82.28 (2)°$	$\mu = 0.96$ mm^{-1}
$\beta = 82.26 (2)°$	$T = 298 (2)$ K
$\gamma = 72.57 (1)°$	Prism, green
$V = 1053.0 (3)$ Å^3	$0.48 \times 0.34 \times 0.30$ mm

Data collection

Siemens *P*4 four-circle	$R_{int} = 0.012$
diffractometer	$\theta_{max} = 25.0°$
ω scans	$h = 0 \rightarrow 9$
Absorption correction: ψ scan	$k = -12 \rightarrow 13$
(North *et al.*, 1968)	$l = -14 \rightarrow 14$
$T_{min} = 0.656, T_{max} = 0.762$	3 standard reflections
3984 measured reflections	every 97 reflections
3697 independent reflections	intensity decay: 2.6%
3277 reflections with $I > 2\sigma(I)$	
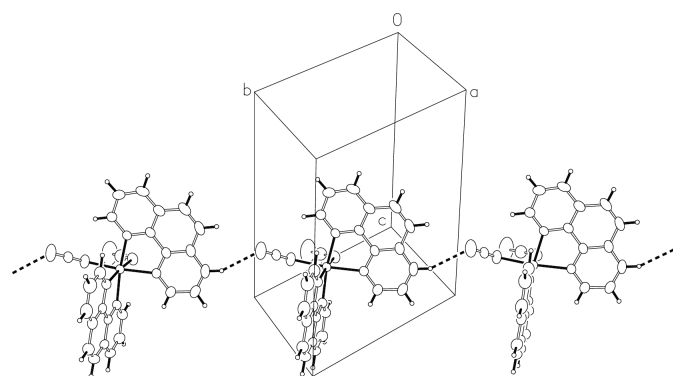
Figure 2
*ORTEP*II plot (Johnson, 1976), showing the intermolecular C—H···N interactions.

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0404P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.027$	$+ 0.2944P]$
$wR(F^2) = 0.072$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} < 0.001$
3697 reflections	$\Delta\rho_{max} = 0.24$ e Å^{-3}
316 parameters	$\Delta\rho_{min} = -0.30$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Ni1—N1	2.073 (2)	Ni1—N10	2.115 (2)
Ni1—N4	2.091 (2)	N1—N2	1.177 (3)
Ni1—N7	2.081 (2)	N2—N3	1.144 (3)
Ni1—N8	2.115 (2)	N4—N5	1.174 (3)
Ni1—N9	2.094 (2)	N5—N6	1.162 (3)
N1—Ni1—N4	91.6 (1)	N7—Ni1—N9	168.0 (1)
N1—Ni1—N7	94.3 (1)	N7—Ni1—N10	92.3 (1)
N1—Ni1—N8	173.3 (1)	N8—Ni1—N9	93.0 (1)
N1—Ni1—N9	93.6 (1)	N8—Ni1—N10	92.3 (1)
N1—Ni1—N10	87.9 (1)	N9—Ni1—N10	79.0 (1)
N4—Ni1—N7	93.4 (1)	N2—N1—Ni1	123.9 (1)
N4—Ni1—N8	88.8 (1)	N1—N2—N3	177.8 (3)
N4—Ni1—N9	95.3 (1)	N5—N4—Ni1	126.6 (2)
N4—Ni1—N10	174.3 (1)	N4—N5—N6	177.6 (2)
N7—Ni1—N8	78.9 (1)		

The H atoms were positioned geometrically, and were allowed to ride on their parent C atoms, with $U(H) = 1.2U_{eq}(C)$.

Data collection: *XSCANS* (Siemens, 1990); cell refinement: *XSCANS*; data reduction: Siemens software; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97 (Sheldrick, 1997).

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