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Key indicators

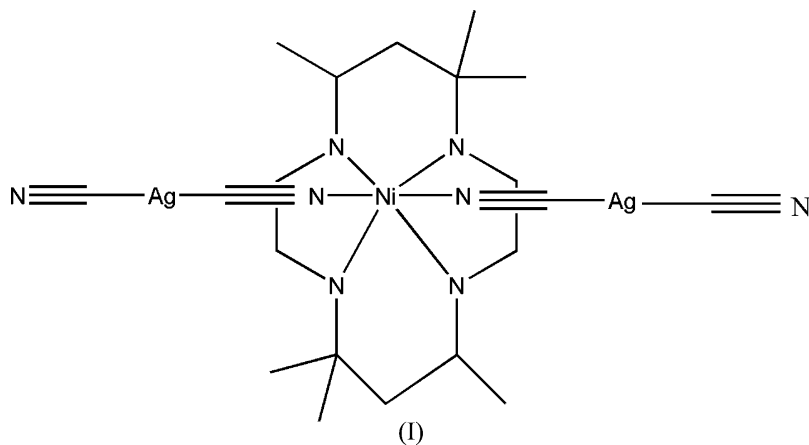
Single-crystal X-ray study
 $T = 294$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
 R factor = 0.028
 wR factor = 0.077
Data-to-parameter ratio = 19.3For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Di- μ -cyano-1:2 κ^2 C:N;2:3 κ^2 N:C-dicyano-
1 κ C,3 κ C-(5,7,7,12,14,14-hexamethyl-
1,4,8,11-tetraazacyclotetradecane-2 κ^4 N)-
nickel(II)disilver(I)

The title compound, $[\text{Ag}_2\text{Ni}(\text{CN})_4(\text{C}_{15}\text{H}_{36}\text{N}_3)]$, has been prepared by the reaction of teta (5,7,7,12,14,14-hexamethyl-1,4,8,11-tetraazacyclotetradecane), $\text{K}[\text{Ag}(\text{CN})_2]$ and $\text{Ni}(\text{ClO}_4)_2$. The Ni atom, lying on a centre of symmetry, is octahedrally coordinated by four teta N atoms and two cyano N atoms.

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Comment

The number of structurally studied compounds of nickel(II) with the cyanide ligand is, to our knowledge, surprisingly low: three isocyanide mononuclear compounds (Tsintsadze *et al.*, 1975; Cortes *et al.*, 1988; Urtiaga *et al.*, 1994) three dinuclear cyanide-bridged compounds (Cortes *et al.*, 1986; Arriortua *et al.*, 1988; Duggan & Hendrickson, 1974; Escuer *et al.*, 1996) and three cyano-bridged one-dimensional chain compounds (Escuer *et al.*, 1996; Colacio *et al.*, 2003; Colacio *et al.*, 2004). In this paper, we report the synthesis and crystal structure of a new mononuclear compound, *viz.* $\text{Ni}(\text{teta})[\text{Ag}(\text{CN})_2]_2$, (I) (teta is 5,7,7,12,14,14-hexamethyl-1,4,8,11-tetraazacyclotetradecane).



In order to study the magnetic behaviour of polynuclear nickel(II) compounds, we used teta as the main ligand and $\text{K}[\text{Ag}(\text{CN})_2]$ as the bridging ligand in the hope of obtaining a one-dimensional chain Ni complex. Unexpectedly, the title complex was obtained.

Single-crystal X-ray diffraction analysis reveals that the structure of the title complex consists of an $[\text{Ni}(\text{teta})]^{2+}$ cation and an $[\text{Ag}(\text{CN})_2]^-$ anion. The Ni^{II} atom, lying on a centre of symmetry, is surrounded by six N atoms, *viz.* four teta N atoms and two cyano N atoms (Fig. 1). The Ni^{II} atom is hexacoordinate. Atoms N3 and N4 and their symmetry equivalents from the teta ligand occupy the four equatorial positions, and cyano atom N2 and its symmetry equivalent are in the axial

sites, giving an elongated octahedron around the Ni^{II} atom. Ni–N bond distances range from 2.079 (3) to 2.131 (3) Å. However, the axial Ni–N bond length [2.131 (3) Å] is longer than the average equatorial Ni–N bond length [2.092 (3) Å]. The *trans* bond angles are, by symmetry, 180°, but the N4–Ni1–N3 [94.84 (10)°], N4–Ni1–N3' [85.16 (10)°] and N4–Ni1–N2 [87.50 (10)°] angles indicate a distorted octahedron. The packing diagram viewed along the *b* axis (Fig. 2) shows zigzag stacks of molecules.

Experimental

A solution of teta (0.1 mmol) in methanol (10 ml) was added to a solution of Ni(ClO₄)₂ (0.1 mmol) in methanol (10 ml). The mixture was refluxed for 30 min and a solution of K[Ag(CN)₂] (0.2 mmol) in H₂O (5 ml) was added to this mixture. The solution was refluxed for another 2 h. After cooling to room temperature and filtration, the filtrate was slowly evaporated at room temperature over a period of one month to yield pale-purple prism-shaped crystals of Ni(teta)-[Ag(CN)₂]₂ suitable for X-ray analysis. Analysis calculated for C₂₀H₃₂Ag₂N₈Ni: C 36.45, H 4.89, Ag 32.74, Ni 8.91, N 17.00%; found: C 36.47, H 4.91%.

Crystal data

[Ag ₂ Ni(CN) ₄ (C ₁₅ H ₃₆ N ₃)]	Z = 4
<i>M_r</i> = 663.02	<i>D_x</i> = 1.762 Mg m ⁻³
Orthorhombic, <i>Pbca</i>	Mo <i>K</i> α radiation
<i>a</i> = 16.435 (2) Å	<i>μ</i> = 2.32 mm ⁻¹
<i>b</i> = 8.6228 (13) Å	<i>T</i> = 294 (2) K
<i>c</i> = 17.634 (3) Å	Prism, pale purple
<i>V</i> = 2498.9 (6) Å ³	0.22 × 0.16 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer	15725 measured reflections
<i>φ</i> and <i>ω</i> scans	2972 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	2229 reflections with <i>I</i> > 2σ(<i>I</i>)
<i>T_{min}</i> = 0.629, <i>T_{max}</i> = 0.737	<i>R_{int}</i> = 0.030
	<i>θ_{max}</i> = 27.8°

Refinement

Refinement on <i>F</i> ²	$w = 1/[\sigma^2(F_o^2) + (0.035P)^2 + 2.1646P]$
$R[F^2 > 2\sigma(F^2)] = 0.028$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.077$	(Δ/σ) _{max} = 0.004
<i>S</i> = 1.04	$\Delta\rho_{max} = 0.93 \text{ e \AA}^{-3}$
2972 reflections	$\Delta\rho_{min} = -0.51 \text{ e \AA}^{-3}$
154 parameters	
H atoms treated by a mixture of independent and constrained refinement	

The H atoms attached to N3 and N4 were refined freely [N–H = 0.85 (3) and 0.94 (3) Å, respectively]. Other H atoms were included in calculated positions and refined as riding on their parent atoms, with C–H = 0.96–0.97 Å; *U*_{iso}(H) = 1.19*U*_{eq}(C) or *U*_{iso}(H) = 1.5*U*_{eq}(C_{methyl}).

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1998); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1998); software used to prepare material for publication: SHELXTL.

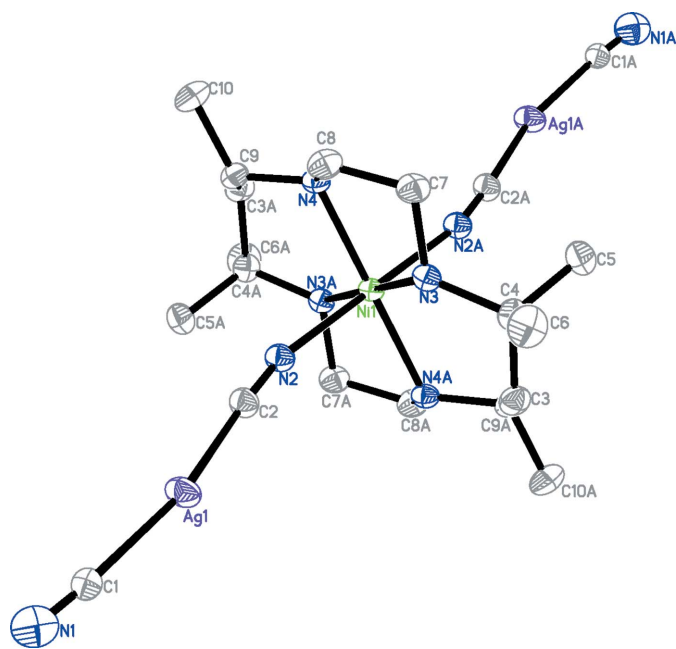


Figure 1
The molecular structure of the title compound with 30% probability displacement ellipsoids. The H atoms are omitted. [Symmetry code: (A) $-x + 2, -y + 2, -z$.]

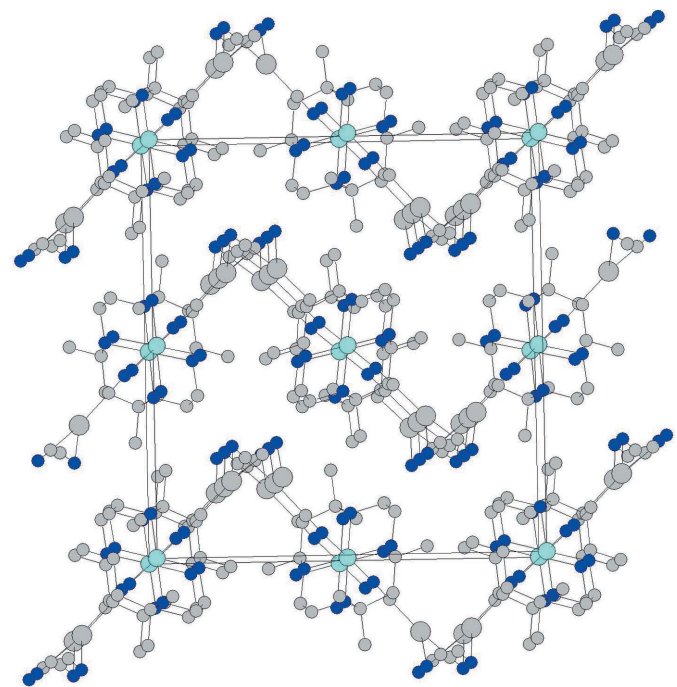


Figure 2
The packing of the title compound, viewed down the *b* axis.

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