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Redetermination of $[Ni(NH_3)_6]Cl_2$ at 173 K

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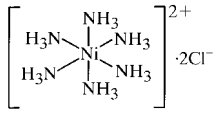
The structure of hexaamminenickel(II) dichloride determined previously by Eßmann *et al.* [Eßmann, Kreiner, Niemann, Rechenbach, Schmieding, Sichla, Zachwieja & Jacobs (1996). *Z. Anorg. Allg. Chem.* **622**, 1161–1166] was redetermined at 173 K. There are no significant differences between these two structures.

Comment

In an attempt to obtain a polymer of Ni^{2+} and 2,5-bis(1-pyrazolyl)hydrochinon *via* a new route, crystals of the title compound, (I), were obtained. After structure determination it turned out that the crystals contained $[Ni(NH_3)_6]Cl_2$, whose structure was determined previously by Eßmann *et al.* (1996). The cell axes are a little bit shorter at 173 K and the anisotropic displacement parameters are, as expected, smaller. However, there are no significant differences between the structures at different temperatures. The difference in the Ni—N bond lengths in both structures is 0.016 Å and the difference between the two N—H bond lengths (0.1 Å) is less than their standard deviation (0.2 Å).



Experimental

A solution of 2,5-bis(1-pyrazolyl)hydrochinon (0.063 g, 0.260 mmol) in 10 ml CH_2Cl_2 was layered with a solution of 5 ml concentrated aqueous ammonia and $NiCl_2{\cdot}6H_2O$ (0.161 g, 0.677 mmol). Formation of purple crystals was observed after one week. The liquid was removed from the crystals by filtration. The remaining crystalline compound was washed with 5 ml CH_2Cl_2 (yield: 0.034 g, 0.147 mmol; 22%).

Crystal data

$[Ni(NH_3)_6]Cl_2$
M_r = 231.81
Cubic, $Fm\bar{3}m$
a = 10.029 (2) Å
V = 1008.7 (3) Å^3
Z = 4
D_x = 1.526 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 339 reflections
θ = 1–20°
μ = 2.401 mm^{-1}
T = 173 (2) K
Octahedron, purple
0.60 × 0.50 × 0.50 mm

Data collection

Siemens CCD three-circle diffractometer
ω scans
Absorption correction: numerical numerical
T_{min} = 0.327, T_{max} = 0.380
4406 measured reflections
116 independent reflections
115 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 31.38°
$h = -14 \rightarrow 14$
$k = -14 \rightarrow 14$
$l = -14 \rightarrow 12$
32 standard reflections
frequency: 720 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.013
$wR(F^2)$ = 0.033
S = 1.319
116 reflections
10 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0135P)^2 + 0.5364P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0173 (12)

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···Cl1	0.859 (16)	2.750 (16)	3.5663 (7)	159 (2)
Ni1—N1···H1				112.0 (15)

The H atom was refined freely with a site-occupation facter of 0.75.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997).

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