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Structure of Chloro(3,7-diazanonanediamide)nickel(II) Perchlorate

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Abstract. $[NiCl(C_7H_{16}N_4O_2)]ClO_4$, $M_r = 381{\cdot}8$, triclinic, $P\bar{1}$, $a = 7{\cdot}822\ (3)$, $b = 7{\cdot}924\ (3)$, $c = 12{\cdot}637\ (4)$ Å, $\alpha = 107{\cdot}37\ (3)$, $\beta = 91{\cdot}52\ (3)$, $\gamma = 110{\cdot}50\ (3)°$, $V = 692{\cdot}6\ (5)$ Å^3, $Z = 2$, $D_x = 1{\cdot}831$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 18{\cdot}2$ cm^{-1}, $F(000) = 392$, $T = 296$ K, $R = 4{\cdot}37\%$, $wR = 4{\cdot}79\%$ for 2189 independent reflections with $I > 3{\cdot}0\sigma(I)$. This compound is a five-coordinate Ni complex. The nickel(II) ion is in a slightly distorted square pyramid with the diaminodiamide equatorial and a Cl anion axial.

Experimental. The ligand, *L*-1,3,1, was prepared from 1,3-propanediamine (41·5 ml, 0·5 mol) and 2-chloroacetamide (55·8 g, 0·6 mol) in *N,N'*-dimethylformamide (50 ml) by heating under reflux for 2 h. The title compound was obtained by adding *L*-1,3,1 (5·26 g) to an aqueous solution of nickel(II) perchlorate (4·76 g) and heating until it was completely dissolved; the solution was filtered immediately while hot. After evaporation of the filtrate, deep-blue rod-like crystals of $[NiCl(L\text{-}1,3,1)]ClO_4$ were formed. A crystal 0·22 × 0·28 × 0·40 mm was selected for the determination of lattice constants (18 reflections, $11{\cdot}0 \le 2\theta \le 27{\cdot}4°$, Mo $K\alpha$ radiation, Nicolet *R*2*m*/V diffractometer, graphite monochromator) as well as for the data collection. Corrections for absorption effects were based on ψ scans of a few suitable reflections with χ values close to 90°. Max./min. transmission factors: 0·925/0·622. Total of 3007 reflections measured with $[(\sin\theta)/\lambda]_{max} = 0{\cdot}595$ Å^{-1} and in the range $0 \le h \le 9$, $-9 \le k \le 8$, $-15 \le l \le 15$. No significant variation in intensities of three standards monitored every 50 reflections. Scan width of 1·2° plus $K\alpha$ separation and scan speed 2·93–14·65° min^{-1}. 2189 unique structure amplitudes with $I > 3{\cdot}0\sigma(I)$. The structure was solved by direct methods and refined by full-matrix least squares based on *F* values. All of the non-H atoms were refined anisotropically. H atoms were placed in idealized positions (C—H = 0·96 Å, H—C—H = 109·4°) and refined with fixed *U* (0·08 Å^2). At convergence $R = 4{\cdot}37\%$, $wR = 4{\cdot}79\%$, $w = [\sigma^2(F) + 0{\cdot}00068F^2]^{-1}$, $\sigma^2(F)$ based on counting statistics,

Table 1. *Atomic coordinates* (×10^4) *and equivalent isotropic displacement coefficients* (Å^2 × 10^3)

Equivalent isotropic *U* defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	*y*	*z*	U_{eq}
Ni	2058 (1)	4438 (1)	1866 (1)	26 (1)
O(1)	1708 (3)	2054 (3)	669 (2)	34 (1)
O(2)	−234 (3)	4545 (3)	1269 (2)	37 (1)
N(1)	1907 (4)	6437 (4)	3223 (2)	31 (1)
N(2)	3860 (4)	3763 (4)	2646 (2)	32 (1)
N(3)	2495 (5)	−495 (5)	282 (3)	44 (1)
N(4)	−1607 (4)	6609 (5)	1348 (3)	38 (1)
C(1)	2743 (4)	1249 (4)	857 (3)	31 (1)
C(2)	4384 (5)	2397 (5)	1774 (3)	37 (1)
C(3)	5433 (5)	5461 (5)	3376 (3)	33 (1)
C(4)	4778 (5)	6715 (5)	4282 (3)	34 (1)
C(5)	3737 (5)	7762 (5)	3906 (3)	34 (1)
C(6)	903 (5)	7448 (5)	2825 (3)	37 (1)
C(7)	−389 (4)	6106 (5)	1749 (3)	29 (1)
Cl(1)	4198 (1)	6812 (1)	1027 (1)	36 (1)
Cl(2)	−761 (1)	1672 (1)	3595 (1)	42 (1)
O(4)	−331 (7)	1048 (6)	2507 (4)	90 (2)
O(5)	−1784 (5)	65 (5)	3884 (4)	86 (2)
O(6)	−1702 (9)	2888 (8)	3663 (6)	131 (4)
O(7)	936 (8)	2694 (9)	4287 (4)	140 (3)

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Table 2. *Bond lengths* (Å) *and angles* (°)

Ni–O(1)	1·959 (2)	Ni–O(2)	1·966 (3)
Ni–N(1)	1·996 (3)	Ni–N(2)	2·004 (3)
Ni–Cl(1)	2·558 (1)	O(1)–C(1)	1·249 (5)
O(2)–C(7)	1·254 (4)	N(1)–C(5)	1·496 (4)
N(1)–C(6)	1·474 (6)	N(2)–C(2)	1·476 (5)
N(2)–C(3)	1·488 (3)	N(3)–C(1)	1·297 (5)
N(4)–C(7)	1·297 (6)	C(1)–C(2)	1·514 (4)
C(3)–C(4)	1·506 (5)	C(4)–C(5)	1·505 (6)
C(6)–C(7)	1·521 (4)	Cl(2)–O(4)	1·408 (5)
Cl(2)–O(5)	1·405 (4)	Cl(2)–O(6)	1·388 (8)
Cl(2)–O(7)	1·395 (5)		

O(1)–Ni–O(2)	92·5 (1)	O(1)–Ni–N(1)	165·6 (1)
O(2)–Ni–N(1)	83·5 (1)	O(1)–Ni–N(2)	84·3 (1)
O(2)–Ni–N(2)	162·9 (1)	N(1)–Ni–N(2)	95·4 (1)
O(1)–Ni–Cl(1)	99·3 (1)	O(2)–Ni–Cl(1)	95·4 (1)
N(1)–Ni–Cl(1)	94·8 (1)	N(2)–Ni–Cl(1)	101·6 (1)
Ni–O(1)–C(1)	113·5 (2)	Ni–O(2)–C(7)	112·3 (2)
Ni–N(1)–C(5)	114·0 (2)	Ni–N(1)–C(6)	106·4 (2)
C(5)–N(1)–C(6)	112·4 (3)	Ni–N(2)–C(2)	106·7 (2)
Ni–N(2)–C(3)	113·2 (3)	C(2)–N(2)–C(3)	115·0 (3)
O(1)–C(1)–N(3)	122·8 (3)	O(1)–C(1)–C(2)	118·0 (3)
N(3)–C(1)–C(2)	119·2 (4)	N(2)–C(2)–C(1)	107·7 (3)
N(2)–C(3)–C(4)	111·4 (3)	C(3)–C(4)–C(5)	116·3 (3)
N(1)–C(5)–C(4)	112·2 (3)	N(1)–C(6)–C(7)	109·3 (3)
O(2)–C(7)–N(4)	121·8 (3)	O(2)–C(7)–C(6)	118·8 (4)
N(4)–C(7)–C(6)	119·3 (3)	O(4)–Cl(2)–O(5)	108·6 (3)
O(4)–Cl(2)–O(6)	111·1 (4)	O(5)–Cl(2)–O(6)	111·9 (3)
O(4)–Cl(2)–O(7)	105·3 (3)	O(5)–Cl(2)–O(7)	111·1 (3)
O(6)–Cl(2)–O(7)	108·6 (4)		

$S = 1{\cdot}44$, $(\Delta/\sigma)_{max} = 0{\cdot}038$, $\Delta\rho_{max} = 0{\cdot}55$, $\Delta\rho_{min} = -0{\cdot}51$ e Å^{-3}. Scattering factors were taken from Cromer & Waber (1974). All calculations were performed on a MicroVAXII computer system using *SHELXTL-Plus* program (Sheldrick, 1986). Atomic parameters are given in Table 1, bond distances and angles in Table 2.* A molecular drawing of the structure and the atomic numbering system are given in Fig. 1.

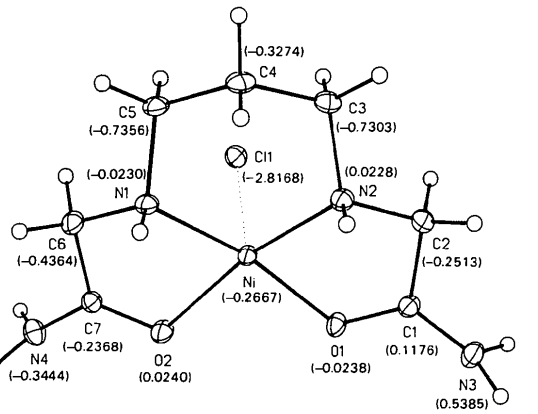

Fig. 1. The molecular drawing of $[NiCl(L\text{-}1,3,1)]^+$ with the deviations of atoms (Å) from the N_2O_2 plane (the atom positions below the plane are indicated by negative signs).

Related literature. Crystal structures of many copper(II) diazadiamide complexes have been determined and reported (Hong, Lee, Lee, Chao & Chung, 1987; Lu, Shan, Chao & Chung, 1987).

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51752 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Third Modification of $[(Ph_3P)_2N][H_3Ru_4(CO)_{12}]$

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Abstract. Bis(triphenylphosphoranylidene)ammonium tri-μ-hydrido-*tetrahedro*-tetrakis(tricarbonylruthenate), $C_{36}H_{30}NP_2^+.C_{12}H_3O_{12}Ru_4^-$, $M_r = 1282$, monoclinic, $P2_1/c$, $a = 11{\cdot}657$ (2), $b = 28{\cdot}921$ (4), $c = 14{\cdot}673$ (3) Å, $\beta = 100{\cdot}13$ (1)°, $V = 4869{\cdot}4$ Å^3, $Z = 4$, $D_x = 1{\cdot}749$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 1{\cdot}3$ mm^{-1}, $F(000) = 2520$, $T = 293$ K. The structure was refined to $R = 0{\cdot}039$ for 6634 unique observed

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