

Table 3. Bond angles (°) in the [FeCl(*tp*)] molecule

N(1)FeN(2)	86.4 (1)	C(b4)C(a4)C(m2)	125.0 (4)
N(1)FeN(3)	151.8 (1)	N(3)C(a5)C(b5)	109.0 (4)
N(1)FeN(4)	86.8 (1)	N(3)C(a5)C(m2)	125.8 (4)
N(2)FeN(3)	87.0 (1)	C(b5)C(a5)C(m2)	125.2 (4)
N(2)FeN(4)	153.4 (1)	N(3)C(a6)C(b6)	109.4 (4)
N(3)FeN(4)	87.0 (1)	N(3)C(a6)C(m3)	126.2 (4)
N(1)FeCl	104.7 (1)	C(b6)C(a6)C(m3)	124.4 (4)
N(2)FeCl	103.5 (1)	N(4)C(a7)C(b7)	110.0 (4)
N(3)FeCl	103.4 (1)	N(4)C(a7)C(m3)	125.6 (4)
N(4)FeCl	103.0 (1)	C(b7)C(a7)C(m3)	124.4 (4)
FeN(1)C(a1)	126.2 (3)	N(4)C(a8)C(b8)	110.0 (3)
FeN(1)C(a2)	126.1 (3)	N(4)C(a8)C(m4)	125.2 (4)
FeN(2)C(a3)	127.2 (3)	C(b8)C(a8)C(m4)	124.7 (4)
FeN(2)C(a4)	126.2 (3)	C(a2)C(m1)C(1)	116.4 (4)
FeN(3)C(a5)	125.7 (3)	C(a3)C(m1)C(1)	120.1 (4)
FeN(3)C(a6)	126.4 (3)	C(a2)C(m1)C(a3)	123.4 (4)
FeN(4)C(a7)	126.8 (3)	C(a4)C(m2)C(7)	117.6 (4)
FeN(4)C(a8)	127.2 (3)	C(a5)C(m2)C(7)	117.5 (3)
C(a1)N(1)C(a2)	105.9 (3)	C(a4)C(m2)C(a5)	124.8 (4)
C(a3)N(2)C(a4)	105.6 (3)	C(a6)C(m3)C(13)	118.0 (4)
C(a5)N(3)C(a6)	106.0 (3)	C(a7)C(m3)C(13)	117.7 (4)
C(a7)N(4)C(a8)	105.5 (3)	C(a6)C(m3)C(a7)	124.1 (4)
N(1)C(a1)C(b1)	109.2 (4)	C(a1)C(b4)C(19)	116.9 (4)
N(1)C(a1)C(m4)	126.4 (4)	C(a8)C(m4)C(19)	119.1 (4)
C(b1)C(a1)C(m4)	124.3 (4)	C(a1)C(m4)C(a8)	124.0 (4)
N(1)C(a2)C(b2)	109.8 (4)	C(a1)C(b1)C(b2)	108.1 (4)
N(1)C(a2)C(m1)	126.9 (4)	C(a2)C(b2)C(b1)	106.9 (4)
C(b2)C(a2)C(m1)	123.2 (4)	C(a3)C(b3)C(b4)	107.0 (4)
N(2)C(a3)C(b3)	110.1 (4)	C(a4)C(b4)C(b3)	107.9 (4)
N(2)C(a3)C(m1)	125.4 (4)	C(a5)C(b5)C(b6)	108.3 (4)
C(b3)C(a3)C(m1)	124.5 (4)	C(a6)C(b6)C(b5)	107.3 (4)
N(2)C(a4)C(b4)	109.3 (3)	C(a7)C(b7)C(b8)	107.1 (4)
N(2)C(a4)C(m2)	125.6 (4)	C(a8)C(b8)C(b7)	107.4 (4)

Scheidt & Lee (1987). Another crystalline form of this compound has been long known (Hoard, Cohen & Glick, 1967). The current form differs significantly from this one in core conformation. The displacement of the iron from the N_4 plane is 0.49 Å and 0.57 Å from the 24-atom plane and hence displays a small but real C_{4v} doming. The previous form has a planar porphyrinato

core and a displacement of iron of 0.38 Å from both the N_4 and 24-atom planes. Further, Fe–N is shorter at 2.049 Å.

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Structure of {4-[2-(2-Aminoethylamino)ethylimino]pentan-2-onato-*N,N',N'',O*}nickel(II) Iodide Monohydrate

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Abstract. [Ni(C₉H₁₈N₃O)]I.H₂O, $M_r = 387.87$, monoclinic, $P2_1/n$, $a = 7.628$ (1), $b = 10.005$ (3), $c = 18.626$ (3) Å, $\beta = 99.05$ (1)°, $V = 1403.7$ (5) Å³, $Z = 4$, $D_m = 1.86$ (3), $D_x = 1.84$ Mg m⁻³, $\lambda(\text{Mo K}\alpha) =$

0.71073 Å, $\mu = 3.56$ mm⁻¹, $F(000) = 768$, $T = 295$ K, $R = 0.0365$ for 2468 unique observed reflections. The structure consists of [Ni(C₉H₁₈N₃O)]⁺ complex cations, iodide anions and molecules of water. The coordination of Ni is almost square-planar with the ligand C₉H₁₈N₃O⁻ bonded to Ni through one O and three N

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Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\times 10^3$) of non-H atoms with e.s.d.'s in parentheses

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	$U_{eq}(\text{\AA}^2)$
I	6821.5 (5)	4488.2 (4)	8420.8 (2)	48.6 (2)
Ni	6900.8 (8)	3920.8 (6)	3961.0 (3)	36.1 (2)
O(1)	7549 (5)	5665 (3)	3942 (2)	44 (1)
O(2)	5160 (7)	1647 (6)	7342 (3)	80 (2)
N(1)	6794 (7)	3827 (5)	2920 (3)	47 (2)
N(2)	6154 (6)	2123 (5)	3903 (3)	46 (1)
N(3)	7061 (6)	3759 (5)	4961 (2)	42 (1)
C(1)	6106 (9)	2512 (7)	2639 (3)	59 (2)
C(2)	6647 (7)	1533 (6)	3245 (4)	52 (2)
C(3)	6829 (9)	1466 (6)	4598 (4)	58 (2)
C(4)	6577 (9)	2439 (7)	5182 (3)	57 (2)
C(5)	7460 (8)	4712 (6)	5419 (3)	47 (2)
C(6)	7881 (7)	6009 (6)	5207 (3)	48 (2)
C(7)	7901 (7)	6414 (6)	4514 (3)	45 (2)
C(8)	7510 (12)	4486 (8)	6237 (3)	71 (3)
C(9)	8315 (11)	7832 (6)	4360 (4)	64 (2)

Table 2. Bond lengths (\AA) and angles ($^\circ$) with e.s.d.'s in parentheses

Ni—O(1)	1.816 (3)	O(1)—Ni—N(1)	89.8 (2)
Ni—N(1)	1.930 (6)	O(1)—Ni—N(3)	97.4 (2)
Ni—N(2)	1.885 (5)	N(1)—Ni—N(2)	86.1 (2)
Ni—N(3)	1.854 (4)	N(2)—Ni—N(3)	86.9 (2)
O(1)—C(7)	1.296 (7)	Ni—O(1)—C(7)	124.2 (3)
N(1)—C(1)	1.481 (9)	Ni—N(1)—C(1)	110.8 (3)
N(2)—C(2)	1.462 (9)	Ni—N(2)—C(2)	108.6 (3)
N(2)—C(3)	1.471 (9)	Ni—N(2)—C(3)	108.4 (4)
N(3)—C(4)	1.448 (8)	C(2)—N(2)—C(3)	117.2 (5)
N(3)—C(5)	1.284 (7)	Ni—N(3)—C(4)	112.7 (3)
C(1)—C(2)	1.502 (9)	Ni—N(3)—C(5)	125.0 (4)
C(3)—C(4)	1.495 (9)	C(4)—N(3)—C(5)	122.2 (4)
C(5)—C(6)	1.408 (8)	N(1)—C(1)—C(2)	105.9 (5)
C(5)—C(8)	1.535 (8)	N(2)—C(2)—C(1)	106.9 (5)
C(6)—C(7)	1.355 (8)	N(2)—C(3)—C(4)	106.4 (5)
C(7)—C(9)	1.491 (9)	N(3)—C(4)—C(3)	108.6 (5)
I—Ni ⁱ	5.130 (2)	N(3)—C(5)—C(6)	122.6 (5)
I—Ni ⁱⁱ	5.141 (1)	N(3)—C(5)—C(8)	121.1 (5)
I—O(2)	3.595 (6)	C(6)—C(5)—C(8)	116.3 (5)
I—O(2 ⁱⁱⁱ)	3.607 (6)	C(5)—C(6)—C(7)	125.4 (5)
I—N(1 ⁱ)	3.809 (5)	O(1)—C(7)—C(6)	125.3 (5)
I—N(2 ^{iv})	3.660 (5)	O(1)—C(7)—C(9)	114.5 (5)
O(2)—N(1 ^v)	2.973 (8)	C(6)—C(7)—C(9)	120.1 (5)

Symmetry code: (i) $1-x, 1-y, 1-z$; (ii) $x-0.5, -y+0.5, z-0.5$; (iii) $-x+1.5, y+0.5, -z+1.5$; (iv) $x+0.5, -y+0.5, z+0.5$; (v) $x-0.5, -y+0.5, z+0.5$.

atoms. There are five $I \cdots H(O)$, $I \cdots H(N)$ and $O(2) \cdots H(N)$ distances, which correspond to hydrogen-bond distances.

Experimental. The crystals were prepared by crystallization from a mixture of nickel(II) iodide, acetylacetone (2,5-pentanedione) and diethylenetriamine from aqueous ethanol. Light-cherry crystals were obtained, stable in air and to X-rays. Density was determined by flotation in a mixture of CH_2I_2 and CCl_4 . A spherical crystal of $r = 0.32$ mm was used for the measurements with an automatic Hilger & Watts four-circle diffractometer; $\omega-2\theta$ scan; 29 reflections with 2θ from 7.38 to 22.12° were used for refinement of lattice

parameters; $\max. \sin\theta/\lambda = 0.595 \text{ \AA}^{-1}$. The correction for absorption of a spherical crystal was applied with min. and max. values 4.741 and 5.148; $h: 0 \rightarrow 9$, $k: 0 \rightarrow 11$, $l: -22 \rightarrow 21$. Three standard reflections were measured after every 30 reflections. They showed no significant variation. 2631 reflections were measured of which 2468 were unique reflections and 236 unobserved [$I < 1.96\sigma(I)$]. The structure was solved by the heavy-atom method; F magnitudes were refined in the full-matrix least-squares refinement. H(C) atoms were placed in calculated positions with C—H distance 1.08 \AA . H(N) and H(O) atoms were localized from a $\Delta\rho$ map and refined with restriction according to bonding in NH_3 and H_2O respectively; all positional parameters were refined with the exception of H(C) atoms. Isotropic thermal parameters of H atoms and anisotropic thermal parameters of the other atoms were refined; $R = 0.0365$, $wR = 0.0474$, $w = 0.028/[\sigma^2(F) + 0.0009F^2]$; $(\Delta/\sigma)_{\max} = 0.219$ for z coordinate of I, max. and min. heights in final $\Delta\rho$ map were 0.47 and $-0.72 e \text{ \AA}^{-3}$. ICL 4-72 computer was used with program *SHELX76* (Sheldrick, 1976). Atomic scattering factors and corrections for anomalous dispersion were taken from *International Tables for X-ray Crystallography* (1974).*

Atomic parameters are given in Table 1, bond distances and angles in Table 2. The complex cation is shown in Fig. 1.

Related literature. The bond distances and angles in the complex cation correspond to those reported e.g. by Haber, Loub, Podlahová, Kopf & Weiss (1988) for

* Lists of atomic coordinates and isotropic thermal parameters for H atoms, bond distances and angles for the H(N) atoms, anisotropic thermal parameters for the non-H atoms and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51812 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

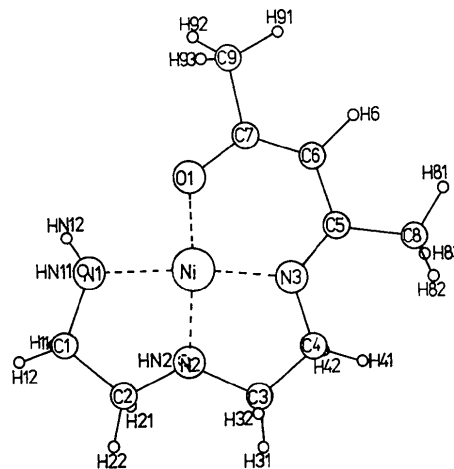


Fig. 1. A view of the $[Ni(C_9H_{18}N_3O)]^+$ complex cation along a.

C₉H₁₈N₃O⁻ and by Podlahová, Knížek, Loub & Hašek (1988) for C₁₁H₁₆N₃O⁻. The assumed hydrogen bonds are in agreement with infrared spectra, in which N—H vibrations are shifted to lower frequencies compared with [Ni(C₁₁H₁₆N₃O)]ClO₄ (Podlahová *et al.*, 1988), where no hydrogen bonds occur (3303→3253, 3254→3225, 3207→3121 cm⁻¹). O—H vibrations are shifted to lower frequencies when compared with H₂O in the matrix (Tursi & Nixon, 1970) (3725→3494, 3632→3430 cm⁻¹).

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Structure of *trans*-Di- μ -chloro-dichlorobis(triethylphosphine)diplatinum

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Abstract. [Pt₂Cl₄{P(C₂H₅)₃}₂], *M_r* = 768.31, monoclinic, *P*2₁/*c*, *a* = 7.6461 (6), *b* = 12.1981 (12), *c* = 12.9280 (20) Å, β = 113.226 (6)°, *V* = 1108.0 Å³, *Z* = 2 (implying that each dimer lies on a crystallographic inversion centre), *D_x* = 2.302 Mg m⁻³, λ (Mo *K* α) = 0.71073 Å, μ = 13.37 mm⁻¹, *F*(000) = 712, *T* = 293 K, *R* = 0.0353 for 1275 unique observed reflections. The chloro-bridged dimer is centrosymmetric, with Pt—Cl distances of 2.282 (3) Å (terminal) and 2.318 (3), 2.431 (3) Å (bridging). Angles around Pt range from 83.52 (9)° for Cl(1)—Pt—Cl(1') to 95.93 (9)° for P—Pt—Cl(1), while the angle subtended at the bridging Cl is 96.48 (10)°.

Experimental. Lamellar yellow crystal, 0.38 × 0.35 × 0.08 mm, Stoe STADI-4 diffractometer, graphite-monochromated Mo*K* α X-radiation, cell parameters from 40 reflections measured at $\pm\omega$ ($2\theta = 25$ – 30°). For data collection, ω – 2θ scans, $2\theta_{\max} = 45^\circ$, *h* –8→7, *k* 0→13, *l* 0→13, no significant crystal movement or decay, semi-empirical absorption correction applied using ψ scans, 1453 unique reflections giving 1275 with $F \geq 6\sigma(F)$ for structure solution (the Pt position was deduced from a Patterson synthesis and subsequent iterative cycles of least-squares refinement and difference Fourier synthesis located the remaining non-H atoms) and refinement [using full-matrix least squares on *F* (Sheldrick, 1976)]. Anisotropic thermal parameters for Pt, Cl and P, isotropic for C, H atoms refined in fixed, calculated positions. At convergence, *R*, *wR* = 0.0353, 0.0512, *S* = 0.658 for 61 parameters,

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(Δ/σ)_{max} in final cycle 0.021, max. and min. residues in final difference Fourier synthesis 1.20, –1.77 e Å⁻³ respectively. The weighting scheme $w^{-1} = \sigma^2(F) + 0.00545F^2$ gave satisfactory agreement analyses. Scattering factors were inlaid (Sheldrick, 1976) except for Pt (Cromer & Mann, 1968). Atom coordinates and equivalent isotropic thermal parameters are given in Table 1 while selected molecular geometry parameters appear in Table 2.† The atom-numbering scheme for the dimer is shown in Fig. 1, which was generated using *ORTEP* (Mallinson & Muir, 1985). Molecular geometry calculations were performed using *CALC* (Gould & Taylor, 1985).

† Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51831 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates and isotropic thermal parameters with e.s.d.'s

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}/U_{iso}</i> (Å ²)
Pt	0.48880 (4)	0.10145 (3)	0.095240 (20)	0.0386 (4)
P	0.3322 (3)	0.13401 (22)	0.20394 (19)	0.0459 (15)
Cl(1)	0.3440 (4)	–0.06527 (21)	0.02682 (20)	0.0599 (17)
Cl(2)	0.6502 (4)	0.26218 (20)	0.15425 (22)	0.0658 (17)
C(1A)	0.1605 (11)	0.0277 (7)	0.1937 (7)	0.0435 (20)
C(1B)	0.4880 (14)	0.1476 (10)	0.3485 (9)	0.062 (3)
C(1C)	0.1981 (15)	0.2602 (10)	0.1678 (9)	0.067 (3)
C(2A)	0.0543 (15)	0.0409 (10)	0.2718 (8)	0.069 (3)
C(2B)	0.6149 (16)	0.0485 (10)	0.4002 (9)	0.071 (3)
C(2C)	0.0840 (17)	0.2732 (10)	0.0470 (10)	0.081 (3)