Table 1. *Fractional coordinates* ($\times 10^5$ *for* Pd *and* Cl *atoms;* $\times 10^4$ *for other atoms) and equivalent isotropic thermal parameters for the non-*H *atoms*

$$B_{eq} = \tfrac{4}{3}\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j$$

	x	y	z	B_{eq}(Å^2)
Pd	0	0	0	1·9
Cl	4194 (7)	5501 (2)	26550 (8)	3·0
N	692 (2)	−558 (1)	1972 (3)	2·4
C(1)	217 (2)	−1100 (1)	1737 (3)	2·1
C(2)	1114 (3)	−1439 (1)	3119 (4)	2·5
C(3)	616 (3)	−1993 (1)	2918 (4)	3·1
C(4)	−1019 (3)	−2049 (1)	3345 (4)	3·3
C(5)	−1912 (3)	−1700 (1)	1987 (4)	3·1
C(6)	−1421 (2)	−1146 (1)	2166 (4)	2·5

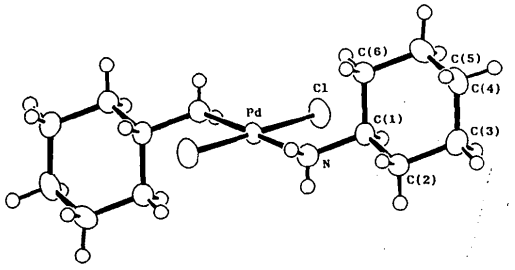

Fig. 1. *ORTEP* drawing of the complex molecule with thermal ellipsoids at the 50% probability level.

bases, and discussed the structures (dmso = dimethyl sulfoxide, tmso = 2,5-dihydrothiophene 1-oxide, chexa = cyclohexylamine, cpen = cyclopentylamine) (Oh & Chung, 1985; Oh & Mo, 1986).

Table 2. *Selected bond distances* (Å) *and angles* (°)

Pd–Cl	2·304 (1)	Cl–Pd–N	85·02 (6)
Pd–N	2·058 (2)	Pd–N–C(1)	121·6 (1)
N–C(1)	1·494 (3)	N–C(1)–C(2)	110·1 (2)
C(1)–C(2)	1·511 (3)	N–C(1)–C(6)	109·7 (2)
C(1)–C(6)	1·512 (3)	C(1)–C(2)–C(3)	110·5 (2)
C(2)–C(3)	1·528 (3)	C(2)–C(3)–C(4)	111·4 (2)
C(3)–C(4)	1·512 (4)	C(3)–C(4)–C(5)	110·6 (2)
C(4)–C(5)	1·516 (4)	C(4)–C(5)–C(6)	112·1 (2)
C(5)–C(6)	1·525 (3)	C(1)–C(6)–C(5)	110·2 (2)

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References

INAGAKI, K. & KIDANI, Y. (1978). *Bioinorg. Chem.* **9**, 333–343.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 71–98, 149. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

OH, S.-O. & CHUNG, D.-Y. (1985). *Daehan Hwahak Hwoejee*, **29**, 406–413.

OH, S.-O. & MO, S.-J. (1986). *Daehan Hwahak Hwoejee*, **30**, 320–326.

PRICE, J. H., WILLIAMSON, A. N., SCHRAMM, R. F. & WAYLAND, B. B. (1972). *Inorg. Chem.* **11**, 1280–1284.

ROSENBERG, B., VANCAMP, L., TROSKO, J. E. & MANSOUR, V. H. (1969). *Nature (London)*, **222**, 385–386.

SAKURAI, T. & KOBAYASHI, K. (1979). *Rikagaku Kenkyusho Hokoku*, **55**, 69–77.

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Structure of Monoclinic Chloro(*meso*-tetraphenylporphyrinato)iron(III)

BY W. ROBERT SCHEIDT* AND MICHAEL G. FINNEGAN

Department of Chemistry, University of Notre Dame, Notre Dame, Indiana 46556, *USA*

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Abstract. $[FeCl(C_{44}H_{28}N_4)]$, $M_r = 704{\cdot}03$, monoclinic, $P2_1/n$, $a = 10{\cdot}254$ (2), $b = 15{\cdot}969$ (3), $c = 20{\cdot}810$ (4) Å, $\beta = 90{\cdot}48$ (2)°, $V = 3407{\cdot}7$ Å^3, $Z = 4$, $D_x = 1{\cdot}37$ g cm^{-3}, Mo $K\alpha$, $\bar{\lambda} = 0{\cdot}71073$ Å, $\mu = 5{\cdot}6$ cm^{-1}, $F(000) = 363$, $T = 293$ K, $R = 0{\cdot}047$ for 3357 unique observed reflections. The iron(III) ion is coordinated to a chloride, Fe–Cl = 2·211 (1) Å, and four porphyrinato N atoms, average Fe–N = 2·070 (9) Å. The iron(III) is displaced 0·57 Å from the mean plane of the 24-atom core.

Experimental. Crystals of the title compound were obtained in the course of an investigation of the reaction of nitrite ion with (*meso*-tetraphenylporphyrinato)-iron(III) (Finnegan, 1988). A deep-purple crystal 0·4 × 0·25 × 0·10 mm mounted on a glass fiber. Intensities measured with a Nicolet $P\bar{1}$ diffractometer using θ–2θ scans at a variable rate of 2–12° in 2θ to a maximum value of 55°. 60 reflections used for measuring lattice parameters, $21{\cdot}2 < \theta < 36{\cdot}4°$. Range of hkl: $-11 \rightarrow 12$, $0 \rightarrow 19$, $0 \rightarrow 24$. 8420 reflections measured, 7581 unique, 3357 with $I > 3\sigma(I)$ considered observed. Merging R 0·035 for 199 duplicates. Four standard reflections, 2% intensity variation. No correction for absorption. Solved by Patterson and Fourier methods. Full-matrix least squares minimized $w(\Delta F)^2$ with a total of 451 variables. The H atoms were positioned according to idealized geometry (C–H

* To whom correspondence should be addressed.

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Table 1. *Fractional coordinates and equivalent isotropic thermal parameters*

The e.s.d.'s of the least significant digits are given in parentheses. $B_{eq} = \frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

	x	y	z	B_{eq}(Å^2)
Fe	0·36716 (6)	0·32539 (4)	−0·351436 (26)	2·95
Cl	0·17639 (11)	0·26438 (8)	−0·33596 (5)	4·55
N(1)	0·3616 (3)	0·36420 (20)	−0·44581 (15)	3·0
N(2)	0·4852 (3)	0·22799 (20)	−0·38271 (15)	3·1
N(3)	0·4598 (3)	0·31402 (20)	−0·26331 (4)	3·0
N(4)	0·3314 (3)	0·44932 (20)	−0·32694 (15)	3·0
C(*a*1)	0·3027 (4)	0·43607 (27)	−0·46895 (19)	3·3
C(*a*2)	0·3857 (4)	0·31516 (26)	−0·49877 (18)	3·1
C(*a*3)	0·4962 (4)	0·19801 (26)	−0·44463 (19)	3·4
C(*a*4)	0·5492 (4)	0·17026 (26)	−0·34401 (19)	3·5
C(*a*5)	0·5235 (4)	0·24271 (28)	−0·24060 (19)	3·4
C(*a*6)	0·4427 (4)	0·36507 (27)	−0·21061 (18)	3·1
C(*a*7)	0·3356 (4)	0·48369 (26)	−0·26589 (19)	3·3
C(*a*8)	0·2815 (4)	0·51138 (26)	−0·36617 (20)	3·5
C(*b*1)	0·2903 (4)	0·43042 (28)	−0·53741 (19)	3·7
C(*b*2)	0·3418 (5)	0·35744 (29)	−0·55602 (19)	3·8
C(*b*3)	0·5668 (5)	0·12059 (29)	−0·44450 (21)	4·0
C(*b*4)	0·5976 (5)	0·10341 (29)	−0·38306 (22)	4·2
C(*b*5)	0·5426 (4)	0·25036 (28)	−0·17288 (19)	3·8
C(*b*6)	0·4925 (4)	0·3237 (3)	−0·15406 (19)	3·7
C(*b*7)	0·2874 (5)	0·56762 (27)	−0·26757 (20)	3·7
C(*b*8)	0·2555 (5)	0·58481 (26)	−0·32928 (21)	4·0
C(*m*1)	0·4487 (4)	0·23761 (26)	−0·49964 (19)	3·2
C(*m*2)	0·5654 (4)	0·17622 (28)	−0·27768 (18)	3·3
C(*m*3)	0·3862 (4)	0·44450 (26)	−0·21087 (18)	3·2
C(*m*4)	0·2649 (4)	0·50513 (26)	−0·43288 (19)	3·3
C(1)	0·4725 (5)	0·20021 (26)	−0·56443 (19)	3·5
C(2)	0·5886 (5)	0·2129 (3)	−0·59448 (23)	4·7
C(3)	0·6057 (5)	0·1882 (4)	−0·65769 (25)	5·7
C(4)	0·5064 (6)	0·1494 (3)	−0·69009 (22)	4·8
C(5)	0·3910 (6)	0·1345 (3)	−0·66049 (24)	5·1
C(6)	0·3734 (5)	0·1601 (3)	−0·59788 (21)	4·6
C(7)	0·6425 (5)	0·1087 (3)	−0·24471 (20)	3·8
C(8)	0·5828 (5)	0·0387 (4)	−0·21992 (24)	5·1
C(9)	0·6561 (8)	−0·0248 (4)	−0·19080 (27)	6·1
C(10)	0·7863 (10)	−0·0172 (5)	−0·1875 (3)	6·0
C(11)	0·8471 (7)	0·0520 (6)	−0·2107 (3)	6·6
C(12)	0·7754 (6)	0·1151 (4)	−0·23964 (24)	5·6
C(13)	0·3884 (5)	0·49359 (26)	−0·14956 (20)	3·4
C(14)	0·2775 (5)	0·5116 (3)	−0·11663 (22)	4·5
C(15)	0·2810 (6)	0·5578 (3)	−0·06013 (23)	5·0
C(16)	0·3990 (7)	0·5880 (3)	−0·03807 (23)	4·8
C(17)	0·5101 (6)	0·5710 (3)	−0·07071 (24)	4·9
C(18)	0·5060 (5)	0·5238 (3)	−0·12612 (21)	4·3
C(19)	0·2044 (5)	0·57705 (27)	−0·46893 (20)	3·4
C(20)	0·2761 (4)	0·62110 (28)	−0·51346 (22)	4·0
C(21)	0·2187 (5)	0·6837 (3)	−0·55042 (21)	3·4
C(22)	0·0895 (6)	0·7023 (3)	−0·54301 (24)	4·6
C(23)	0·0187 (5)	0·6608 (3)	−0·49844 (27)	5·1
C(24)	0·0747 (5)	0·59741 (29)	−0·46127 (22)	4·5

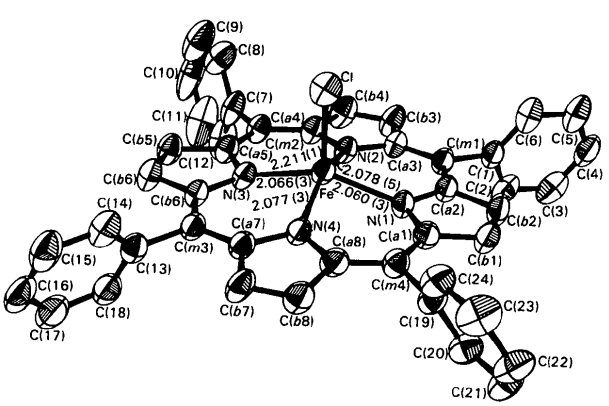

Fig. 1. *ORTEP* diagram (Johnson, 1976) and numbering scheme for all heavy atoms. 50% probability surfaces.

Table 2. *Bond lengths* (Å) *in the* [FeCl(*tpp*)] *molecule*

Fe–N(1)	2·060 (3)	C(*b*5)–C(*b*6)	1·338 (6)
Fe–N(2)	2·078 (3)	C(*b*7)–C(*b*8)	1·351 (5)
Fe–N(3)	2·066 (3)	C(*m*1)–C(1)	1·496 (5)
Fe–N(4)	2·077 (3)	C(*m*2)–C(7)	1·499 (6)
Fe–Cl	2·211 (1)	C(*m*3)–C(13)	1·498 (5)
N(1)–C(*a*1)	1·382 (5)	C(*m*4)–C(19)	1·503 (6)
N(1)–C(*a*2)	1·376 (5)	C(1)–C(2)	1·365 (6)
N(2)–C(*a*3)	1·380 (5)	C(2)–C(3)	1·386 (6)
N(2)–C(*a*4)	1·386 (5)	C(3)–C(4)	1·364 (7)
N(3)–C(*a*5)	1·394 (5)	C(4)–C(5)	1·360 (7)
N(3)–C(*a*6)	1·379 (5)	C(5)–C(6)	1·379 (6)
N(4)–C(*a*7)	1·384 (5)	C(1)–C(6)	1·384 (6)
N(4)–C(*a*8)	1·379 (5)	C(7)–C(8)	1·377 (7)
C(*a*1)–C(*b*1)	1·432 (5)	C(8)–C(9)	1·398 (7)
C(*a*2)–C(*b*2)	1·438 (5)	C(9)–C(10)	1·342 (10)
C(*a*3)–C(*b*3)	1·433 (6)	C(10)–C(11)	1·359 (11)
C(*a*4)–C(*b*4)	1·433 (6)	C(11)–C(12)	1·383 (8)
C(*a*5)–C(*b*5)	1·426 (5)	C(7)–C(12)	1·370 (7)
C(*a*6)–C(*a*6)	1·440 (5)	C(13)–C(14)	1·363 (6)
C(*a*7)–C(*b*7)	1·429 (6)	C(14)–C(15)	1·388 (6)
C(*a*8)–C(*b*8)	1·428 (6)	C(15)–C(16)	1·377 (7)
C(*a*1)–C(*m*4)	1·391 (5)	C(16)–C(17)	1·358 (7)
C(*a*2)–C(*m*1)	1·397 (5)	C(17)–C(18)	1·378 (6)
C(*a*3)–C(*m*1)	1·392 (5)	C(13)–C(18)	1·383 (6)
C(*a*4)–C(*m*2)	1·392 (5)	C(19)–C(20)	1·380 (6)
C(*a*5)–C(*m*2)	1·383 (5)	C(20)–C(21)	1·389 (6)
C(*a*6)–C(*m*3)	1·394 (6)	C(21)–C(22)	1·368 (6)
C(*a*7)–C(*m*3)	1·401 (5)	C(22)–C(23)	1·356 (7)
C(*a*8)–C(*m*4)	1·401 (5)	C(23)–C(24)	1·394 (6)
C(*b*1)–C(*b*2)	1·338 (6)	C(19)–C(24)	1·380 (6)
C(*b*3)–C(*b*4)	1·343 (6)		

= 0·95 Å) and isotropic B 1·3 times B of attached atom. All non-H atoms refined anisotropically. $R = 0·047$, $wR = 0·045$, $S = 1·45$. Weighting scheme $w = 1/[\sigma(F_o)]^2$. Final difference peak 0·37 e Å^{-3} near an N atom. Final $(\Delta/\sigma)_{max} = 0·02$. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). Data reduction carried out using the Blessing (1987) suite for data reduction, a locally modified version of *ALLS* (Lapp & Jacobson, 1979) used for structure refinement and *ORFFE* (Busing, Martin & Levy, 1964) for structural parameters with e.s.d.'s. Table 1 gives atomic coordinates and Tables 2 and 3 bond distances and angles.* Fig. 1 gives the atom numbering and perspective drawing of the molecule.

Related literature. The structures of a number of five-coordinate high-spin iron(III) porphyrinates have been reported and have been summarized by Scheidt & Gouterman (1983). [FeCl($C_{44}H_{28}N_4$)] is isomorphous with a number of five-coordinate metallotetraphenylporphyrinates, including (tetraphenylporphyrinato)-iron(III) derivatives having methoxide (Lecomte, Chadwick, Coppens & Stevens, 1983), bromide (Skelton & White, 1977), nitrate (Phillipi, Baenziger & Goff, 1981) and iodide (Hatano & Scheidt, 1979) as the axial ligand. A complete tabulation of cell constants for the ten members of this isomorphous set has been given by

* Lists of all structure factors, anisotropic thermal parameters and fixed hydrogen positions have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51745 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

N(1)FeN(2)	86·4 (1)	C(*b*4)C(*a*4)C(*m*2)	125·0 (4)
N(1)FeN(3)	151·8 (1)	N(3)C(*a*5)C(*b*5)	109·0 (4)
N(1)FeN(4)	86·8 (1)	N(3)C(*a*5)C(*m*2)	125·8 (4)
N(2)FeN(3)	87·0 (1)	C(*b*5)C(*a*5)C(*m*2)	125·2 (4)
N(2)FeN(4)	153·4 (1)	N(3)C(*a*6)C(*b*6)	109·4 (4)
N(3)FeN(4)	87·0 (1)	N(3)C(*a*6)C(*m*3)	126·2 (4)
N(1)FeCl	104·7 (1)	C(*b*6)C(*a*6)C(*m*3)	124·4 (4)
N(2)FeCl	103·5 (1)	N(4)C(*a*7)C(*b*7)	110·0 (4)
N(3)FeCl	103·4 (1)	N(4)C(*a*7)C(*m*3)	125·6 (4)
N(4)FeCl	103·0 (1)	C(*b*7)C(*a*7)C(*m*3)	124·4 (4)
FeN(1)C(*a*1)	126·2 (3)	N(4)C(*a*8)C(*b*8)	110·0 (3)
FeN(1)C(*a*2)	126·1 (3)	N(4)C(*a*8)C(*m*4)	125·2 (4)
FeN(2)C(*a*3)	127·2 (3)	C(*b*8)C(*a*8)C(*m*4)	124·7 (4)
FeN(2)C(*a*4)	126·2 (3)	C(*a*2)C(*m*1)C(1)	116·4 (4)
FeN(3)C(*a*5)	125·7 (3)	C(*a*3)C(*m*1)C(1)	120·1 (4)
FeN(3)C(*a*6)	126·4 (3)	C(*a*2)C(*m*1)C(*a*3)	123·4 (4)
FeN(4)C(*a*7)	126·8 (3)	C(*a*4)C(*m*2)C(7)	117·6 (4)
FeN(4)C(*a*8)	127·2 (3)	C(*a*5)C(*m*2)C(7)	117·5 (3)
C(*a*1)N(1)C(*a*2)	105·9 (3)	C(*a*4)C(*m*2)C(*a*5)	124·8 (4)
C(*a*3)N(2)C(*a*4)	105·6 (3)	C(*a*6)C(*m*3)C(13)	118·0 (4)
C(*a*5)N(3)C(*a*6)	106·0 (3)	C(*a*7)C(*m*3)C(13)	117·7 (4)
C(*a*7)N(4)C(*a*8)	105·5 (3)	C(*a*6)C(*m*3)C(*a*7)	124·1 (4)
N(1)C(*a*1)C(*b*1)	109·2 (4)	C(*a*1)C(*b*4)C(19)	116·9 (4)
N(1)C(*a*1)C(*m*4)	126·4 (4)	C(*a*8)C(*m*4)C(19)	119·1 (4)
C(*b*1)C(*a*1)C(*m*4)	124·3 (4)	C(*a*1)C(*m*4)C(*a*8)	124·0 (4)
N(1)C(*a*2)C(*b*2)	109·8 (4)	C(*a*1)C(*b*1)C(*b*2)	108·1 (4)
N(1)C(*a*2)C(*m*1)	126·9 (4)	C(*a*2)C(*b*2)C(*b*1)	106·9 (4)
C(*b*2)C(*a*2)C(*m*1)	123·2 (4)	C(*a*3)C(*b*3)C(*b*4)	107·0 (4)
N(2)C(*a*3)C(*b*3)	110·1 (4)	C(*a*4)C(*b*4)C(*b*3)	107·9 (4)
N(2)C(*a*3)C(*m*1)	125·4 (4)	C(*a*5)C(*b*5)C(*b*6)	108·3 (4)
C(*b*3)C(*a*3)C(*m*1)	124·5 (4)	C(*a*6)C(*b*6)C(*b*5)	107·3 (4)
N(2)C(*a*4)C(*b*4)	109·3 (3)	C(*a*7)C(*b*7)C(*b*8)	107·1 (4)
N(2)C(*a*4)C(*m*2)	125·6 (4)	C(*a*8)C(*b*8)C(*b*7)	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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References

BLESSING, R. H. (1987). *Cryst. Rev.* **1**, 3–58.

BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1964). *ORFFE*. Report ORNL-TM-306. Oak Ridge National Laboratory, Tennessee, USA.

FINNEGAN, M. G. (1988). PhD Thesis, Univ. of Notre Dame, Indiana, USA.

HATANO, K. & SCHEIDT, W. R. (1979). *Inorg. Chem.* **18**, 877–879.

HOARD, J. L., COHEN, G. H. & GLICK, M. D. (1967). *J. Am. Chem. Soc.* **89**, 1992–1996.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

LAPP, R. L. & JACOBSON, R. A. (1979). *ALLS. A Generalized Crystallographic Least Squares Program.* National Technical Information Services IS-4708 UC-4, Springfield, Virginia, USA.

LECOMTE, C., CHADWICK, D. L., COPPENS, P. & STEVENS, E. D. (1983). *Inorg. Chem.* **22**, 2982–2992.

PHILLIPI, M. A., BAENZIGER, N. & GOFF, H. M. (1981). *Inorg. Chem.* **20**, 3904–3911.

SCHEIDT, W. R. & GOUTERMAN, M. (1983). *Iron Porphyrins*, Part One, edited by A. B. P. LEVER & H. B. GRAY, pp. 89–139. Reading, MA: Addison–Wesley.

SCHEIDT, W. R. & LEE, Y. J. (1987). *Struct. Bonding (Berlin)*, **64**, 1–70.

SKELTON, B. W. & WHITE, A. H. (1977). *Aust. J. Chem.* **30**, 2655–2660.

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

BY J. PODLAHOVÁ, V. HABER, K. KNÍŽEK AND J. LOUB*

Department of Inorganic Chemistry, Charles University, Hlavova 8/2030, 128 40 *Praha* 2, *Czechoslovakia*

AND K. MALÝ

Institute of Physics, Czechoslovak Academy of Sciences, Na Slovance 2, 180 40 *Praha* 8, *Czechoslovakia*

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Abstract. $[Ni(C_9H_{18}N_3O)]I.H_2O$, $M_r = 387·87$, 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) Å^3, $Z = 4$, $D_m = 1·86$ (3), $D_x = 1·84$ Mg m^{-3}, λ(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 $[Ni(C_9H_{18}N_3O)]^+$ complex cations, iodide anions and molecules of water. The coordination of Ni is almost square-planar with the ligand $C_9H_{18}N_3O^-$ bonded to Ni through one O and three N

*To whom correspondence should be addressed.

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