Related literature. For molecular structure of parent carbonyl, $W(CO)_6$, by electron diffraction see Arnesen & Seip (1966). For another $M(CO)_3(NCCH_3)_3$ compound (M = Re, BF_4^- salt) see Chan, Isaacs & Graham (1977).

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[*N*,*N*′-Bis(2-aminoethyl)-1,4-diazacycloheptane-*N*,*N*′,*N*″,*N*‴]nickel(II) Diperchlorate

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Abstract. $[Ni(C_9H_{22}N_4)](ClO_4)_2$, $M_r = 443{\cdot}9$, orthorhombic, $Pmn2_1$, $a = 9{\cdot}463\ (2)$, $b = 7{\cdot}489\ (1)$, $c = 11{\cdot}750\ (2)$ Å, $V = 833\ (2)$ Å^3, $Z = 2$, $D_m = 1{\cdot}80$, $D_x = 1{\cdot}770$ Mg m^{-3}, λ(Mo $K\bar{\alpha}$) = 0·7107 Å, $\mu = 1{\cdot}48$ mm^{-1}, $F(000) = 456$, $T = 295\ (2)$ K, $R = 0{\cdot}055$ for 1517 reflections with $I \geq 2{\cdot}5\sigma(I)$. The complex cation is situated about a crystallographic mirror plane and the NiN_4 chromophore is nearly planar with the Ni atom 0·03 Å above the four N atoms [in the direction of the C(5) atom]. In the solid state the 1,4-diazacycloheptane ring adopts a boat conformation so that the apical C(5) atom does not lie directly above the Ni atom.

Experimental. Orange crystals of $[Ni(C_9H_{22}N_4)](ClO_4)_2$ prepared according to the literature procedure (Phillip, 1969). Enraf–Nonius CAD-4*F* diffractometer controlled by a PDP8/A computer, graphite-monochromated Mo $K\bar{\alpha}$ radiation; ω:2θ scan technique. Cell parameters, on crystal 0·25 × 0·25 × 0·75 mm, from least-squares procedure (De Boer & Duisenberg, 1984) on 25 reflections ($10 \leq \theta \leq 17°$). Analytical absorption correction: max./min. transmission factors 0·7177, 0·5757 (Sheldrick, 1976). Total of 3595 reflections ($1 \leq \theta \leq 25°$) measured in the range $-11 \leq h \leq 1$, $-8 \leq k \leq 8$, $-13 \leq l \leq 13$. No significant variation in the intensities of three standards (435, 514, 422) monitored every 3600 s. 1552 unique reflections ($R_{int} = 0{\cdot}079$), 1517 satisfied $I \geq 2{\cdot}5\sigma(I)$. Structure solved from Patterson method, full-matrix least-squares refinement of 361 parameters based on F (Sheldrick, 1976). Anisotropic thermal parameters for non-H atoms and H atoms located from difference map and refined. At convergence $R = 0{\cdot}055$, $wR = 0{\cdot}058$, $w = [\sigma^2(F) + 0{\cdot}0046F^2]^{-1}$, $S = 0{\cdot}29$, $(\Delta/\sigma)_{max} \leq 0{\cdot}42$, $\Delta\rho_{max} = 1{\cdot}29$, $\Delta\rho_{min} = -1{\cdot}66$ e Å^{-3}; no extinction correction. Scattering factors for H, C, Cl, N and O given in *SHELX76* (Sheldrick, 1976) and those for Ni^{2+} corrected for f' and f'' (Hamilton & Ibers, 1974). University of Adelaide's VAX11/785 computer system. Atomic parameters are given in Table 1, bond distances and angles in Table 2* and the numbering scheme is shown in Fig. 1.

Related literature. It has been suggested in an earlier study (Phillip, 1969) that the 1,4-diazacycloheptane ring in $[Ni(C_9H_{22}N_4)](ClO_4)_2$ adopted a chair conformation in which the apical C(5) atom was situated above the Ni atom; as a result further coordination by additional ligands was prevented due to possible steric hindrance. The solid-state study reveals that the seven-membered ring adopts a 'boat' conformation and also the presence of a weak interionic interaction of 2·89 (1) Å between the Ni atom and O(2), derived from one of the perchlorate anions, compared with the sum of the van der Waals radii for these atoms of 3·10 Å (Bondi, 1964).

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, interatomic parameters involving the perchlorate anions and mean-plane data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43737 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Fractional atomic coordinates and* B_{eq}(Å^2) *values*

$B_{eq} = 8\pi(U_{11} + U_{22} + U_{33})/3.$

	x	y	z	B_{eq}
Ni	0·0	0·28546 (7)	0·0	1·99
N(1)	0·1329 (5)	0·2684 (5)	0·1198 (4)	3·04
N(2)	0·1514 (5)	0·3077 (5)	−0·1056 (4)	3·16
C(1)	0·0832 (5)	0·3926 (6)	0·2099 (4)	3·61
C(2)	0·2747 (5)	0·3214 (8)	0·0728 (5)	4·49
C(3)	0·2819 (5)	0·2510 (8)	−0·0488 (6)	4·38
C(4)	0·1354 (5)	0·0778 (6)	0·1596 (4)	3·82
C(5)	0·0	0·0301 (8)	0·2215 (6)	4·43
Cl(1)	0·0	−0·2311 (2)	−0·1034 (2)	3·42
O(1)	0·0	−0·2996 (7)	0·0085 (9)	9·92
O(2)	0·0	−0·0526 (9)	−0·1125 (9)	13·83
O(3)	0·0969 (11)	−0·3111 (10)	−0·1677 (9)	22·48
Cl(2)	0·0	0·2246 (2)	0·5763 (2)	3·39
O(4)	0·0	0·3625 (9)	0·4903 (6)	5·81
O(5)	0·0	0·0621 (8)	0·5263 (6)	7·48
O(6)	0·1261 (6)	0·2439 (7)	0·6440 (4)	5·79

Table 2. *Interatomic distances* (Å) *and angles* (°)

Ni–N(1)	1·892 (5)	N(2)–C(3)	1·467 (7)
Ni–N(2)	1·902 (4)	C(2)–C(3)	1·524 (9)
N(1)–C(1)	1·486 (6)	N(1)–C(4)	1·502 (5)
C(1)–C(1′)*	1·575 (9)	C(4)–C(5)	1·517 (7)
N(1)–C(2)	1·504 (7)		
N(1)–Ni–N(2)	89·5 (2)	C(1)–N(1)–C(4)	112·2 (4)
N(1)–Ni–N(1′)	83·4 (3)	C(2)–N(1)–C(4)	110·5 (4)
Ni–N(1)–C(1)	106·1 (3)	N(1)–C(1)–C(1′)	108·5 (6)
Ni–N(1)–C(2)	107·6 (3)	N(1)–C(2)–C(3)	107·0 (4)
Ni–N(1)–C(4)	107·8 (3)	N(1)–C(4)–C(5)	111·1 (4)
Ni–N(2)–C(3)	108·2 (4)	N(2)–C(3)–C(2)	106·8 (5)
C(1)–N(1)–C(2)	112·3 (4)	C(4)–C(5)–C(4′)	115·4 (6)

* Primed atoms are related by the mirror plane.

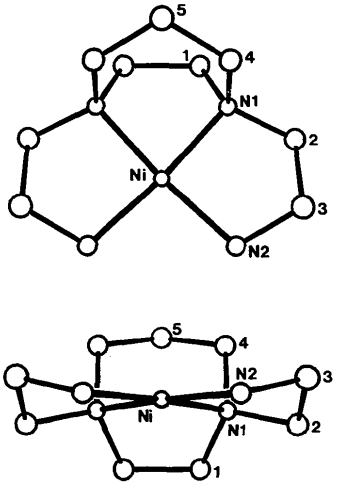

Fig. 1. A plan and a side-on view of the cation in $[Ni(C_9H_{22}N_4)](ClO_4)_2$ highlighting the disposition of the 1,4-diazacycloheptane ring; the numbering scheme employed is also shown (Johnson, 1971). Atoms otherwise not indicated are C atoms.

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Structure of an η^2-Phosphaalkene Nickel Complex

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Abstract. 2,2′-Bipyridyl[1-(2,6-dimethylphenyl)-2,2-diphenyl-1-phosphamethylene]nickel(0)–tetrahydrofuran solvate, $[Ni(C_{10}H_8N_2)(C_{21}H_{19}P)].C_4H_8O$, M_r = 589·37, monoclinic, $P2_1/n$, a = 11·271 (1), b = 16·926 (1), c = 16·058 (2) Å, β = 96·21 (1)°, V = 3045·5 (5) Å^3, Z = 4, $F(000)$ = 1240, D_x = 1·285 g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, μ = 15·96 cm^{-1}, T = 298 K, R = 0·0638 for 3204 reflections with $I > 2{\cdot}5\sigma(I)$. The geometry at nickel is close to square-planar and the phosphaalkene is η^2-bonded to the nickel atom.

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