We thank Dr Arped Phillip for the crystals and the Australian Research Grants Scheme for financial support.

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.

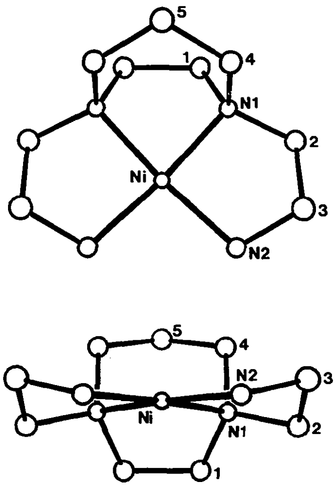

Fig. 1. A plan and a side-on view of the cation in $[Ni(C_9H_{22}N_4)](ClO_4)_2$ highlighting the disposition of the 1,4-diazacycloheptane ring; the numbering scheme employed is also shown (Johnson, 1971). Atoms otherwise not indicated are C atoms.

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Acta Cryst. (1987). C**43**, 1216–1218

Structure of an η^2-Phosphaalkene Nickel Complex

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(*Received* 7 *January* 1987; *accepted* 28 *January* 1987)

Abstract. 2,2'-Bipyridyl[1-(2,6-dimethylphenyl)-2,2-diphenyl-1-phosphamethylene]nickel(0)–tetrahydrofuran solvate, $[Ni(C_{10}H_8N_2)(C_{21}H_{19}P)].C_4H_8O$, $M_r = 589{\cdot}37$, monoclinic, $P2_1/n$, $a = 11{\cdot}271\,(1)$, $b = 16{\cdot}926\,(1)$, $c = 16{\cdot}058\,(2)$ Å, $\beta = 96{\cdot}21\,(1)°$, $V = 3045{\cdot}5\,(5)$ Å^3, $Z = 4$, $F(000) = 1240$, $D_x = 1{\cdot}285$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $\mu = 15{\cdot}96$ cm^{-1}, $T = 298$ K, $R = 0{\cdot}0638$ 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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Experimental. Crystals were prepared as described by van der Knaap *et al.* (1983). A dark green crystal (0·62 × 0·62 × 0·75 mm) was sealed under nitrogen in a Lindemann-glass capillary. Diffraction data were collected on an Enraf–Nonius CAD-4F diffractometer using Ni-filtered Cu $K\alpha$ radiation. Lattice parameters and their e.s.d.'s were derived from the setting angles of ten reflections ($2\theta < 65°$) and confirmed with rotation photographs about the three axes. The space group was determined as $P2_1/n$ from the observed systematic absences. Two reference reflections ($1\bar{1}0$ and $\bar{1}\bar{1}0$) measured every hour of X-ray exposure time showed variations less than 1% and no decay over the 96 h of X-ray exposure time. The intensity data of 6041 unique reflections [$2{\cdot}6 < \theta < 70°$; $0 \le h \le 13$,

Table 1. *Fractional atomic coordinates with equivalent isotropic thermal parameters for the non-*H *atoms (e.s.d.'s in parentheses)*

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$(Å^2)
Ni	0·64345 (9)	0·31833 (6)	0·47266 (6)	0·0543 (3)
P(1)	0·6408 (1)	0·19232 (9)	0·4996 (1)	0·0537 (5)
N(1)	0·5053 (4)	0·3562 (3)	0·4013 (3)	0·057 (1)
N(2)	0·6903 (4)	0·4293 (3)	0·4728 (3)	0·055 (1)
C(1)	0·4120 (5)	0·3127 (4)	0·3687 (4)	0·065 (1)
C(2)	0·3146 (6)	0·3458 (4)	0·3215 (4)	0·072 (1)
C(3)	0·3132 (6)	0·4256 (4)	0·3084 (4)	0·074 (2)
C(4)	0·4072 (6)	0·4716 (4)	0·3421 (4)	0·067 (1)
C(5)	0·5018 (5)	0·4345 (3)	0·3885 (4)	0·054 (1)
C(6)	0·6080 (5)	0·4771 (3)	0·4278 (4)	0·056 (1)
C(7)	0·6270 (6)	0·5574 (4)	0·4196 (4)	0·068 (1)
C(8)	0·7322 (6)	0·5896 (4)	0·4569 (4)	0·079 (2)
C(9)	0·8168 (6)	0·5414 (4)	0·4999 (4)	0·078 (2)
C(10)	0·7904 (6)	0·4627 (4)	0·5082 (4)	0·068 (1)
C(11)	0·7504 (5)	0·2582 (3)	0·5564 (4)	0·052 (1)
C(12)	0·7190 (5)	0·2902 (3)	0·6374 (3)	0·050 (1)
C(13)	0·6094 (6)	0·2780 (4)	0·6666 (4)	0·061 (1)
C(14)	0·5786 (6)	0·3089 (4)	0·7406 (4)	0·069 (1)
C(15)	0·6611 (7)	0·3550 (4)	0·7906 (4)	0·075 (2)
C(16)	0·7708 (7)	0·3685 (4)	0·7634 (4)	0·069 (1)
C(17)	0·8012 (6)	0·3371 (3)	0·6883 (4)	0·063 (1)
C(18)	0·8790 (5)	0·2481 (3)	0·5445 (4)	0·052 (1)
C(19)	0·9566 (6)	0·2100 (4)	0·6072 (4)	0·067 (1)
C(20)	1·0736 (6)	0·1951 (4)	0·5943 (5)	0·078 (2)
C(21)	1·1178 (6)	0·2165 (4)	0·5210 (5)	0·079 (2)
C(22)	1·0430 (6)	0·2549 (4)	0·4601 (5)	0·080 (2)
C(23)	0·9251 (5)	0·2706 (4)	0·4731 (4)	0·061 (1)
C(24)	0·7328 (5)	0·1345 (3)	0·4334 (4)	0·053 (1)
C(25)	0·7965 (5)	0·0685 (4)	0·4711 (4)	0·063 (1)
C(26)	0·8666 (6)	0·0244 (4)	0·4250 (5)	0·074 (2)
C(27)	0·8776 (6)	0·0397 (4)	0·3425 (5)	0·086 (2)
C(28)	0·8118 (6)	0·1008 (4)	0·3040 (4)	0·075 (2)
C(29)	0·7395 (5)	0·1477 (4)	0·3489 (4)	0·062 (1)
C(30)	0·7845 (7)	0·0441 (4)	0·5603 (4)	0·084 (2)
C(31)	0·6663 (7)	0·2126 (4)	0·3014 (4)	0·086 (2)
Tetrahydrofuran solvate				
O(1)	0·0127 (9)	0·4863 (6)	0·2477 (8)	0·229 (2)
C(32)	−0·115 (1)	0·3977 (8)	0·1765 (9)	0·187 (2)
C(34)	−0·021 (1)	0·4541 (9)	0·1695 (9)	0·191 (2)
C(35)	−0·129 (1)	0·3996 (9)	0·2596 (9)	0·201 (2)
C(36)	−0·060 (1)	0·455 (1)	0·3021 (9)	0·212 (2)

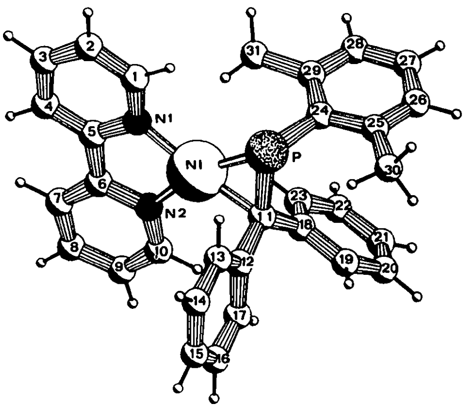

Fig. 1. View of the title compound, showing the atom-numbering scheme.

Table 2. *Bond lengths* (Å), *bond angles* (°) *and selected torsion angles* (°) *involving non-hydrogen atoms*

Ni–P(1)	2·177 (2)	C(12)–C(13)	1·384 (8)
Ni–N(1)	1·940 (5)	C(12)–C(17)	1·412 (8)
Ni–N(2)	1·951 (5)	C(13)–C(14)	1·377 (9)
Ni–C(11)	1·987 (6)	C(14)–C(15)	1·40 (1)
P(1)–C(11)	1·832 (6)	C(15)–C(16)	1·37 (1)
P(1)–C(24)	1·845 (6)	C(16)–C(17)	1·394 (9)
N(1)–C(1)	1·343 (8)	C(18)–C(19)	1·415 (8)
N(1)–C(5)	1·341 (7)	C(18)–C(23)	1·364 (8)
N(2)–C(6)	1·376 (8)	C(19)–C(20)	1·380 (9)
N(2)–C(10)	1·333 (8)	C(20)–C(21)	1·37 (1)
C(1)–C(2)	1·383 (9)	C(21)–C(22)	1·38 (1)
C(2)–C(3)	1·37 (1)	C(22)–C(23)	1·392 (9)
C(3)–C(4)	1·38 (1)	C(24)–C(25)	1·427 (8)
C(4)–C(5)	1·383 (9)	C(24)–C(29)	1·385 (8)
C(5)–C(6)	1·480 (8)	C(25)–C(26)	1·362 (9)
C(6)–C(7)	1·384 (8)	C(25)–C(30)	1·51 (1)
C(7)–C(8)	1·38 (1)	C(26)–C(27)	1·37 (1)
C(8)–C(9)	1·38 (1)	C(27)–C(28)	1·38 (1)
C(9)–C(10)	1·374 (9)	C(28)–C(29)	1·393 (9)
C(11)–C(12)	1·488 (8)	C(29)–C(31)	1·53 (1)
C(11)–C(18)	1·491 (8)		

P(1)–Ni–N(1)	114·4 (1)	P(1)–C(11)–C(12)	116·4 (4)
P(1)–Ni–N(2)	162·3 (2)	P(1)–C(11)–C(18)	118·7 (4)
P(1)–Ni–C(11)	52·0 (2)	C(12)–C(11)–C(18)	118·7 (5)
N(1)--Ni–N(2)	83·2 (2)	C(11)–C(12)–C(13)	123·2 (5)
N(1)–Ni–C(11)	163·5 (2)	C(11)–C(12)–C(17)	120·5 (5)
N(2)–Ni–C(11)	110·4 (2)	C(13)–C(12)–C(17)	116·2 (5)
Ni–P(1)–C(11)	58·7 (2)	C(12)–C(13)–C(14)	123·6 (6)
Ni–P(1)–C(24)	112·6 (2)	C(13)–C(14)–C(15)	119·6 (6)
C(11)–P(1)–C(24)	102·6 (3)	C(14)–C(15)–C(16)	118·4 (6)
Ni–N(1)–C(1)	126·5 (4)	C(15)–C(16)–C(17)	121·7 (6)
Ni–N(1)–C(5)	115·2 (4)	C(12)–C(17)–C(16)	120·5 (6)
C(1)–N(1)–C(5)	118·2 (5)	C(11)–C(18)–C(19)	119·7 (5)
Ni–N(2)–C(6)	113·5 (4)	C(11)–C(18)–C(23)	122·6 (5)
Ni–N(2)–C(10)	128·7 (4)	C(19)–C(18)–C(23)	117·6 (5)
C(6)–N(2)–C(10)	117·8 (5)	C(18)–C(19)–C(20)	120·2 (6)
N(1)–C(1)–C(2)	122·3 (6)	C(19)–C(20)–C(21)	121·4 (7)
C(1)–C(2)–C(3)	118·7 (6)	C(20)–C(21)–C(22)	118·7 (7)
C(2)–C(3)–C(4)	120·2 (6)	C(21)–C(22)–C(23)	120·2 (7)
C(3)–C(4)–C(5)	118·0 (6)	C(18)–C(23)–C(22)	121·9 (6)
N(1)–C(5)–C(4)	122·7 (5)	P(1)–C(24)–C(25)	117·3 (4)
N(1)–C(5)–C(6)	114·0 (5)	P(1)–C(24)–C(29)	125·0 (5)
C(4)–C(5)–C(6)	123·3 (5)	C(25)–C(24)–C(29)	117·6 (5)
N(2)–C(6)–C(5)	113·9 (5)	C(24)–C(25)–C(26)	119·5 (6)
N(2)–C(6)–C(7)	121·7 (6)	C(24)–C(25)–C(30)	121·6 (6)
C(5)–C(6)–C(7)	124·4 (5)	C(26)–C(25)–C(30)	118·8 (6)
C(6)–C(7)–C(8)	118·7 (6)	C(25)–C(26)–C(27)	122·8 (7)
C(7)–C(8)–C(9)	119·8 (6)	C(26)–C(27)–C(28)	118·4 (7)
C(8)–C(9)–C(10)	118·4 (7)	C(27)–C(28)–C(29)	120·8 (7)
N(2)–C(10)–C(9)	123·5 (6)	C(24)–C(29)–C(28)	120·8 (6)
Ni–C(11)–P(1)	69·4 (2)	C(24)–C(29)–C(31)	121·4 (5)
Ni–C(11)–C(12)	102·7 (4)	C(28)–C(29)–C(31)	117·8 (6)
Ni–C(11)–C(18)	120·0 (4)		

C(11)–P–C(24)–C(25)	−101·4 (6)	C(18)–C(11)–P–C(24)	−4·8 (5)
C(23)–C(18)–C(11)–P	73·6 (7)	C(13)–C(12)–C(11)–P	−5·7 (8)

Tetrahydrofuran solvate

O(1)–C(34)	1·39 (2)	C(32)–C(35)	1·36 (2)
O(1)–C(36)	1·37 (2)	C(35)–C(36)	1·36 (2)
C(32)–C(34)	1·44 (2)		

C(34)–O(1)–C(36)	108 (1)	C(32)–C(35)–C(36)	113 (1)
C(34)–C(32)–C(35)	103 (1)	O(1)–C(36)–C(35)	107 (1)
O(1)–C(34)–C(32)	109 (1)		

$0 \leq k \leq 20$, $-19 \leq l \leq 19$; $\omega/2\theta$ scan mode with $\Delta\omega = (1{\cdot}0 + 0{\cdot}14\tan\theta)°$] were corrected for Lp. No correction for absorption was applied in view of a < 6% intensity variation of the 360° ψ scan of the close-to-axial reflection $2\bar{4}0$. The structure was solved with standard heavy-atom methods and refined with blocked full-matrix least-squares techniques on F (*SHELX76*; Sheldrick, 1976). H atoms were included on calculated positions (C—H = 1·08 Å) with one overall isotropic thermal parameter [$U = 0{\cdot}137$ (3) Å^2]. Convergence was reached at $R = 0{\cdot}0638$, $wR = 0{\cdot}0644$ [$w = 1$, 3204 reflections with $I > 2{\cdot}5\sigma(I)$, 369 variables, $\langle\Delta/\sigma\rangle = 0{\cdot}1$, $S = 1{\cdot}88$]. A final difference synthesis showed no residual density higher than 0·44 e Å^{-3}. Scattering factors from Cromer & Mann (1969); anomalous-dispersion factors from Cromer & Liberman (1970). Geometrical calculations and illustrations by programs of the *EUCLID* package (Spek, 1982). Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.* Fig. 1 shows the atom-numbering scheme and geometry of the molecule. Bond lengths and angles and torsion angles are listed in Table 2.

Related literature. For the preparation of the compound and a discussion of the results see van der Knaap *et al.* (1983). For a related η^2-phosphaalkene nickel complex see Cowley, Jones, Stewart, Stuart, Atwood, Hunting & Zhang (1983).

Part of this work was supported by the Netherlands Foundation for Chemical Research (SON) with financial aid from the Netherlands Organization for the Advancement of Pure Research (ZWO).

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* Lists of anisotropic thermal parameters, H-atom positions, complete lists of bond distances and angles and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43762 (29 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1987). C**43**, 1218–1219

Structure of Vanadyl Diformate Monohydrate

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(*Received* 7 *July* 1986; *accepted* 13 *January* 1987)

Abstract. Aquadiformatooxovanadium(IV), $[VO(HCOO)_2(H_2O)]$, $M_r = 175{\cdot}0$, orthorhombic, *Pcca*, $a = 8{\cdot}395$ (2), $b = 7{\cdot}433$ (1), $c = 8{\cdot}510$ (1) Å, $V = 531{\cdot}0$ Å^3, $Z = 4$, $D_m = 2{\cdot}2$, $D_x = 2{\cdot}19$ g cm^{-3}, $\lambda(\text{Mo } K\overline{\alpha}) = 0{\cdot}71073$ Å, $\mu = 19{\cdot}3$ cm^{-1}, $F(000) = 348$, room temperature, $R = 0{\cdot}047$ for 680 unique observed reflections. Distorted $VO(H_2O)(HCOO)_{4/2}$ octahedra with the V atom and *trans* oxo and aqua O atoms on the twofold rotation axes parallel to [010] are linked into polymeric layers perpendicular to that direction by the bidentate formato ligands. The layers are connected in pairs by weak, bifurcated hydrogen bonds between aqua and formato O atoms.

Experimental. Blue crystals obtained by hydrothermal synthesis from vanadium(V) oxide and formic acid at 423 K. Crystal 0·2 × 0·2 × 0·1 mm, D_m by flotation, Syntex $P2_1$ diffractometer with graphite monochromator. Lattice constants from setting angles of 15 reflections with $25 < 2\theta < 27°$, intensities by ω scan with scan rates of 0·7–58·6° min^{-1}. Three standard reflections every 100 reflections; only small, random variations. No correction for absorption. 1171 unique reflections measured with $(\sin\theta)/\lambda$ up to 0·807 Å^{-1} and $0 \leq h \leq 13$, $0 \leq k \leq 11$, $0 \leq l \leq 13$; 680 observed reflections with $I > 1{\cdot}96\sigma(I)$.

Heavy-atom method, full-matrix least-squares refinement based on F magnitudes; observed reflections only, weighted according to $w = [\sigma^2(F) + (0{\cdot}025\,|F_o|)^2]^{-1}$. H atoms located by the difference Fourier method and included in the final refinement of 51 parameters (one scale factor and all variable coordinates and thermal

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