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Dimeric Nickel(II) Carboxylates and a Silanecarboxylate: $[Ni(Me_3CCOO)_2(2,5\text{-lutidine})]_2$, $[Ni(MePh_2CCOO)_2(quinoline)]_2 \cdot 2CHCl_3$, $[Ni(Me_2PhCCOO)_2(quinoline)]_2$, $[Ni(Me_3CCOO)_2(2\text{-ethylpyridine})]_2$, $[Ni(Me_3CCOO)_2(2\text{-picoline})]_2$ and $[Ni(MePh_2SiCOO)_2(Ph_3P)]_2$

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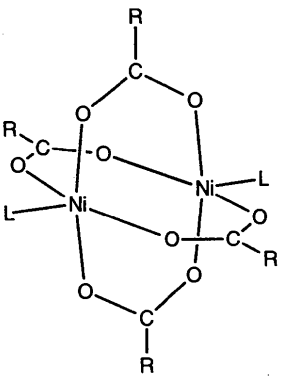
Abstract

The structures of five dimeric Ni^{II} carboxylates and one dimeric Ni^{II} silanecarboxylate have been determined: (I), tetrakis(μ-2,2-dimethylpropanoato-*O*,*O*′)-bis(2,5-lutidine)dinickel, $[Ni(C_5H_9O_2)_2(C_7H_9N)]_2$; (II), tetrakis($\mu$-2,2-diphenylpropanoato-*O*,*O*′)-diquinolinedinickel bis(trichloromethane), $[Ni(C_{15}H_{13}O_2)_2(C_9H_7N)]_2.2CHCl_3$; (III), tetrakis($\mu$-2-methyl-2-phenylpropanoato-*O*,*O*′)-diquinolinedinickel, $[Ni(C_{10}H_{11}O_2)_2(C_9H_7N)]_2$; (IV), tetrakis($\mu$-2,2-dimethylpropanoato-*O*,*O*′)-bis(2-ethylpyridine)dinickel, $[Ni(C_5H_9O_2)_2(C_7H_9N)]_2$; (V), tetrakis-($\mu$-2,2-dimethylpropanoato-*O*,*O*′)-bis(2-picoline)dinickel, $[Ni(C_5H_9O_2)_2(C_6H_7N)]_2$; (VI), tetrakis ($\mu$-methyl diphenylsilanecarboxylato-*O*,*O*′)-bis(triphenylphosphine)dinickel, $[Ni(C_{14}H_{13}O_2Si)_2(C_{18}H_{15}P)]_2$. The Ni atoms are arranged in a square-pyramidal geometry and the $Ni_2(COO)_4$ moiety forms a slightly distorted cage structure. The Ni···Ni distances are in the range 2.7079(8)–2.765(1) Å.

Comment

Dimeric Cu^{II} carboxylates show antiferromagnetism as a result of spin superexchange between the two metal centers through the carboxylate bridges; the magnitude of the spin-exchange interaction depends on the electric structure of the carboxylate bridges (Yamanaka *et al.*, 1991). The cryomagnetic behavior of the Ni^{II} dimers resembles that of the corresponding Cu^{II} dimers (Bencini, Benelli, Gatteschi & Zanchini, 1980; Hirashima *et al.*, 1990) although the nature of the demagnetization has yet to be clarified. Structural data have been reported for the Ni^{II} carboxylate dimers, $[Ni(Me_3CCOO)_2L]_2$ with L = 2-methylquinoline (Kirillova *et al.*, 1980) and L = 2,4-lutidine (Hirashima *et al.*, 1990). In the present paper, the structures of six Ni^{II} dimers are reported. Magnetic susceptibility measurements indicate that the demagnetization in $[Ni(Me_3CCOO)_2(2,5\text{-lutidine})]_2$ (I) is smaller than in the 2,4-lutidine adduct (Hirashima *et al.*, 1990). However, no notable differences were found between the dimensions of the $Ni_2(COO)_4$ cages and further investigations are required to establish the relationship between structure and magnetism. Although silanecarboxylate bridges significantly enhance the antiferromagnetism of the Cu^{II} dimers (Uekusa *et al.*, 1990), their effect in the Ni^{II} dimers is not currently well defined.

The complexes (I)–(VI) all have a crystallographic center of symmetry. The Ni atom has a slightly distorted square-pyramidal environment with four O atoms of the bridging carboxylates in the basal plane and an N or P atom in the axial position. The two Ni atoms in the complexes are shifted in the opposite direction from the O_4 basal plane by 0.243(1)–0.274(1) Å. The cage of the



	L	*R*
(I)	2,5-Lutidine	Me_3C
(II)	Quinoline	$MePh_2C$
(III)	Quinoline	Me_2PhC
(IV)	2-Ethylpyridine	Me_3C
(V)	2-Picoline	Me_3C
(VI)	Ph_3P	$MePh_2Si$

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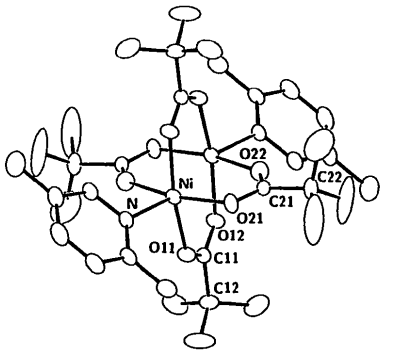
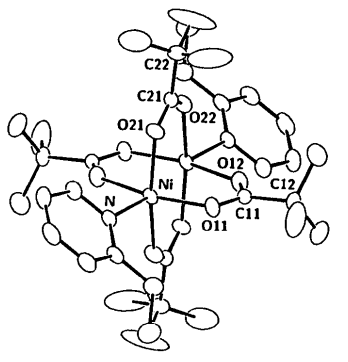
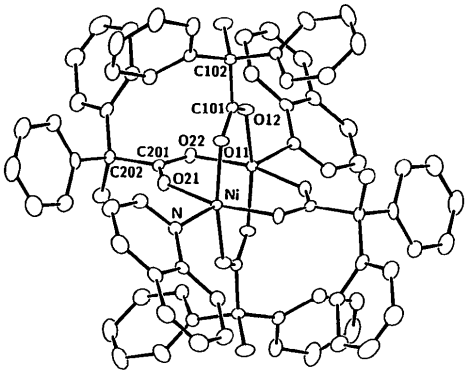


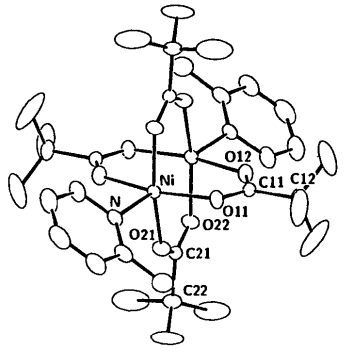
(I)



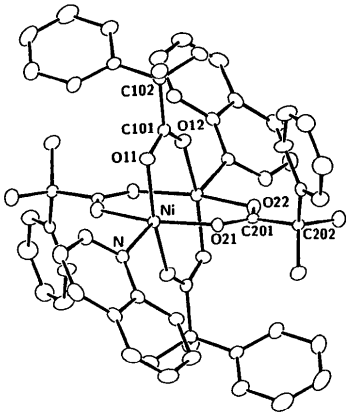
(IV)



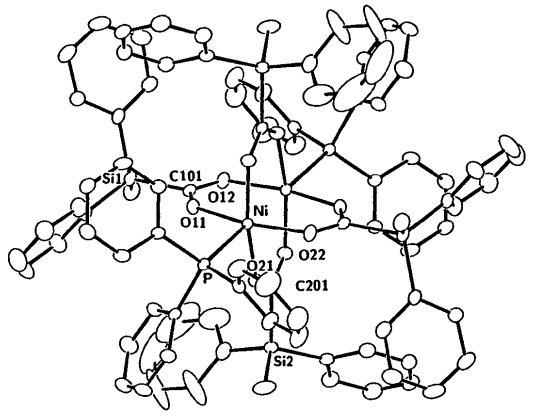
(II)



(V)



(III)



(VI)

Fig. 1. Views of the molecules with thermal ellipsoids scaled at 20% probability level. The H atoms are omitted for clarity.

$Ni_2(COO)_4$ moiety is deformed in one of the carboxylate planes. A comparison of (III) with the corresponding Cu^{II} complex, $[Cu(Me_2PhCCOO)_2(quinoline)]_2$ (Uekusa *et al.*, 1990), shows that the Ni—N bond is 0.180(3) Å shorter than Cu—N, suggesting a stronger bond, and that on average the Ni—O bonds are longer than Cu—O by 0.040(2) Å.

Experimental

Compound (I)

Crystal data

$[Ni(C_5H_9O_2)_2(C_7H_9N)]_2$
M_r = 736.2
Monoclinic
$C2/c$

Density measured by flotation in KI solution
Mo $K\alpha$ radiation
λ = 0.71073 Å

a = 20.520 (2) Å
b = 10.647 (1) Å
c = 18.260 (2) Å
β = 91.015 (8)°
V = 3988.8 (7) Å^3
Z = 4
D_x = 1.226 Mg m^{-3}
D_m = 1.22 (2) Mg m^{-3}

Cell parameters from 49 reflections
θ = 10-15°
μ = 0.9913 mm^{-1}
T = 299 K
Prism
0.37 × 0.35 × 0.25 mm
Dark green

Data collection

Rigaku AFC-5 four-circle diffractometer
θ-2θ scans
Absorption correction: by integration from crystal shape
T_{min} = 0.71, T_{max} = 0.79
3930 measured reflections
3803 independent reflections
2123 observed reflections [$|F_o|>3\sigma(|F_o|)$]

R_{int} = 0.016
θ_{max} = 27.5°
$h = -26 \rightarrow 26$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 23$
5 standard reflections monitored every 100 reflections
intensity variation: 1.8%

Refinement

Refinement on F
Final R = 0.043
wR = 0.031
S = 2.290
2123 reflections
316 parameters

All H-atom parameters refined
Calculated weights, $w=1/\sigma$
$(\Delta/\sigma)_{max}$ = 0.056
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *for* (I)

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

	x	y	z	U_{eq}
Ni	0.24203 (3)	0.62747 (5)	0.51891 (3)	0.0487 (2)
O11	0.1870 (2)	0.6998 (3)	0.5976 (2)	0.062 (1)
O12	0.2972 (2)	0.5989 (3)	0.4304 (2)	0.061 (1)
O21	0.1626 (1)	0.5965 (3)	0.4550 (2)	0.062 (1)
O22	0.3222 (2)	0.7027 (3)	0.5709 (2)	0.065 (1)
N	0.2609 (2)	0.4535 (3)	0.5600 (2)	0.048 (1)
C1	0.2181 (3)	0.3702 (5)	0.5856 (2)	0.055 (2)
C2	0.2389 (4)	0.2480 (6)	0.6027 (3)	0.075 (3)
C3	0.3017 (4)	0.2144 (6)	0.5954 (3)	0.080 (3)
C4	0.3466 (3)	0.2991 (6)	0.5720 (3)	0.067 (2)
C5	0.3233 (3)	0.4172 (5)	0.5548 (3)	0.055 (2)
C6	0.1498 (3)	0.4111 (7)	0.5946 (4)	0.082 (3)
C7	0.4189 (4)	0.2725 (9)	0.5664 (6)	0.110 (4)
C11	0.1789 (2)	0.8156 (5)	0.6075 (3)	0.050 (2)
C12	0.1382 (3)	0.8552 (6)	0.6725 (3)	0.070 (2)
C13	0.1051 (6)	0.745 (1)	0.7073 (6)	0.131 (5)
C14	0.0890 (5)	0.953 (1)	0.6469 (6)	0.127 (5)
C15	0.1851 (5)	0.9167 (9)	0.7283 (4)	0.112 (4)
C21	0.1478 (2)	0.6960 (5)	0.4222 (2)	0.049 (2)
C22	0.0899 (3)	0.6946 (5)	0.3691 (3)	0.068 (2)
C23	0.0516 (8)	0.810 (1)	0.373 (1)	0.22 (1)
C24	0.048 (1)	0.586 (2)	0.381 (2)	0.29 (2)
C25	0.1133 (7)	0.678 (4)	0.2982 (8)	0.36 (2)

Table 2. *Geometric parameters* (Å, °) *for* (I)

Ni—O11	1.997 (3)	O11—C11	1.258 (6)
Ni—O12	2.013 (3)	O12—C11^i	1.250 (6)
Ni—O21	2.015 (3)	O21—C21	1.252 (6)
Ni—O22	2.048 (3)	O22—C21^i	1.248 (6)
Ni—N	2.034 (3)	C11—C12	1.523 (7)
Ni—Ni^i	2.7202 (8)	C21—C22	1.520 (7)
O11—Ni—O12	165.7 (1)	O22—Ni—Ni^i	69.12 (9)
O11—Ni—O21	91.0 (1)	N—Ni—Ni^i	160.8 (1)
O11—Ni—O22	88.6 (1)	Ni—O11—C11	124.0 (3)
O11—Ni—N	101.1 (1)	Ni—O12—C11^i	124.5 (3)
O11—Ni—Ni^i	83.4 (1)	Ni—O21—C21	109.0 (3)
O12—Ni—O21	88.3 (1)	Ni—O22—C21^i	140.9 (3)
O12—Ni—O22	88.4 (1)	O11—C11—C12	117.4 (4)
O12—Ni—N	93.0 (1)	O11—C11—O12^i	125.5 (4)
O12—Ni—Ni^i	82.50 (9)	C12—C11—O12^i	117.1 (5)
O21—Ni—O22	165.4 (1)	O21—C21—C22	118.5 (4)
O21—Ni—N	102.2 (1)	O21—C21—O22^i	124.6 (4)
O21—Ni—Ni^i	96.30 (9)	C22—C21—O22^i	116.9 (4)
O22—Ni—N	92.2 (1)		

Symmetry code: (i) $1/2-x, 1/2-y, -z$.

Compound (II)

Crystal data

$[Ni(C_{15}H_{13}O_2)_2(C_9H_7N)]_2.2CHCl_3$
M_r = 1515.5
Triclinic
$P\bar{1}$
a = 13.231 (1) Å
b = 13.857 (1) Å
c = 11.425 (1) Å
α = 99.48 (1)°
β = 104.63 (1)°
γ = 109.68 (1)°
V = 1835.3 (3) Å^3
Z = 1
D_x = 1.371 Mg m^{-3}

D_m = 1.38 (1) Mg m^{-3}
Density measured by flotation in ethanol/ tetrachloromethane
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 36 reflections
θ = 10-15°
μ = 0.790 mm^{-1}
T = 297 K
Prism
0.60 × 0.50 × 0.20 mm
Yellow

Data collection

Rigaku AFC-5 four-circle diffractometer
ω scans
Absorption correction: by integration from crystal shape
T_{min} = 0.64, T_{max} = 0.79
8757 measured reflections
8408 independent reflections
5847 observed reflections [$|F_o|>3\sigma(|F_o|)$]

R_{int} = 0.014
θ_{max} = 27.49°
$h = -17 \rightarrow 17$
$k = -17 \rightarrow 0$
$l = -14 \rightarrow 14$
5 standard reflections monitored every 100 reflections
intensity variation: 1.8%

Refinement

Refinement on F
Final R = 0.081
wR = 0.087
S = 6.176
5847 reflections
545 parameters

All H-atom parameters refined
Calculated weights, $w=1/\sigma$
$(\Delta/\sigma)_{max}$ = 0.484
$\Delta\rho_{max}$ = 1.194 e Å^{-3}
$\Delta\rho_{min}$ = −1.571 e Å^{-3}

Table 3. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *for* (II)

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

	x	y	z	U_{eq}
Ni	−0.00021 (8)	0.02506 (8)	0.12216 (9)	0.0243 (4)
O11	0.1496 (4)	0.0088 (4)	0.1654 (4)	0.034 (2)
O12	−0.1547 (4)	0.0213 (4)	0.0305 (4)	0.038 (2)
O21	0.0802 (4)	0.1635 (4)	0.0835 (4)	0.039 (2)
O22	−0.0736 (4)	−0.1296 (4)	0.1173 (4)	0.039 (2)

N	0.0218 (5)	0.1009 (5)	0.2997 (5)	0.034 (3)
C1	0.1234 (8)	0.1787 (7)	0.3620 (8)	0.047 (4)
C2	0.1609 (9)	0.2309 (8)	0.4911 (8)	0.062 (4)
C3	0.0891 (9)	0.1970 (8)	0.5576 (8)	0.061 (5)
C4	−0.0197 (7)	0.1180 (7)	0.4962 (7)	0.043 (4)
C5	−0.099 (1)	0.0798 (9)	0.5611 (9)	0.057 (5)
C6	−0.202 (1)	0.006 (1)	0.498 (1)	0.069 (6)
C7	−0.2392 (8)	−0.0372 (8)	0.3654 (9)	0.063 (5)
C8	−0.1655 (7)	−0.0079 (7)	0.3022 (8)	0.045 (4)
C9	−0.0552 (7)	0.0710 (6)	0.3658 (7)	0.035 (3)
C101	0.1972 (6)	−0.0058 (6)	0.0853 (6)	0.029 (3)
C102	0.3164 (6)	−0.0072 (6)	0.1332 (6)	0.031 (3)
C103	0.3808 (9)	0.026 (1)	0.043 (1)	0.051 (5)
C104	0.2957 (6)	−0.1220 (6)	0.1383 (7)	0.035 (3)
C105	0.2453 (7)	−0.1627 (7)	0.2205 (8)	0.045 (4)
C106	0.2277 (9)	−0.2647 (9)	0.230 (1)	0.061 (5)
C107	0.259 (1)	−0.3299 (9)	0.155 (1)	0.076 (6)
C108	0.310 (1)	−0.292 (1)	0.073 (1)	0.089 (7)
C109	0.3279 (9)	−0.1870 (9)	0.065 (1)	0.067 (5)
C110	0.3869 (6)	0.0733 (6)	0.2623 (7)	0.035 (3)
C111	0.3771 (7)	0.1684 (7)	0.2963 (8)	0.044 (4)
C112	0.4446 (8)	0.2443 (8)	0.4094 (9)	0.061 (5)
C113	0.524 (1)	0.2221 (9)	0.494 (1)	0.079 (5)
C114	0.5387 (9)	0.1283 (9)	0.4614 (9)	0.077 (5)
C115	0.4689 (8)	0.0542 (7)	0.3459 (9)	0.056 (4)
C201	0.1010 (6)	0.1920 (6)	−0.0102 (7)	0.033 (3)
C202	0.1600 (6)	0.3116 (6)	0.0038 (7)	0.035 (3)
C203	0.0654 (9)	0.3420 (9)	−0.068 (1)	0.058 (5)
C204	0.2114 (8)	0.3749 (6)	0.1455 (7)	0.043 (4)
C205	0.3273 (8)	0.4189 (7)	0.2080 (8)	0.052 (4)
C206	0.369 (1)	0.4760 (9)	0.334 (1)	0.073 (5)
C207	0.300 (1)	0.4886 (9)	0.398 (1)	0.079 (6)
C208	0.187 (1)	0.445 (1)	0.336 (1)	0.086 (7)
C209	0.140 (1)	0.3883 (9)	0.210 (1)	0.066 (5)
C210	0.2542 (7)	0.3298 (6)	−0.0578 (7)	0.041 (4)
C211	0.3170 (8)	0.2677 (7)	−0.0511 (9)	0.055 (4)
C212	0.4046 (9)	0.2826 (9)	−0.101 (1)	0.077 (6)
C213	0.425 (1)	0.365 (1)	−0.160 (1)	0.101 (8)
C214	0.368 (1)	0.427 (1)	−0.169 (1)	0.080 (7)
C215	0.2804 (9)	0.4107 (8)	−0.1152 (9)	0.060 (5)
C301	−0.2043 (5)	0.3083 (4)	0.3253 (4)	0.249 (2)
Cl1	−0.3345 (7)	0.304 (2)	0.2630 (8)	0.249 (2)
Cl2	−0.150 (2)	0.254 (1)	0.258 (1)	0.249 (2)
Cl3	−0.132 (2)	0.4571 (4)	0.326 (1)	0.249 (2)
Cl4	−0.1328 (9)	0.322 (1)	0.4557 (6)	0.249 (2)

Table 4. *Geometric parameters* (Å, °) *for* (II)

Ni—O11	2.016 (6)	O11—C101	1.26 (1)
Ni—O12	2.026 (5)	O12—C101^{i}	1.249 (8)
Ni—O21	2.037 (5)	O21—C201	1.26 (1)
Ni—O22	2.018 (5)	O22—C201^{i}	1.261 (9)
Ni—N	2.024 (6)	C101—C102	1.54 (1)
Ni—Ni^{i}	2.765 (1)	C201—C202	1.54 (1)
O11—Ni—O12	164.2 (2)	O22—Ni—Ni^{i}	92.7 (2)
O11—Ni—O21	87.6 (2)	N—Ni—Ni^{i}	165.0 (2)
O11—Ni—O22	87.0 (2)	Ni—O11—C101	123.0 (4)
O11—Ni—N	95.6 (2)	Ni—O12—C101^{i}	126.6 (6)
O11—Ni—Ni^{i}	83.8 (1)	Ni—O21—C201	137.3 (4)
O12—Ni—O21	91.3 (2)	Ni—O22—C201^{i}	112.7 (5)
O12—Ni—O22	89.9 (2)	O11—C101—C102	117.6 (6)
O12—Ni—N	100.2 (3)	O11—C101—O12^{i}	125.3 (7)
O12—Ni—Ni^{i}	80.9 (2)	C102—C101—O12^{i}	117.1 (7)
O21—Ni—O22	164.3 (2)	O21—C201—C202	118.4 (6)
O21—Ni—N	92.9 (2)	O21—C201—O22^{i}	124.9 (7)
O21—Ni—Ni^{i}	72.1 (1)	C202—C201—O22^{i}	116.7 (7)
O22—Ni—N	102.3 (2)		

Symmetry code: (i) $-x, -y, -z$.

Compound (III)

Crystal data

$[Ni(C_{10}H_{11}O_2)_2(C_9H_7N)]_2$
M_r = 1028.5

Density measured by flotation in KI solution
Monoclinic
$P2_1/n$
a = 10.992 (1) Å
b = 20.932 (1) Å
c = 10.964 (1) Å
β = 90.587 (5)°
V = 2522.5 (3) Å^3
Z = 2
D_x = 1.354 Mg m^{-3}
D_m = 1.31 (2) Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 38 reflections
θ = 10–15°
μ = 0.804 mm^{-1}
T = 300 K
Plate
0.60 × 0.25 × 0.10 mm
Dark green

Data collection

Rigaku AFC-5 four-circle diffractometer
ω scans
Absorption correction: by integration from crystal shape
T_{min} = 0.82, T_{max} = 0.92
4671 measured reflections
4431 independent reflections
3029 observed reflections [$|F_o|>3\sigma(|F_o|)$]

R_{int} = 0.014
θ_{max} = 25°
h = 0 → 13
k = 0 → 24
l = −13 → 13
5 standard reflections monitored every 100 reflections
intensity variation: 1.8%

Refinement

Refinement on F
Final R = 0.041
wR = 0.026
S = 1.726
3029 reflections
432 parameters

All H-atom parameters refined
Calculated weights, $w=1/\sigma$
$(\Delta/\sigma)_{max}$ = 0.068
$\Delta\rho_{max}$ = 0.276 e Å^{-3}
$\Delta\rho_{min}$ = −0.335 e Å^{-3}

Table 5. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *for* (III)

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

	x	y	z	U_{eq}
Ni	0.61870 (4)	0.51310 (2)	0.52872 (4)	0.0295 (2)
O11	0.6367 (2)	0.5892 (1)	0.4166 (2)	0.040 (2)
O12	0.5541 (2)	0.4376 (1)	0.6245 (2)	0.042 (2)
O21	0.6492 (2)	0.4541 (1)	0.3886 (2)	0.036 (2)
O22	0.5444 (2)	0.5720 (1)	0.6532 (2)	0.038 (2)
N	0.7901 (2)	0.5145 (2)	0.6017 (2)	0.033 (2)
C1	0.8303 (4)	0.5719 (2)	0.6287 (4)	0.043 (3)
C2	0.9499 (4)	0.5841 (2)	0.6715 (4)	0.052 (3)
C3	1.0273 (4)	0.5341 (2)	0.6837 (4)	0.052 (3)
C4	0.9897 (3)	0.4719 (2)	0.6579 (3)	0.041 (3)
C5	1.0660 (4)	0.4182 (3)	0.6700 (4)	0.056 (3)
C6	1.0256 (4)	0.3594 (3)	0.6457 (4)	0.063 (4)
C7	0.9049 (4)	0.3497 (2)	0.6051 (4)	0.054 (3)
C8	0.8285 (4)	0.4002 (2)	0.5919 (4)	0.040 (3)
C9	0.8679 (3)	0.4623 (2)	0.6172 (3)	0.034 (2)
C101	0.5359 (3)	0.5983 (2)	0.3626 (3)	0.032 (2)
C102	0.5199 (3)	0.6566 (2)	0.2812 (3)	0.032 (2)
C103	0.6425 (4)	0.6740 (2)	0.2215 (5)	0.048 (3)
C104	0.4281 (5)	0.6414 (3)	0.1803 (4)	0.053 (3)
C105	0.4803 (3)	0.7121 (2)	0.3631 (3)	0.036 (2)
C106	0.3769 (4)	0.7478 (2)	0.3406 (5)	0.056 (3)
C107	0.3451 (5)	0.7987 (3)	0.4143 (7)	0.076 (4)
C108	0.4153 (6)	0.8142 (3)	0.5129 (6)	0.072 (4)
C109	0.5173 (6)	0.7797 (2)	0.5381 (5)	0.063 (4)
C110	0.5489 (4)	0.7292 (2)	0.4634 (4)	0.051 (3)
C201	0.5685 (3)	0.4240 (2)	0.3293 (3)	0.029 (2)
C202	0.6147 (3)	0.3793 (2)	0.2294 (3)	0.032 (2)
C203	0.5099 (4)	0.3425 (2)	0.1702 (4)	0.049 (3)
C204	0.7016 (5)	0.3320 (2)	0.2905 (5)	0.049 (3)
C205	0.6782 (3)	0.4207 (2)	0.1333 (3)	0.034 (2)

C206	0.7814 (4)	0.4005 (2)	0.0734 (4)	0.053 (3)
C207	0.8360 (5)	0.4384 (3)	−0.0140 (4)	0.068 (4)
C208	0.7887 (5)	0.4965 (2)	−0.0424 (4)	0.067 (4)
C209	0.6877 (5)	0.5173 (2)	0.0147 (4)	0.072 (3)
C210	0.6323 (4)	0.4801 (2)	0.1032 (4)	0.056 (3)

Table 6. *Geometric parameters* (Å, °) *for* (III)

Ni—O11	2.023 (2)	O11—C101	1.265 (4)
Ni—O12	2.030 (2)	O12—C101^{i}	1.250 (4)
Ni—O21	2.001 (2)	O21—C201	1.263 (4)
Ni—O22	2.019 (2)	O22—C201^{i}	1.260 (4)
Ni—N	2.040 (3)	C101—C102	1.522 (5)
Ni—Ni^{i}	2.7337 (7)	C201—C202	1.533 (5)
O11—Ni—O12	164.7 (1)	O22—Ni—Ni^{i}	83.48 (7)
O11—Ni—O21	90.1 (1)	N—Ni—Ni^{i}	165.70 (9)
O11—Ni—O22	88.4 (1)	Ni—O11—C101	108.1 (2)
O11—Ni—N	97.5 (1)	Ni—O12—C101^{i}	143.8 (2)
O11—Ni—Ni^{i}	96.72 (7)	Ni—O21—C201	125.5 (2)
O12—Ni—O21	88.8 (1)	Ni—O22—C201^{i}	123.3 (2)
O12—Ni—O22	88.8 (1)	O11—C101—C102	119.3 (3)
O12—Ni—N	97.8 (1)	O11—C101—O12^{i}	123.2 (3)
O12—Ni—Ni^{i}	67.96 (7)	C102—C101—O12^{i}	117.5 (3)
O21—Ni—O22	165.44 (9)	O21—C201—C202	115.9 (3)
O21—Ni—N	98.6 (1)	O21—C201—O22^{i}	125.2 (3)
O21—Ni—Ni^{i}	82.32 (7)	C202—C201—O22^{i}	118.9 (3)
O22—Ni—N	96.0 (1)		

Symmetry code: (i) $-x, -y, -z$.

Table 7. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *for* (IV)

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

	x	y	z	U_{eq}
Ni	0.87537 (4)	0.01927 (4)	1.02957 (4)	0.0441 (3)
O11	0.7447 (2)	0.1288 (2)	0.8481 (2)	0.058 (2)
O12	1.0496 (2)	−0.1018 (2)	1.1894 (2)	0.064 (2)
O21	0.8903 (2)	−0.1484 (2)	0.9093 (2)	0.058 (2)
O22	0.9037 (2)	0.1797 (2)	1.1323 (2)	0.060 (2)
N	0.7251 (2)	0.0195 (2)	1.1169 (2)	0.045 (2)
C1	0.5933 (3)	0.1228 (3)	1.1185 (3)	0.054 (3)
C2	0.4950 (3)	0.1019 (4)	1.1664 (4)	0.066 (3)
C3	0.5322 (4)	−0.0209 (4)	1.2170 (4)	0.076 (4)
C4	0.6693 (4)	−0.1253 (4)	1.2218 (4)	0.070 (3)
C5	0.7622 (3)	−0.1021 (3)	1.1688 (3)	0.059 (3)
C6	0.5568 (4)	0.2577 (3)	1.0658 (4)	0.073 (3)
C7	0.4582 (7)	0.3741 (5)	1.1221 (7)	0.198 (7)
C11	0.8122 (3)	0.1456 (3)	0.7745 (3)	0.045 (2)
C12	0.7250 (3)	0.2234 (3)	0.6318 (3)	0.058 (3)
C13	0.7592 (5)	0.3420 (4)	0.6370 (5)	0.103 (4)
C14	0.7825 (6)	0.1288 (4)	0.5140 (4)	0.130 (5)
C15	0.5619 (4)	0.2707 (5)	0.6003 (5)	0.145 (6)
C21	0.9916 (3)	−0.2094 (3)	0.8520 (3)	0.046 (2)
C22	0.9893 (3)	−0.3285 (3)	0.7540 (4)	0.059 (3)
C23	1.1163 (7)	−0.3989 (6)	0.7021 (8)	0.236 (9)
C24	0.9674 (8)	−0.4202 (5)	0.8278 (8)	0.206 (9)
C25	0.8530 (7)	−0.2735 (6)	0.6330 (7)	0.295 (8)

Compound (IV)

Crystal data

$[Ni(C_5H_9O_2)_2(C_7H_9N)]_2$
M_r = 736.2
Triclinic
$P\bar{1}$
a = 10.616 (1) Å
b = 11.059 (1) Å
c = 9.826 (1) Å
α = 98.22 (1)°
β = 109.03 (1)°
γ = 62.52 (1)°
V = 967.5 (2) Å^3
Z = 1
D_x = 1.264 Mg m^{-3}
D_m = 1.25 (2) Mg m^{-3}
Density measured by flotation in KI solution
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 38 reflections
θ = 10-15°
μ = 1.022 mm^{-1}
T = 299 K
Plate
0.50 × 0.40 × 0.30 mm
Pale green

Data collection

Rigaku AFC-5 four-circle diffractometer
θ-2θ scans
Absorption correction: by integration from crystal shape
T_{min} = 0.67, T_{max} = 0.74
4706 measured reflections
4444 independent reflections
3344 observed reflections [$|F_o|>3\sigma(|F_o|)$]
R_{int} = 0.010
θ_{max} = 27.49°
$h = -13 \rightarrow 13$
$k = -14 \rightarrow 14$
$l = 0 \rightarrow 12$
5 standard reflections monitored every 100 reflections
intensity variation: 1.4%

Refinement

Refinement on F
Final R = 0.042
wR = 0.037
S = 3.624
3344 reflections
317 parameters
All H-atom parameters refined
Calculated weights, $w=1/\sigma$
$(\Delta/\sigma)_{max}$ = 0.324
$\Delta\rho_{max}$ = 0.582 e Å^{-3}
$\Delta\rho_{min}$ = −0.479 e Å^{-3}

Table 8. *Geometric parameters* (Å, °) *for* (IV)

Ni—O11	2.006 (2)	O11—C11	1.253 (5)
Ni—O12	2.025 (2)	O12—C11^{i}	1.252 (3)
Ni—O21	2.021 (2)	O21—C21	1.242 (4)
Ni—O22	2.010 (2)	O22—C21^{i}	1.256 (5)
Ni—N	2.042 (3)	C11—C12	1.525 (4)
Ni—Ni^{i}	2.7227 (7)	C21—C22	1.521 (4)
O11—Ni—O12	165.0 (1)	O22—Ni—Ni^{i}	79.93 (7)
O11—Ni—O21	87.07 (8)	N—Ni—Ni^{i}	166.00 (6)
O11—Ni—O22	91.80 (9)	Ni—O11—C11	115.1 (2)
O11—Ni—N	102.62 (9)	Ni—O12—C11^{i}	135.6 (2)
O11—Ni—Ni^{i}	91.38 (8)	Ni—O21—C21	121.1 (2)
O12—Ni—O21	89.14 (8)	Ni—O22—C21^{i}	128.2 (2)
O12—Ni—O22	88.23 (9)	O11—C11—C12	119.8 (3)
O12—Ni—N	92.2 (1)	O11—C11—O12^{i}	123.9 (3)
O12—Ni—Ni^{i}	73.85 (8)	C12—C11—O12^{i}	116.3 (3)
O21—Ni—O22	165.4 (1)	O21—C21—C22	118.2 (3)
O21—Ni—N	94.7 (1)	O21—C21—O22^{i}	125.0 (3)
O21—Ni—Ni^{i}	85.56 (8)	C22—C21—O22^{i}	116.8 (3)
O22—Ni—N	99.7 (1)		

Symmetry code: (i) $-x, -y, -z$.

Compound (V)

Crystal data

$[Ni(C_5H_9O_2)_2(C_6H_7N)]_2$
M_r = 708.1
Triclinic
$P\bar{1}$
a = 10.541 (1) Å
b = 10.724 (1) Å
c = 9.680 (1) Å
α = 97.20 (1)°
β = 108.48 (1)°
γ = 63.31 (1)°
V = 927.1 (2) Å^3
Z = 1
D_x = 1.268 Mg m^{-3}
D_m = 1.23 (2) Mg m^{-3}
Density measured by flotation in KI solution
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 41 reflections
θ = 12-15°
μ = 1.064 mm^{-1}
T = 299 K
Prism
0.45 × 0.40 × 0.30 mm
Pale green

Data collection

Rigaku AFC-5 four-circle diffractometer
ω scans
Absorption correction: by integration from crystal shape
T_{min} = 0.63, T_{max} = 0.75
4515 measured reflections
4264 independent reflections
3505 observed reflections [$|F_o|>3\sigma(|F_o|)$]

R_{int} = 0.009
θ_{max} = 27.5°
$h = -13 \rightarrow 13$
$k = -13 \rightarrow 13$
$l = 0 \rightarrow 12$
5 standard reflections monitored every 100 reflections
intensity variation: 1.4%

Refinement

Refinement on F
Final R = 0.039
wR = 0.035
S = 3.36
3505 reflections
300 parameters

All H-atom parameters refined
Calculated weights, $w=1/\sigma$
$(\Delta/\sigma)_{max}$ = 0.252
$\Delta\rho_{max}$ = 0.459 e Å^{-3}
$\Delta\rho_{min}$ = −0.341 e Å^{-3}

Table 9. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *for* (V)

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

	x	y	z	U_{eq}
Ni	0.10741 (4)	0.01601 (4)	0.96595 (4)	0.0383 (2)
O11	0.1330 (2)	0.1246 (2)	1.1495 (2)	0.054 (2)
O12	0.0484 (2)	−0.1024 (2)	0.8042 (2)	0.056 (2)
O21	−0.0744 (2)	0.1844 (2)	0.8656 (2)	0.056 (2)
O22	0.2533 (2)	−0.1585 (2)	1.0860 (2)	0.055 (2)
N	0.2557 (2)	0.0250 (2)	0.8791 (2)	0.045 (2)
C1	0.2864 (3)	0.1320 (3)	0.8774 (3)	0.062 (3)
C2	0.4055 (3)	0.1160 (4)	0.8285 (4)	0.078 (3)
C3	0.4851 (4)	−0.0083 (5)	0.7803 (4)	0.090 (4)
C4	0.4534 (3)	−0.1199 (4)	0.7755 (4)	0.087 (3)
C5	0.3391 (3)	−0.1003 (3)	0.8269 (3)	0.066 (3)
C6	0.1957 (4)	0.2669 (4)	0.9290 (4)	0.084 (3)
C11	0.0476 (3)	0.1477 (2)	1.2238 (3)	0.042 (2)
C12	0.0562 (3)	0.2394 (3)	1.3565 (3)	0.062 (3)
C13	0.1955 (5)	0.2604 (5)	1.4012 (5)	0.151 (6)
C14	0.0506 (7)	0.1736 (6)	1.4813 (5)	0.173 (8)
C15	−0.0708 (6)	0.3728 (5)	1.3194 (7)	0.254 (6)
C21	−0.2077 (3)	0.2204 (2)	0.8543 (3)	0.043 (2)
C22	−0.3232 (3)	0.3458 (3)	0.7553 (3)	0.059 (2)
C23	−0.2565 (4)	0.4336 (4)	0.7265 (5)	0.129 (4)
C24	−0.4487 (5)	0.4312 (4)	0.8172 (7)	0.185 (5)
C25	−0.3837 (7)	0.2863 (5)	0.6146 (6)	0.212 (7)

Table 10. *Geometric parameters* (Å, °) *for* (V)

Ni—O11	2.012 (2)	O11—C11	1.247 (4)
Ni—O12	2.011 (2)	O12—C11^i	1.252 (4)
Ni—O21	2.007 (1)	O21—C21	1.250 (4)
Ni—O22	2.012 (2)	O22—C21^i	1.250 (4)
Ni—N	2.037 (3)	C11—C12	1.521 (4)
Ni—Ni^i	2.7171 (7)	C21—C22	1.523 (3)
O11—Ni—O12	165.3 (1)	O22—Ni—Ni^i	86.74 (7)
O11—Ni—O21	90.32 (7)	N—Ni—Ni^i	169.47 (7)
O11—Ni—O22	87.74 (7)	Ni—O11—C11	117.7 (2)
O11—Ni—N	101.8 (1)	Ni—O12—C11^i	132.2 (2)
O11—Ni—Ni^i	88.61 (7)	Ni—O21—C21	129.6 (2)
O12—Ni—O21	89.07 (7)	Ni—O22—C21^i	119.7 (2)
O12—Ni—O22	89.19 (7)	O11—C11—C12	118.8 (3)
O12—Ni—N	92.8 (1)	O11—C11—O12^i	124.5 (2)
O12—Ni—Ni^i	76.83 (7)	C12—C11—O12^i	116.6 (3)
O21—Ni—O22	165.5 (1)	O21—C21—C22	117.2 (3)
O21—Ni—N	99.12 (9)	O21—C21—O22^i	125.0 (2)
O21—Ni—Ni^i	78.89 (7)	C22—C21—O22^i	117.8 (2)
O22—Ni—N	95.30 (9)		

Symmetry code: (i) $-x, -y, -z$.

Compound (VI)

Crystal data

$[Ni(C_{14}H_{13}O_2Si)_2(C_{18}H_{15}P)]_2$
M_r = 1607.3
Monoclinic
$P2_1/n$
a = 14.461 (1) Å
b = 23.432 (1) Å
c = 13.542 (1) Å
β = 112.921 (4)°
V = 4226.4 (4) Å^3
Z = 2
D_x = 1.263 Mg m^{-3}
D_m = 1.25 (1) Mg m^{-3}

Density measured by flotation in n-butanol/ tetrachloromethane
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 33 reflections
θ = 12–15°
μ = 0.592 mm^{-1}
T = 299 K
Prism
0.60 × 0.50 × 0.35 mm
Dark green

Data collection

Rigaku AFC-5 four-circle diffractometer
θ–2θ scans
Absorption correction: by integration from crystal shape
T_{min} = 0.76, T_{max} = 0.84
8385 measured reflections
7989 independent reflections
4341 observed reflections [$|F_o|>3\sigma(|F_o|)$]

R_{int} = 0.017
θ_{max} = 27.5°
$h = -18 \rightarrow 18$
$k = -30 \rightarrow 0$
$l = 0 \rightarrow 17$
5 standard reflections monitored every 100 reflections
intensity variation: 13.2%

Refinement

Refinement on F
Final R = 0.044
wR = 0.035
S = 2.666
4341 reflections
652 parameters

All H-atom parameters refined
Calculated weights, $w=1/\sigma$
$(\Delta/\sigma)_{max}$ = 0.222
$\Delta\rho_{max}$ = 0.420 e Å^{-3}
$\Delta\rho_{min}$ = −0.535 e Å^{-3}

Table 11. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *for* (VI)

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

	x	y	z	U_{eq}
Ni	0.47838 (4)	0.54671 (2)	0.43614 (5)	0.0304 (2)
P	0.42624 (8)	0.63672 (5)	0.3538 (1)	0.0331 (6)
Si1	0.18655 (9)	0.49288 (6)	0.4903 (1)	0.0393 (7)
Si2	0.60156 (9)	0.61091 (5)	0.7933 (1)	0.0403 (7)
O11	0.3472 (2)	0.5308 (1)	0.4481 (2)	0.043 (2)
O12	0.6198 (2)	0.5443 (1)	0.4426 (2)	0.048 (2)
O21	0.5333 (2)	0.5831 (1)	0.5813 (2)	0.044 (2)
O22	0.4373 (2)	0.4928 (1)	0.3107 (2)	0.043 (2)
C1	0.3987 (3)	0.6876 (2)	0.4402 (3)	0.033 (2)
C2	0.3470 (3)	0.6686 (2)	0.4993 (4)	0.052 (3)
C3	0.3169 (4)	0.7056 (2)	0.5613 (4)	0.067 (3)
C4	0.3394 (4)	0.7628 (2)	0.5638 (4)	0.063 (3)
C5	0.3897 (4)	0.7817 (2)	0.5057 (4)	0.061 (3)
C6	0.4186 (3)	0.7460 (2)	0.4430 (4)	0.046 (3)
C7	0.3131 (3)	0.6383 (2)	0.2309 (3)	0.035 (2)
C8	0.2580 (3)	0.6877 (2)	0.1959 (4)	0.060 (3)
C9	0.1728 (3)	0.6880 (2)	0.1044 (4)	0.069 (3)
C10	0.1394 (3)	0.6399 (2)	0.0459 (4)	0.056 (3)
C11	0.1923 (3)	0.5908 (2)	0.0785 (4)	0.063 (3)
C12	0.2797 (3)	0.5895 (2)	0.1723 (4)	0.050 (3)
C13	0.5217 (3)	0.6702 (2)	0.3177 (3)	0.039 (2)
C14	0.6103 (3)	0.6883 (2)	0.3980 (4)	0.059 (3)
C15	0.6867 (4)	0.7105 (2)	0.3732 (5)	0.084 (4)
C16	0.6774 (4)	0.7156 (3)	0.2700 (5)	0.100 (5)

C17	0.5924 (4)	0.6973 (3)	0.1896 (5)	0.110 (5)
C18	0.5143 (4)	0.6753 (2)	0.2141 (4)	0.075 (4)
C101	0.3215 (3)	0.4941 (2)	0.4993 (3)	0.037 (2)
C102	0.1891 (3)	0.4721 (2)	0.6219 (4)	0.062 (3)
C103	0.1185 (3)	0.4371 (2)	0.3891 (4)	0.043 (3)
C104	0.0248 (3)	0.4447 (2)	0.3085 (4)	0.051 (3)
C105	−0.0295 (3)	0.4016 (2)	0.2439 (4)	0.072 (3)
C106	0.0112 (4)	0.3478 (2)	0.2579 (4)	0.080 (4)
C107	0.1050 (4)	0.3374 (2)	0.3336 (5)	0.087 (4)
C108	0.1570 (3)	0.3829 (2)	0.3981 (4)	0.071 (3)
C109	0.1256 (3)	0.5644 (2)	0.4432 (4)	0.044 (3)
C110	0.1143 (3)	0.5861 (2)	0.3450 (4)	0.061 (3)
C111	0.0654 (4)	0.6371 (2)	0.3077 (4)	0.077 (4)
C112	0.0287 (4)	0.6666 (2)	0.3693 (5)	0.099 (4)
C113	0.0392 (4)	0.6477 (2)	0.4678 (6)	0.107 (5)
C114	0.0880 (4)	0.5958 (2)	0.5045 (4)	0.068 (4)
C201	0.5618 (3)	0.5605 (2)	0.6727 (3)	0.033 (2)
C202	0.6254 (4)	0.6829 (2)	0.7465 (4)	0.080 (3)
C203	0.7215 (3)	0.5847 (2)	0.8996 (3)	0.037 (2)
C204	0.7374 (3)	0.5818 (2)	1.0065 (4)	0.055 (3)
C205	0.8309 (4)	0.5670 (2)	1.0851 (4)	0.065 (3)
C206	0.9075 (3)	0.5556 (2)	1.0550 (4)	0.063 (3)
C207	0.8949 (3)	0.5568 (2)	0.9505 (4)	0.057 (3)
C208	0.8027 (3)	0.5712 (2)	0.8732 (4)	0.050 (3)
C209	0.4993 (3)	0.6127 (2)	0.8421 (4)	0.067 (3)
C210	0.4525 (5)	0.5669 (3)	0.8525 (7)	0.176 (7)
C211	0.3758 (7)	0.5700 (7)	0.8869 (9)	0.39 (2)
C212	0.3457 (5)	0.6076 (5)	0.9173 (6)	0.26 (1)
C213	0.3869 (6)	0.6629 (4)	0.9059 (6)	0.217 (8)
C214	0.4664 (6)	0.6626 (3)	0.8706 (6)	0.160 (7)

Table 12. *Geometric parameters* (Å, °) *for* (VI)

Ni—P	2.368 (1)	Si1—C101	1.908 (5)
Ni—O11	2.001 (3)	Si2—C201	1.914 (4)
Ni—O12	2.013 (3)	O11—C101	1.249 (6)
Ni—O21	2.002 (3)	O12—C101^{i}	1.275 (5)
Ni—O22	2.013 (3)	O21—C201	1.259 (5)
Ni—Ni^{i}	2.7079 (8)	O22—C201^{i}	1.268 (5)
P—Ni—O11	93.78 (8)	O21—Ni—Ni^{i}	79.19 (8)
P—Ni—O12	100.23 (9)	O22—Ni—Ni^{i}	87.17 (8)
P—Ni—O21	90.86 (8)	Ni—O11—C101	132.0 (2)
P—Ni—O22	103.07 (8)	Ni—O12—C101^{i}	117.9 (3)
P—Ni—Ni^{i}	166.66 (5)	Ni—O21—C201	129.7 (2)
O11—Ni—O12	166.0 (1)	Ni—O22—C201^{i}	119.2 (3)
O11—Ni—O21	91.5 (1)	Si1—C101—O11	119.5 (3)
O11—Ni—O22	88.5 (1)	Si1—C101—O12^{i}	116.6 (3)
O11—Ni—Ni^{i}	77.78 (8)	O11—C101—O12^{i}	123.9 (4)
O12—Ni—O21	87.8 (1)	Si2—C201—O21	116.9 (3)
O12—Ni—O22	88.8 (1)	Si2—C201—O22^{i}	118.5 (3)
O12—Ni—Ni^{i}	88.35 (9)	O21—C201—O22^{i}	124.5 (4)
O21—Ni—O22	166.0 (1)		

Symmetry code: (i) $-x, -y, -z$.

In compound (II), the chloroform solvent molecules exhibit rotational disorder. Four possible positions of the Cl atoms around C301, labeled as Cl1—Cl4, were each assumed to have occupation probabilities of 3/4 and were refined with a rigid-body approximation. The relatively large R value for (II) may be the result of the disorder of the $CHCl_3$ molecule.

The programs used to solve the structures were *Xtal*3.0 *FOURR* and *Xtal*3.0 *SIMPEL* (Hall & Stewart, 1990). The structures were refined using *Xtal*3.0 *CRYSLQ*, molecular graphics were produced using *Xtal*3.0 *ORTEP* and material produced for publication with *Xtal*3.0 *BONDLA* and *Xtal*3.0 *CIFIO*. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B) were used for non-H atoms. For the H atoms the values were taken from Stewart, Davidson & Simpson (1965).

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55199 (105 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1007]

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Structure of [2-(Fluorodimethylstannyl)-ethyl]diphenylphosphine Oxide

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Abstract

The atoms bound to Sn form a distorted trigonal bipyramid with the O and F atoms in the apical positions [Sn—O 2.454(3), Sn—F 2.035(2) Å; O—Sn—F 172.66(9)°] and the C atoms in the equatorial positions [Sn—C 2.123(5), 2.124(4), 2.158(3) Å; C—Sn—C 123.9(2), 115.7(2), 118.2(2)°; C—Sn—F 93.6(1), 94.3(1), 97.3(1)°; C—Sn—O 80.5(1), 85.5(1), 89.2(2)°]. The atoms bound to P form a slightly distorted tetrahedron with bond angles in the range 106.2(2)-112.1(2)°. The five-membered ring has an envelope conformation; the atoms P, O, Sn and C(3) are nearly coplanar.

Comment

The compounds [2-(bromodimethylstannyl)ethyl]diphenylphosphine sulfide (Preut, Godry & Mitchell, 1992*a*) and [2-(chlorodimethylstannyl)ethyl]diphenylphosphine