Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

[1,2-Bis(diethylphosphino)ethane]dichloronickel(II)

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Received 11 July 2005
Accepted 26 July 2005
Online 6 August 2005

The neutral title complex, $[NiCl_2(C_{10}H_{24}P_2)]$ or $[NiCl_2(depe)]$, where depe is 1,2-bis(diethylphosphino)ethane, has two independent molecules in the asymmetric unit. The Ni atoms in both molecules are coordinated in a slightly distorted square-planar geometry by the two P atoms and two Cl^- ions, with bond dimensions as expected. The geometry of the depe ligand in one of the molecules is typical; there is disorder in the ethyl groups in the second molecule, however, leading to some slightly distorted dimensions. The two independent molecules form discrete columns parallel to the crystallographic *a* axis; these 'ordered' and 'disordered' columns alternate along the crystallographic *b* and *c* directions, with short Cl···H van der Waals contacts linking four columns.

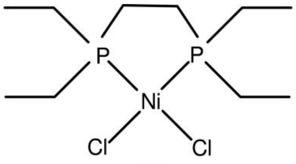
Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.033
wR factor = 0.089
Data-to-parameter ratio = 21.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The title compound, (I), was prepared as a starting material for the preparation of new heterometallic nickel–iron complexes, which have structural and functional properties related to those of the active site of the enzyme NiFe-hydrogenase (Smith *et al.*, 2002, 2003; Evans & Pickett, 2003). Good quality crystals of (I) were obtained, as unreacted starting material, from an attempted preparation of novel dinickel coordination complexes that have an analogy to the active site structures of certain other metalloenzymes (Duff *et al.*, 2005; Evans, 2005).



(I)

There are two independent molecules in the asymmetric unit, 1 and 2, the second being disordered (see Fig. 1). Two distinct orientations were determined for the ethyl C atoms (except for one shared terminal C) in the second molecule; relative occupancies were 83.3 (4) and 16.7 (4)%. Owing to the low scattering power of the minor disordered component, bond lengths were restrained to be effectively equivalent to those in molecule 1. Each Ni atom is slightly distorted square-planar coordinated by the two P and two Cl^- ions; the bond dimensions about the Ni atoms are as expected (Table 1). The Ni atoms lie 0.0185 (4) Å from the Cl_2P_2 mean plane in molecule 1, and 0.047 (2) and −0.061 (11) Å in 2*a* and 2*b* respectively (+ and − indicate opposite sides of the plane). The geometry of the depe ligand in molecule 1 is typical, with the bridging C atoms lying −0.120 (3) and 0.246 (3) Å from the NiP_2 plane; in molecule 2 the equivalent distances are −0.387 (4) and 0.062 (5) Å in 2*a*, and 0.762 (15) and 0.839 (14) Å in 2*b*. The torsion angles in the major component of molecule 2 are also slightly larger than those in molecule 1 (Table 1). In the ordered molecule, the atoms lie in two intersecting planes, one formed by Cl_2NiP_2 and the bridging C atoms [designated plane 1(i)], and the second formed by P_2 and the ethyl C atoms [designated plane 1(ii)] (see Fig. 2). The largest deviation from the 1(i) NiP_2 sub-plane is 0.246 (3) Å for C2 and that from the 1(ii) P_2C111 sub-plane is −0.170 (4) Å, for C122, with an angle between the normals to the sub-planes of 88.77 (9)°. The disorder in the ethyl groups of the second molecule results in some degree of loss of planarity in the equivalent planes. For the major component, the largest deviation from the 2*a*(i) NiP_2 sub-plane is 0.387 (4) Å for C3, while the 2*a*(ii) P_2C311 sub-plane shows a more marked loss of planarity, the largest deviations from the sub-plane being 0.959 (5) Å for C312 and −0.430 (6) Å for C412. The angle between the normals to the 2*a*(i) and 2*a*(ii) sub-planes is 85.01 (13)°. For the minor component, planarity is virtually completely removed, the largest distances to the 2*b*(i) NiP_2 sub-plane being 0.839 (14) and 0.762 (15) Å for C4*b* and C3*b* respectively. The largest distances to the 2*b*(ii) P_2C331 sub-plane are −1.246 (5), −1.156 (17) and 0.477 (23) Å for atoms C322, C442 and C332, respectively; the angle between the normals to the 2*b*(i) and 2*a*(ii) sub-planes is 71.0 (8)°. The major orientation of the disordered molecule is similar to that of the ordered molecule, the two being related by a pseudo-twofold screw axis on which the two Ni atoms lie; no crystallographic pseudosymmetry relates the minor disordered C atoms to those in the ordered molecule and so the pseudo-monoclinic symmetry of the crystal system is reduced to triclinic.

The two independent molecules of (I) in the crystal structure form discrete columns parallel to the crystallographic *a* direction; these 'ordered' and 'disordered' columns alternate along the crystallographic *a* and *b* axial directions (see Fig. 3). Short van der Waals contacts (Table 2), or weak hydrogen bonds, between an H atom of each bridging alkyl C atom in the ordered molecule and a Cl atom of the major component of the disordered molecule, are present; these bonds link two ordered and two disordered molecules into tetrads about a centre of symmetry. The Cl_2H_4 plane is puckered, with the H2*B* atoms lying ±1.345 Å from the $Cl4_2H1B_2$ mean plane.

Experimental

To a solution of $NiCl_2\cdot6H_2O$ (13.2 g, 55 mmol) in ethanol (50 ml), under an atmosphere of dinitrogen, was added a solution of depe (5 g, 55 mmol) in ethanol (10 ml). The red–orange solution that formed immediately was stirred for 1 h. After reducing the volume *in vacuo* to approximately 30 ml, the solution was placed in a freezer overnight, during which time orange material separated out. Filtration gave the product (I), which was washed with diethyl ether then dried *in vacuo* (8.63 g, 47%). Analysis expected for $C_{10}H_{24}Cl_2NiP_2$: C 35.8, H 7.2%; found C 35.9, H 7.3%. Solution ^{31}P NMR (CD_3CN; ref. phosphoric acid): 78.04 p.p.m. Crystals were obtained as recovered

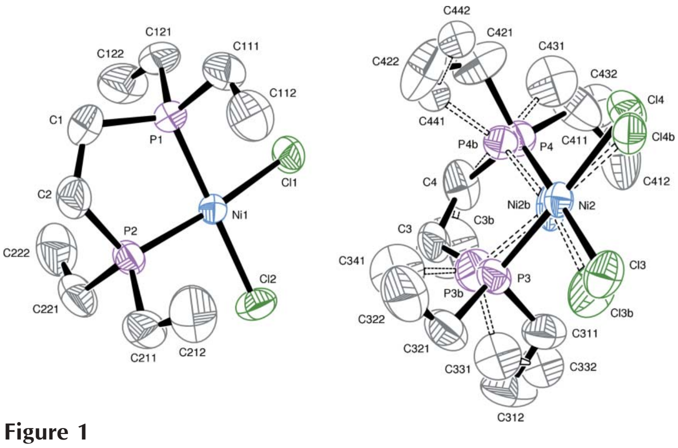


Figure 1
A view of both molecules in (I), showing the disorder in the second and relative orientations. The minor disordered component is shown with dashed bonds. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted.

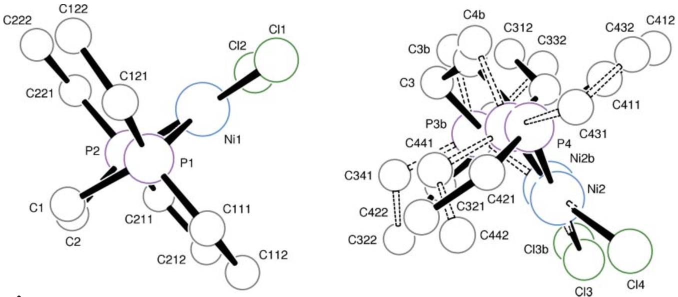


Figure 2
View showing the pseudo-twofold screw axis relating the two molecules. Atoms are represented by arbitrary spheres for clarity, and H atoms have been omitted.

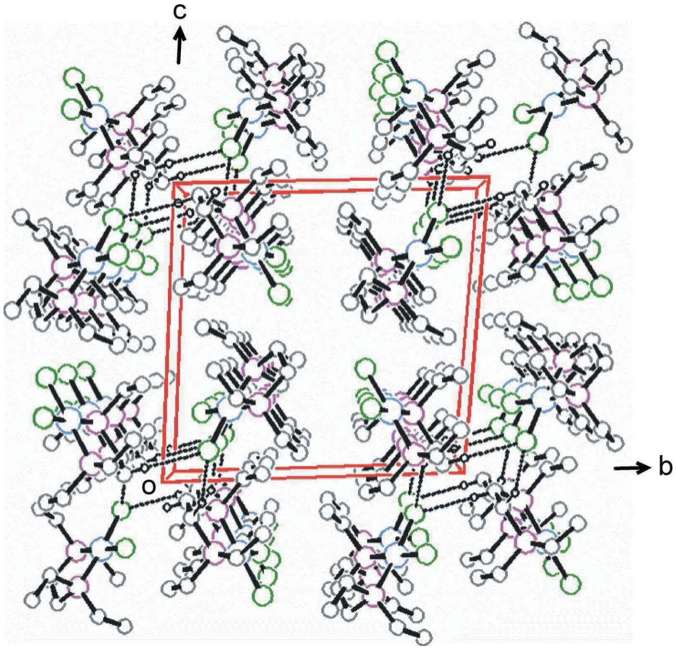


Figure 3
Packing arrangement in (I), as viewed along the [100] vector, showing tetrads generated by weak hydrogen bonds, which are drawn as dashed lines. Atoms are drawn as arbitrary spheres for clarity, and only H atoms involved in the hydrogen bonding are shown.

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starting material from an attempted reaction of [Ni(depe)Cl_2] with (NEt_4)[Fe{SCH_2CH_2)$_3$N}(CO)] in acetonitrile solvent.

Crystal data

$C_{10}H_{24}Cl_2NiP_2$
M_r = 335.84
Triclinic, $P\bar{1}$
a = 8.947 (2) Å
b = 14.082 (8) Å
c = 13.604 (2) Å
α = 91.51 (2)°
β = 98.18 (2)°
γ = 112.27 (2)°
V = 1564.0 (10) Å^3
Z = 4
D_x = 1.426 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10–11°
μ = 1.76 mm^{-1}
T = 150 (2) K
Block, orange
0.55 × 0.48 × 0.41 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω/θ scans
Absorption correction: ψ scan (*EMPABS*; Sheldrick *et al.*, 1977)
$T_{\min}$ = 0.395, $T_{\max}$ = 0.486
7878 measured reflections
7515 independent reflections
5564 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.011
$\theta_{\max}$ = 28.0°
$h = -11 \rightarrow 11$
$k = -18 \rightarrow 18$
$l = 0 \rightarrow 17$
3 standard reflections frequency: 167 min intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.089
S = 1.07
7514 reflections
358 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.034P)^2 + 0.317P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ = 0.001
$\Delta\rho_{\max}$ = 0.34 e Å^{-3}
$\Delta\rho_{\min}$ = −0.37 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—Cl1	2.2073 (12)	Ni2—Cl3	2.2035 (18)
Ni1—Cl2	2.2012 (8)	Ni2—Cl4	2.214 (2)
Ni1—P1	2.1372 (8)	Ni2—P3	2.139 (2)
Ni1—P2	2.1376 (12)	Ni2—P4	2.140 (2)
P1—C1	1.833 (3)	P3—C3	1.839 (4)
P1—C111	1.823 (3)	P3—C311	1.840 (5)
P1—C121	1.808 (3)	P3—C321	1.818 (4)
C1—C2	1.507 (4)	C3—C4	1.476 (6)
C2—P2	1.829 (3)	C4—P4	1.837 (4)
P2—C211	1.822 (3)	P4—C411	1.808 (6)
P2—C221	1.822 (3)	P4—C421	1.820 (5)
Cl2—Ni1—Cl1	95.24 (4)	Cl3—Ni2—Cl4	96.68 (11)
P1—Ni1—Cl1	87.71 (4)	P3—Ni2—Cl3	89.43 (8)
P2—Ni1—Cl1	174.57 (3)	P4—Ni2—Cl3	176.09 (13)
P1—Ni1—Cl2	174.44 (3)	P3—Ni2—Cl4	173.37 (12)
P2—Ni1—Cl2	89.51 (4)	P4—Ni2—Cl4	86.30 (12)
P1—Ni1—P2	87.78 (4)	P3—Ni2—P4	87.49 (10)
C1—P1—Ni1	111.96 (10)	C3—P3—Ni2	111.43 (14)
C2—C1—P1	111.48 (19)	C4—C3—P3	108.9 (2)
C1—C2—P2	112.39 (19)	C3—C4—P4	114.0 (3)
C2—P2—Ni1	111.28 (10)	C4—P4—Ni2	109.96 (18)
Ni1—P1—C1—C2	18.2 (3)	C3—C4—P4—Ni2	21.1 (4)
P1—C1—C2—P2	−24.5 (3)	Ni2*B*—P3*B*—C3*B*—C4*B*	−18.4 (14)
C1—C2—P2—Ni1	21.8 (3)	P3*B*—C3*B*—C4*B*—P4*B*	−5.2 (14)
Ni2—P3—C3—C4	28.6 (4)	C3*B*—C4*B*—P4*B*—Ni2*B*	27.5 (12)
P3—C3—C4—P4	−30.5 (5)		

Table 2
Table of weak hydrogen-bond interactions (Å, °).

	D—H	H···A	D···A	D—H···A
C1—H1*B*···Cl4$^{\rm i}$	0.99	2.784 (3)	3.716 (4)	157.2 (2)
C2—H2*B*···Cl4$^{\rm ii}$	0.99	2.793 (4)	3.683 (5)	149.8 (2)
H1*B*$^{\rm iii}$···Cl4···H2*B*$^{\rm ii}$	–	–	–	77.56 (9)

Symmetry codes: (i) $x, y-1, z$; (ii) $1-x, 1-y, -z$; (iii) $x, 1+y, z$.

The reflection ($\bar{1}20$) was found to be unreliable and was not used in the final refinement cycles. In the minor disordered molecule, with an occupancy factor of 0.167 (4), bond lengths were restrained to be effectively equivalent to those in the ordered molecule; the C atoms of the minor disorder component were refined isotropically. H atoms were included in idealized positions and set to ride on their parent atoms, with C—H distances of 0.99 and 0.98 A for ethyl and methyl C atoms, respectively; isotropic displacement parameters were set to be 1.2 and 1.5 times $U_{\rm eq}/U_{\rm iso}$(C), respectively.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1992); cell refinement: *CAD-4 EXPRESS*; data reduction: *CAD4* (Hursthouse, 1976); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

The authors thank the Biotechnology and Biological Sciences Research Council, UK, and the John Innes Foundation (SED) for financial support.

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