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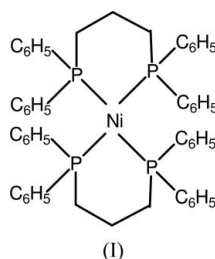
Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.036
 wR factor = 0.092
Data-to-parameter ratio = 17.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Bis[1,3-bis(diphenylphosphino)propane- κ^2P,P']-
nickel(0)

The neutral title complex, $[\text{Ni}(\text{C}_{27}\text{H}_{26}\text{P}_2)_2]$, contains a distorted tetrahedrally coordinated Ni^0 atom lying on a twofold rotation axis. The bridging propane groups of the ligands are twisted by approximately 90° with respect to each other.

Comment

As part of our ongoing studies to synthesize nickel compounds relevant to the structure and function of the microbial nickel-containing enzyme acetyl-CoA synthase, a range of new starting materials is required (Duff *et al.*, 2005; Evans, 2005). The title compound, (I), was obtained as a minor product from the attempted methylation of $[\text{NiCl}_2(\text{dppp})]$, where dppp is 1,3-bis(diphenylphosphino)propane. Nickel(0) diphosphine (diphos) compounds of the type $[\text{Ni}(\text{diphos})_2]$ were first prepared 45 years ago by Chatt & Hart (1960). Compound (I) has since been prepared by various methods (van Hecke & Horrocks, 1966; Giannoccaro & Vasapollo, 1983; Fisher & Alyea, 1989; Bricout *et al.*, 1995), but its structure has not been reported until now.



In (I), the Ni atom, which lies on a twofold rotation axis, displays a distorted tetrahedral coordination geometry; bond lengths to the P atoms are as expected, while the P–Ni–P angles range from $99.52(2)^\circ$ within one dppp ligand to $120.49(3)^\circ$ between the ligands (see Table 1). The ligands are arranged unsymmetrically about the Ni atom, with P–Ni–P angles between the ligands of $106.38(3)$ and $115.43(3)^\circ$. The P atoms are also distorted tetrahedral, with angles ranging from $98.58(9)$ to $122.21(6)^\circ$; the trend is for angles involving the Ni atom and the ligating phenyl C atoms to be the largest and the angles involving the bridging C atoms and the ligating phenyl C atoms to be the smallest. The P–C–C and C–C–C angles about the bridging C atoms are also distorted, being slightly larger than the ideal tetrahedral value (see Table 1). Dimensions within the phenyl rings are not unusual.

Each molecule is arranged with the bridging propane groups twisted by approximately 90° with respect to each other, the angle between the normals to the NiP_2 planes being $92.44(2)^\circ$. When viewed along the twofold rotation axis, three

Received 9 February 2005

Accepted 15 February 2005

Online 26 February 2005

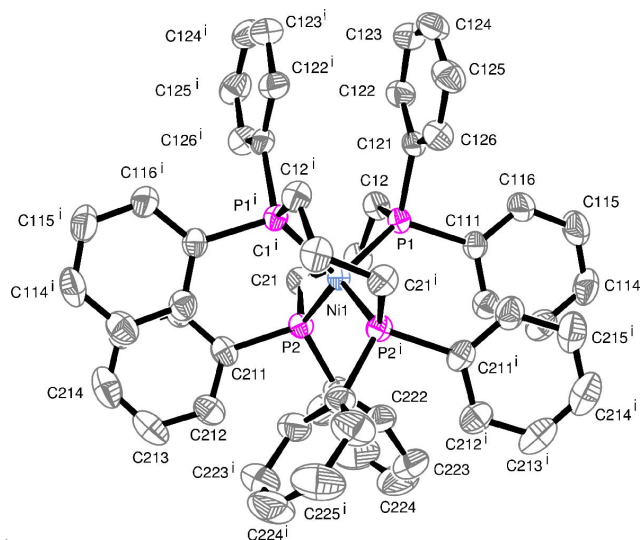


Figure 1
A view of (I), with displacement ellipsoids drawn at the 50% probability level. H atoms have been omitted for clarity. [Symmetry code: (i) $-x, y, \frac{1}{2} - z$.]

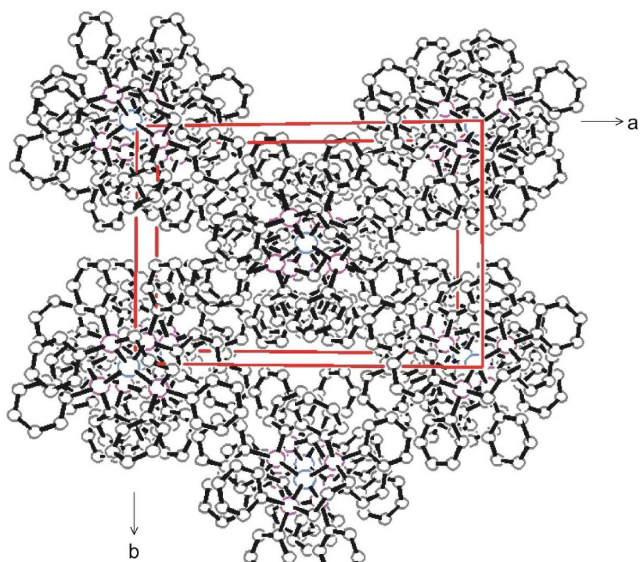


Figure 2
Packing diagram of (I), viewed along the crystallographic c axis. Atoms are represented by arbitrary spheres. H atoms have been omitted.

of the phenyl rings of each dppp ligand lie with their normals at approximately right angles to the crystallographic b axis. The normal to the fourth phenyl ring in each ligand is approximately parallel to the crystallographic b axis. The molecules are arranged in chains parallel to the crystallographic c axis, with four chains enclosing a channel parallel to the c axis with normal van der Waals contacts binding the molecules within and across the rows (see Fig. 2).

The structure of the related compound $[\text{Ni}(\text{dppe})_2]$, (II), where dppe is 1,2-bis(diphenylphosphino)ethane, has been reported previously (Hartung *et al.*, 1989). The distorted tetrahedral coordination about the Ni atom is similar in both molecules, with bond lengths to the Ni atom in (II) lying in the range 2.152 (3)–2.177 (3) Å. Bond angles are slightly different in the two, due to the steric effects of an ethyl rather than a

propyl bridging group; in complex (II), the P–Ni–P angles within ligands are 90.8 (1) and 90.1 (1)°, while interligand P–Ni–P angles lie between 113.8 (1) and 129.3 (1)°.

Experimental

To a stirred slurry of $[\text{NiCl}_2(\text{dppp})]$ (0.20 g, 0.37 mmol) in tetrahydrofuran (13 ml) was added a solution of methyl magnesium iodide in diethyl ether (3 M solution, 1.6 ml, 4.8 mmol) and additional tetrahydrofuran (20 ml). After 2 d, the mixture was filtered, and the filtrate was allowed to stand for two weeks; the solvent was then removed *in vacuo*. The solid residue was washed with diethyl ether to give a mixture of a brown solid and orange crystals of (I).

Crystal data

$[\text{Ni}(\text{C}_{27}\text{H}_{26}\text{P}_2)_2]$
 $M_r = 883.55$
 Monoclinic, $C2/c$
 $a = 18.303$ (6) Å
 $b = 13.212$ (2) Å
 $c = 20.023$ (4) Å
 $\beta = 109.67$ (2)°
 $V = 4559$ (2) Å³
 $Z = 4$

$D_x = 1.287$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 10$ –11°
 $\mu = 0.60$ mm⁻¹
 $T = 293$ (2) K
 Rhomb, orange
 $0.67 \times 0.52 \times 0.24$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 ω/θ scans
 Absorption correction: ψ scan (EMPABS; Sheldrick *et al.*, 1977)
 $T_{\text{min}} = 0.823, T_{\text{max}} = 0.865$
 7286 measured reflections
 6631 independent reflections
 4569 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.008$
 $\theta_{\text{max}} = 30^\circ$
 $h = -1 \rightarrow 25$
 $k = -1 \rightarrow 18$
 $l = -28 \rightarrow 28$
 3 standard reflections
 frequency: 167 min
 intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.092$
 $S = 1.07$
 6631 reflections
 371 parameters
 All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.033P)^2 + 0.7751P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.003$
 $\Delta\rho_{\text{max}} = 0.38 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

Ni1–P1	2.1752 (6)	P1–C111	1.8449 (18)
Ni1–P1 ⁱ	2.1753 (6)	P1–C12	1.8484 (19)
Ni1–P2	2.1790 (6)	P2–C21	1.8473 (19)
Ni1–P2 ⁱ	2.1790 (6)	P2–C211	1.8486 (19)
P1–C121	1.8413 (18)	P2–C221	1.8552 (19)
P1–Ni1–P1 ⁱ	106.38 (3)	C12–P1–Ni1	115.31 (7)
P1–Ni1–P2	99.52 (2)	C21–P2–C211	98.92 (9)
P1 ⁱ –Ni1–P2	115.43 (3)	C21–P2–C221	98.58 (9)
P1–Ni1–P2 ⁱ	115.43 (3)	C211–P2–C221	99.78 (9)
P1 ⁱ –Ni1–P2 ⁱ	99.51 (2)	C21–P2–Ni1	112.49 (7)
P2–Ni1–P2 ⁱ	120.49 (3)	C211–P2–Ni1	122.21 (6)
C121–P1–C111	100.36 (8)	C221–P2–Ni1	120.47 (7)
C121–P1–C12	100.74 (9)	C1–C12–P1	113.75 (14)
C111–P1–C12	98.71 (9)	C12–C1–C21	113.54 (17)
C121–P1–Ni1	118.59 (6)	C1–C21–P2	113.02 (13)
C111–P1–Ni1	119.59 (6)		
Ni1–P1–C12–C1	–46.88 (17)	C12–C1–C21–P2	–78.6 (2)
P1–C12–C1–C21	73.0 (2)	C1–C21–P2–Ni1	55.34 (16)

Symmetry code: (i) $-x, y, \frac{1}{2} - z$.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1992); cell refinement: *CAD-4 EXPRESS*; data reduction: *CAD-4 Processing Program* (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 BBSRC (UK) and the John Innes Foundation (SED) are thanked for financial support.

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