

Although Hg–N(1') bonding is weak in complexes of py₂CH₂ and pyz₂CH₂, ¹H NMR studies show that these ligands remain chelated to MeHg^{II} on dissolution of the [MeHgL]NO₃ complexes in methanol (Canty, Chaichit, Gatehouse, George & Hayhurst, 1981; Canty, 1980).

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The Structure of Bis(triethylphosphine)[η²-bis(trimethylsilyl)diphosphene]nickel, [(C₂H₅)₃P]₂Ni[PSi(CH₃)₃]₂, at 175 K, a Complex Containing a P–Ni–P Ring System*

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Abstract

The crystal structure of [(C₂H₅)₃P]₂Ni[PSi(CH₃)₃]₂, C₁₈H₄₈NiP₄Si₂, has been determined by three-dimensional X-ray diffraction data collected at 175 K. The compound crystallizes in the monoclinic space group *P2₁/c*, with *a* = 10.026 (5), *b* = 17.466 (9), *c* = 20.69 (1) Å, β = 127.45 (3)°, *Z* = 4. The structure was solved independently by *MULTAN* as well as by the heavy-atom method and refined by block-diagonal least-squares techniques. The final discrepancy factor *R* is 0.038 for 3719 symmetrical independent reflections

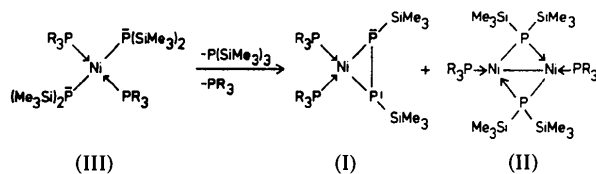
of the low-temperature measurement. In the complex the Ni atom is nearly planar coordinated by four P atoms. The Ni–P bond lengths to the P(C₂H₅)₃ groups are about 0.07 Å shorter than the Ni–P bond distances to the PSi(CH₃)₃ groups [2.236 (2) and 2.258 (2) Å]. The bond length between the P atoms of the PSi(CH₃)₃ groups [2.149 (2) Å] is significantly shorter than is expected for a normal P–P single bond.

Introduction

During our investigations concerning the ligand properties of the P(SiMe₃)₂ group towards transition metals (Schäfer, 1978, 1979*a, b*, 1980) we studied reactions of LiP(SiMe₃)₂ with (R₃P)₂NiCl₂ complexes. It was found that definite compounds could be isolated only if *R* is

* Parts of this work were presented as a poster contribution at the 19th Diskussionstagung der Arbeitsgemeinschaft Kristallographie at Aachen (Deppisch & Schäfer, 1979).

an alkyl group. The intermediately formed red complexes of the type $(R_3P)_2Ni[P(SiMe_3)_2]_2$ (III) decomposed at temperatures around 268 to 263 K in a complex condensation reaction (Schäfer, 1979b) yielding $(R_3P)_2Ni(PSiMe_3)_2$ (I) and $[R_3PNiP(SiMe_3)_2]_2$ (II).



From the reaction with the triethylphosphine complex ($R = C_2H_5$) dark violet crystals could be isolated, which are extremely air-sensitive. The structure of compound (I) was determined to ascertain the correct constitution of the molecule and the coordination pattern of the Ni atom. Of special interest was the type of bonding within the PNiP ring system. The structure of compound (II) will be described later.

Experimental

For the X-ray investigations some crystals were sealed in quartz capillaries filled with argon. The space group and preliminary unit-cell dimensions were determined by Weissenberg and precession photographs. Systematic absences were $h0l$ for l odd and $0k0$ for k odd indicating the space group $P2_1/c$. The cell parameters were determined by a least-squares fit to the angular settings of 16 carefully centered reflections. The Picker diffractometer FACS-I, which was also used for the intensity measurements, was equipped with a graphite monochromator and a scintillation detector and Mo $K\alpha$ radiation was used ($\lambda = 0.71073 \text{ \AA}$). We used the θ - 2θ scan technique with stationary counter for the measurement of the background at each limit of the scan. The scan range was varied according to the $\bar{\alpha}_1$ - α_2 dispersion. The cooling for the measurement at 175 K was carried out with an N_2 -gas stream, the relative position of which was always opposite the crystal. Three standard reflections were measured after every 100 reflections. Their intensities decreased about 5% during the data collection. We collected 4243 reflections within the range $3.5 \leq 2\theta \leq 45^\circ$, the intensities of which were scaled according to the decay of the standard reflections. The intensities were corrected for Lorentz and polarization effects, but no absorption correction was applied ($\mu = 0.98 \text{ mm}^{-1}$).

Structure determination and refinement

For the first steps of structure determination the intensities of the measurement at room temperature were used. The position of the Ni atom was derived

from a Patterson map. The following Fourier syntheses showed only a few well identified new atoms with very different peak heights, one nearly as high as the maximum of the Ni atom. Thus the structure model could be completed only rather slowly.

Simultaneously we had used the *MULTAN* program system (Germain, Main & Woolfson, 1971). The *E* map based on the set of signs for the 200 largest *E* values with the best combined figure of merit revealed all non-carbon and non-hydrogen atoms. The heights of the first five peaks were nearly equal. The following ΔF syntheses gave the positions of all non-hydrogen atoms. The atomic positions were the same as those found with the heavy-atom method. The model was further refined with anisotropic temperature factors.

Only a few of the H atoms could be located in a ΔF map which was calculated with the room-temperature data. Therefore, we introduced the low-temperature data. All H atoms were now found and could be refined with isotropic temperature factors. During the last cycles of the block-diagonal least-squares refinement an isotropic extinction parameter was refined (Larson, 1967) with the XRAY 76 system (Stewart, 1976). The scattering factors were those of Cromer & Mann (1968) for the nonhydrogen atoms with a dispersion correction for the Ni atom ($f' = 0.285$, $f'' = 1.113$) taken from *International Tables for X-ray Crystallography* (1974). The scattering factors of the H

Table 1. Final atomic parameters of the nonhydrogen atoms

The positional parameters are multiplied by 10^4 , the equivalent isotropic temperature factors (\AA^2) by 10^4 for Ni, P and Si, by 10^3 for C. E.s.d.'s are given in parentheses. $U_{eq} = \frac{1}{3} \sum_{i=1}^3 U_{ii}$ with orthogonal U_{ii} .

	x	y	z	U_{eq}
Ni	5768.2 (0.7)	7389.9 (0.3)	7592.7 (0.3)	217 (3)
P(1)	5803 (2)	6257 (1)	8039 (1)	291 (7)
P(2)	7894 (1)	7938 (1)	8753 (1)	220 (6)
P(3)	6493 (1)	8630 (1)	7672 (1)	220 (6)
P(4)	3699 (1)	7349 (1)	6294 (1)	266 (6)
Si(1)	6905 (2)	8456 (1)	9383 (1)	249 (7)
Si(2)	8441 (2)	8654 (1)	7454 (1)	257 (7)
C(1)	1790 (9)	4427 (4)	2055 (5)	58 (4)
C(2)	6432 (6)	5480 (3)	7685 (3)	35 (3)
C(3)	7289 (12)	5317 (4)	9475 (5)	63 (5)
C(4)	2780 (7)	3889 (3)	852 (3)	46 (3)
C(5)	3074 (10)	6374 (4)	8094 (5)	60 (5)
C(6)	3781 (8)	9099 (3)	2743 (4)	41 (4)
C(7)	4466 (8)	6197 (4)	5611 (4)	49 (4)
C(8)	6864 (7)	3547 (3)	4283 (3)	38 (3)
C(9)	8423 (7)	1625 (3)	3661 (4)	46 (4)
C(10)	1674 (6)	7423 (3)	1080 (3)	32 (3)
C(11)	7646 (8)	3050 (4)	256 (3)	60 (4)
C(12)	6171 (6)	1972 (3)	4345 (3)	39 (3)
C(13)	5337 (7)	1245 (3)	1282 (3)	36 (3)
C(14)	7177 (8)	7801 (3)	10178 (3)	40 (3)
C(15)	1733 (8)	688 (4)	74 (4)	51 (4)
C(16)	747 (8)	2307 (3)	2547 (4)	44 (3)
C(17)	2392 (7)	860 (3)	3538 (3)	36 (3)
C(18)	213 (7)	5744 (4)	3274 (4)	44 (3)

Table 2. Final atomic parameters of the H atoms

E.s.d.'s are given in parentheses, the coordinates are multiplied by 10^3 .

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ($\text{\AA}^2 \times 10^3$)
H(11)	837 (7)	891 (3)	278 (3)	6 (2)
H(12)	895 (8)	946 (4)	346 (4)	7 (2)
H(13)	160 (7)	484 (3)	231 (3)	7 (2)
H(21)	431 (5)	452 (2)	287 (2)	3 (1)
H(22)	379 (6)	500 (3)	218 (3)	4 (1)
H(31)	386 (9)	483 (4)	74 (4)	9 (3)
H(32)	243 (9)	502 (4)	73 (4)	9 (3)
H(33)	815 (8)	969 (3)	511 (4)	7 (2)
H(41)	320 (6)	357 (3)	71 (3)	6 (2)
H(42)	166 (6)	371 (3)	66 (3)	4 (1)
H(51)	815 (8)	379 (3)	221 (4)	7 (2)
H(52)	713 (7)	313 (4)	214 (4)	7 (2)
H(53)	389 (8)	867 (4)	374 (4)	8 (2)
H(61)	691 (6)	410 (3)	280 (3)	3 (1)
H(62)	387 (6)	960 (3)	288 (3)	5 (2)
H(71)	567 (7)	433 (4)	455 (3)	6 (2)
H(72)	442 (8)	848 (4)	25 (4)	7 (2)
H(73)	580 (7)	878 (3)	119 (3)	5 (1)
H(81)	201 (6)	849 (3)	18 (3)	4 (1)
H(82)	695 (6)	396 (3)	399 (3)	4 (1)
H(91)	957 (6)	154 (3)	382 (3)	4 (1)
H(92)	740 (8)	152 (4)	306 (4)	8 (2)
H(93)	164 (7)	624 (3)	103 (4)	6 (2)
H(101)	82 (6)	750 (3)	52 (3)	3 (1)
H(102)	849 (6)	284 (3)	370 (3)	4 (1)
H(111)	259 (7)	849 (4)	452 (4)	7 (2)
H(112)	867 (8)	313 (3)	38 (4)	6 (2)
H(113)	770 (8)	254 (4)	47 (4)	10 (2)
H(121)	513 (8)	708 (4)	83 (4)	8 (2)
H(122)	406 (6)	646 (3)	92 (3)	5 (1)
H(131)	611 (7)	166 (3)	150 (3)	6 (2)
H(132)	572 (7)	100 (3)	174 (4)	6 (2)
H(133)	455 (7)	595 (3)	407 (3)	6 (2)
H(141)	717 (7)	817 (3)	57 (4)	7 (2)
H(142)	612 (7)	757 (3)	489 (3)	6 (2)
H(143)	823 (7)	756 (3)	50 (3)	5 (2)
H(151)	948 (7)	915 (3)	32 (3)	6 (2)
H(152)	797 (8)	960 (4)	20 (4)	7 (2)
H(153)	204 (9)	34 (4)	51 (5)	10 (2)
H(161)	30 (8)	256 (4)	209 (4)	8 (2)
H(162)	963 (10)	226 (4)	246 (4)	11 (3)
H(163)	822 (10)	762 (4)	196 (5)	11 (3)
H(171)	845 (8)	573 (3)	146 (4)	6 (2)
H(172)	711 (7)	542 (4)	141 (4)	6 (2)
H(173)	670 (8)	619 (4)	96 (4)	7 (2)
H(181)	934 (7)	97 (3)	124 (4)	5 (2)
H(182)	896 (8)	72 (4)	180 (4)	8 (2)
H(183)	21 (7)	22 (4)	179 (3)	6 (2)

atoms were those of Stewart, Davidson & Simpson (1965). The refinement was finished at an *R* value of 0.038 ($R_w = 0.043$, $\omega = 1$) for 3719 reflections, of which 377 reflections were classified as unobserved [$I < 3\sigma(I)$]. All parameter shifts were smaller than 0.05 times the corresponding error. Final atomic coordinates are given in Tables 1 and 2.*

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36304 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Description and discussion of the structure

Fig. 1 shows a perspective drawing of the molecule with the labeling of the atoms. Fig. 2 is a stereoscopic plot of the molecule with the H atoms. The bond distances of the nonhydrogen atoms are given in Table 3 and Fig. 3, the main bond angles in Table 4 and Fig. 3.

The bonding features of the inner coordination of the Ni atom are the main interest in this molecule (Fig. 3). As far as we know this structure is the first example of a molecule showing a bond between a Ni atom and a three-coordinated P atom. The Ni atom is nearly planar coordinated by four P atoms. P(1) lies 0.13 (1) Å out of the plane formed by Ni, P(2) and P(3), P(4) lies 0.03 (1) Å below this plane.

The Ni—P bond lengths of the tetrahedrally coordinated atoms P(1) and P(4) are 2.175 (2) and 2.179 (2) Å. The bond distances to P(2) and P(3), 2.236 (2) and 2.258 (2) Å, are significantly longer and agree rather well with the sum of the covalent radii (2.26 Å) for a square coordination of nickel as derived by Kilburn & Powell (1970). But also the shorter bond lengths to P(1) and P(4) are not unusual. Dreissig & Dietrich (1968) found 2.13 (3) and 2.18 (3) Å in (ethylene)bis(triphenylphosphine)nickel (*a*), Guggenberger (1973) found 2.121 (4) and 2.096 (4) Å in (acrylonitrile)bis(tri-*o*-tolyl phosphite)nickel and an average length of 2.095 (5) Å in (ethylene)bis(tri-*o*-tolyl phosphite)nickel (*b*), and Churchill & O'Brien

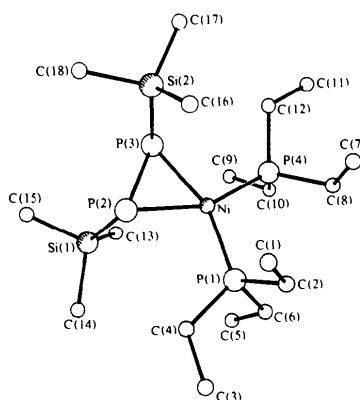


Fig. 1. Perspective drawing of the molecule with the labeling of the nonhydrogen atoms.

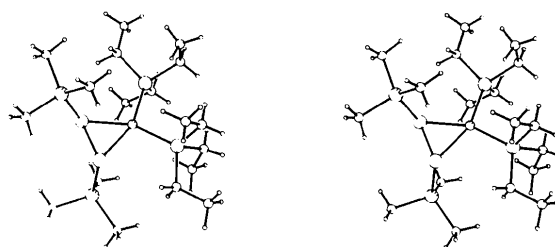


Fig. 2. Stereoscopic view of the molecule with hydrogen atoms.

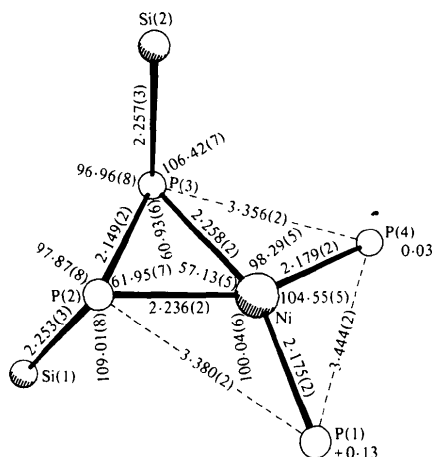


Fig. 3. Inner coordination sphere of $C_{18}H_{48}NiP_4Si_2$. (Distances in Å, angles in deg.)

Table 3. The main bond lengths (not shown in Fig. 3) of $C_{18}H_{48}NiP_4Si_2$ in Å

E.s.d.'s are given in parentheses.

P(1)–C(2)	1.828 (7)	C(1)–C(2)	1.525 (11)
P(1)–C(4)	1.839 (6)	C(3)–C(4)	1.526 (9)
P(1)–C(6)	1.834 (8)	C(5)–C(6)	1.527 (15)
P(4)–C(8)	1.836 (6)	C(7)–C(8)	1.543 (13)
P(4)–C(10)	1.840 (7)	C(9)–C(10)	1.517 (9)
P(4)–C(12)	1.839 (7)	C(11)–C(12)	1.534 (6)
Si(1)–C(13)	1.859 (6)	Si(2)–C(16)	1.866 (7)
Si(1)–C(14)	1.883 (7)	Si(2)–C(17)	1.883 (7)
Si(1)–C(15)	1.870 (6)	Si(2)–C(18)	1.865 (6)

Table 4. The main bond angles ($^\circ$) of $C_{18}H_{48}NiP_4Si_2$

E.s.d.'s are given in parentheses.

Ni–P(1)–C(2)	115.1 (2)	C(2)–P(1)–C(4)	101.8 (3)
Ni–P(1)–C(4)	117.2 (2)	C(2)–P(1)–C(6)	101.3 (3)
Ni–P(1)–C(6)	116.0 (2)	C(4)–P(1)–C(6)	103.2 (4)
Ni–P(4)–C(8)	121.0 (2)	C(8)–P(4)–C(10)	99.8 (3)
Ni–P(4)–C(10)	111.9 (2)	C(8)–P(4)–C(12)	102.1 (3)
Ni–P(4)–C(12)	115.8 (1)	C(10)–P(4)–C(12)	103.7 (3)
P(1)–C(2)–C(1)	113.2 (4)	P(4)–C(8)–C(7)	112.9 (4)
P(1)–C(4)–C(3)	118.1 (4)	P(4)–C(10)–C(9)	114.3 (4)
P(1)–C(6)–C(5)	113.3 (4)	P(4)–C(12)–C(11)	117.6 (5)

(1970) found 2.148 (8) and 2.172 (9) Å in (η^5 -cyclopentadienyl)(σ -trifluoromethyl)(triphenylphosphine)-nickel. Though a whole series of nickel–phosphorus complexes have been studied so far using X-ray techniques the factors influencing the Ni–P bond lengths such as ligands and coordination are not yet fully understood (Corbridge, 1974).

The bond length between the two three-coordinated P atoms, P(2)–P(3) = 2.149 (2) Å, is significantly shorter than the average value of 2.22 Å for a covalent P–P single bond (Corbridge, 1978), but also much longer than the estimated length of the hypothetical

double bond with approximately 2.00 Å (Corbridge, 1974; Campana, Vizi-Orosz, Palyi, Marko & Dahl, 1979). Similar short bond lengths between three-coordinated P atoms were found in *cis*-tetracarboxyl(hexamethyltetraphosphane- P^1, P^4)molybdenum(0) with 2.14 (1) Å (Sheldrick, 1975), in (η^5 - C_5H_5) $_2$ MoP $_2$ H $_2$ (c) with 2.146 (3) Å (Cannillo, Coda, Prout & Daran, 1977), and in $[P(C_6H_5)_3]_2Pt(PC_6F_5)_2$ (d) with 2.156 (7) Å (Elmes, Scudder & West, 1976).

The bis(cyclopentadienyl)molybdenum complex (c) and the platinum complex (d) are the only other examples known to us so far showing a (PR) $_2$ unit coordinated to a transition metal. The close similarity of the P–P bond distances in these molecules and (I) seems to support the assumption that the bonding situations within these metal–P $_2$ ring systems are closely related, though the positions (*cis* or *trans*) of the H atoms in (c) could not be determined beyond any doubt and the structure model of (c) could not be refined without constraints.

Some transition-metal complexes in which a P $_3$ ring system forms a ligand show comparable short P–P linkages of about 2.14 Å (Cecconi, Dapporto, Midollini & Sacconi, 1978; Di Vaira, Ghilardi, Midollini & Sacconi, 1978; Midollini, Orlandini & Sacconi, 1979). Campana *et al.* (1979) found a remarkably short P–P bond of 2.019 (9) Å within the P $_2$ ligand of Co $_2$ (CO) $_5$ [P(C $_6$ H $_5$) $_3$](μ -P $_2$). An analogous shortening of the As–As single bond is observed in (CO) $_4$ Fe(AsPh) $_2$ (e), with an As–As distance of 2.365 (2) Å, which has to be compared with a normal As–As single bond of 2.43–2.46 Å (Jacob & Weiss, 1978), and in (CO) $_4$ Fe(AsC $_6$ F $_5$) $_2$ (f) with 2.388 (7) Å (Elmes, Leverett & West, 1971).

On the other hand, in three-membered-ring compounds in which boron, silicon or germanium are linked to two P atoms the P–P bonds show a 'normal' length with a mean value of 2.226 Å (Feher, Fröhlich & Tebbe, 1980; Tebbe, 1980).

When we compare these data it looks as though the shrinkage of P–P bonds occurs if these units are coordinated to transition metals. The bonding situation within the three-membered NiP $_2$ ring in (I) can be compared to transition-metal ethylene complexes such as (a). This would mean that the so far unknown species Me $_3$ Si–P=P–SiMe $_3$ is coordinated by a synergistic donor–acceptor bond to the Ni atom and that the 'P=P bond' is lengthened by strong back bonding. But if we assume strong back bonding the overall electron distribution would be the same as in a three-membered ring with two Ni–P σ bonds as is discussed for ethylene complexes (Dewar & Ford, 1979). The shortening of the P–P bond within a normal three-membered ring may then be explained assuming only weak overlap between the Ni and P orbitals [see bond distances of Ni to P(2) 2.236 (2) Å and to P(3) 2.258 (2) Å], which would result in a

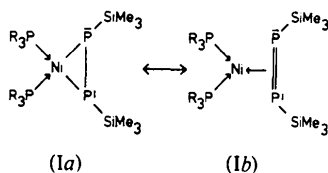
strengthening of the P–P bond in a similar way to that shown for ethylene complexes (Dewar & Ford, 1979).

The $Si(Me)_3$ groups bonded to P(2) and P(3) adopt the *trans* position in contrast to the assumed position in (c), but according to the phenyl groups in (d), (e) and (f) with a torsion angle $Si(1)-P(2)-P(3)-Si(2)$ of $147.7(2)^\circ$.

The other bond lengths in the molecule are quite normal. The P–C bonds have $1.836(6)$ Å, the P–Si bonds $2.255(2)$ Å as mean values. The C–H bonds range from 0.86 to 1.13 Å with a mean value of $0.97(7)$ Å.

Conclusion

The observed shortening of the P–P bond may be interpreted in a similar way to the C–C bond lengths in ethylene complexes such as (a) and (b) as well as the As–As bond length in (e) and (f). Therefore, we think that the bonding feature of the $PNiP$ group is best explained as an intermediate state between the two mesomeric forms (Ia) and (Ib).



All calculations were performed on a Univac 1108 computer at the Rechenzentrum der Universität Karlsruhe. The structure plots were made with the *SHELXTL* (Sheldrick, 1979) program. We wish to thank Mr Mattern for his technical assistance.

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