Journal of Organometallic Chemistry, 258 (1983) 373–381
Elsevier Sequoia S.A., Lausanne – Printed in The Netherlands

THE P_4Se_3 CAGE MOLECULE AS A LIGAND. CRYSTAL STRUCTURE OF $[\{N(CH_2CH_2PPh_2)_3\}Ni(P_4Se_3)]\cdot 2C_6H_6$

MASSIMO DI VAIRA, MAURIZIO PERUZZINI and PIERO STOPPIONI

Dipartimento di Chimica, Università di Firenze and Istituto I.S.S.E.C.C., C.N.R., Via J. Nardi, 39, 50132 Firenze (Italy)

(Received July 13th, 1983)

Summary

The structure of the compound $[(np_3)Ni(P_4Se_3)]\cdot 2C_6H_6$, obtained from reaction of the nickel(0) complex $(np_3)Ni$ (np_3 = tris(2-diphenylphosphinoethyl)amine) with tetraphosphorus triselenide, P_4Se_3, has been determined by X-ray diffraction studies. Crystal data: cubic, space group $P2_13$, a 17.413(7) Å, $Z = 4$; final $R = 0.050$. The intact P_4Se_3 entity is coordinated to the metal through the apical phosphorus atom. The small changes occurring in the geometry of the cage molecule upon coordination are analyzed by a comparison with the structure of uncoordinated P_4Se_3, which has been refined to $R = 0.045$.

Introduction

We recently found that the P_4S_3 and P_4Se_3 cage molecules react with d^{10} metal complexes formed by the tetradentate tripod ligand tris(2-diphenylphosphinoethyl)amine, np_3, yielding compounds of formula $[(np_3)M(P_4X_3)]\cdot nC_6H_6$ ($M = Ni$, $X = S$ or Se, $n = 2$; $M = Pd$, $X = S$, $n = 0$) [1]. An X-ray structural investigation of the compound $[(np_3)Ni(P_4S_3)]\cdot 2C_6H_6$ showed that the intact P_4S_3 entity is coordinated to the metal through its apical P atom [1]. It was of interest to obtain detailed structural information also on the isomorphous Se derivative [1], because the P_4Se_3 molecule had never been used before as a ligand for transition metal complexes. However, this study was prevented for some time by the bad quality of crystals. Suitable crystals were recently obtained for $[(np_3)Ni(P_4Se_3)]\cdot 2C_6H_6$, the structure of which is reported here. In addition, refinement of the structure of the isolated P_4Se_3 molecule, determined several years ago from two-dimensional film data [2], was undertaken in order to allow detailed comparisons between the coordinated and uncoordinated cage molecule.

0022-328X/83/$03.00

Experimental

Crystals of the compound $[(np_3)Ni(P_4Se_3)] \cdot 2C_6H_6$ suitable for X-ray investigation were obtained as previously described [1], except that the temperature was maintained at ca. 0°C. All operations were performed under nitrogen, but no N_2 was bubbled through the solution. Tetraphosphorus triselenide, prepared by the published procedure [3], was recrystallized from benzene under an inert atmosphere.

X-ray data collection

A crystal of the compound $[(np_3)Ni(P_4Se_3)] \cdot 2C_6H_6$ of a truncated tetrahedron shape with a 0.40 mm edge was used for the intensity data collection. A Philips PW 1100 automated diffractometer and graphite-monochromated Cu-K_α radiation were used. The lattice constant as determined at 22°C from refinement of the setting angles of 24 reflections with $24° < \theta < 31°$ is given in Table 1, which lists details of the experimental procedure and structure refinement. The intensities of the symmetry-independent reflections were measured, stationary background counts being taken at each end of the asymmetric scan for a time equal to half the scan time. The intensities of three standard reflections were monitored every 120 min, and showed a smooth decay of ca. 5%. The raw intensity data were corrected for such decay as well as for Lorentz-polarization effects and for absorption (transmission coefficients ranging from 0.44 to 0.49). The main computer program used in the crystallographic calculations is specified in ref. 4.

The crystal of P_4Se_3 used for data collection had dimensions 0.06 × 0.15 × 0.50

TABLE 1

CRYSTAL DATA AND SUMMARY OF INTENSITY DATA COLLECTION AND STRUCTURE REFINEMENT

	$[(np_3)Ni(P_4Se_3)] \cdot 2C_6H_6$	P_4Se_3
Formula	$C_{54}H_{54}NNiP_7Se_3$	P_4Se_3
Formula wt.	1229.45	360.78
Space group	$P2_13$	*Pbnm*
Cell constants (Å)	*a* 17.413(7)	*a* 26.278(12) *b* 11.816(7) *c* 9.746(5)
V (Å^3)	5279.8	3026.1
Z	4	16
ρ_{calcd} (g cm^{-3})	1.546	3.166
Radiation (Å)	Cu-K_α 1.5418	Mo-K_α 0.71069
μ (cm^{-1})	52.96	151.6
Scan type	θ–2θ	θ–2θ
Scan width (°)	$1.0 + 0.3 \tan\theta$	$1.2 + 0.3 \tan\theta$
Scan speed (° min^{-1})	5	5
$2\theta_{max}$ (°)	110	50
Unique data measured	1369	3042
Observed data with $I > 3\sigma(I)$	974	1046
Number of variables	107	145
R factors	$R = 0.050$ [a], $R_w = 0.050$ [b]	$R = 0.045$ [a], $R_w = 0.037$ [b]

[a] $R = \Sigma||F_0| - |F_c||/\Sigma|F_0|$. [b] $R_w = [\Sigma w(|F_0| - |F_c|)^2/\Sigma w|F_0|^2]^{1/2}$.

mm. Graphite-monochromated Mo-K_α radiation was used. Cell parameters were determined at 22°C by least-squares fit of 24 reflections with $15° < \theta < 19°$. A setting (space group *Pbnm*) different from that used previously (space group *Pmnb*) [2] was adopted, both settings being alternatives to the standard one (space group *Pnma*, No. 62 in ref. 5). The data collection procedure was similar to that followed for the nickel compound; details are given in Table 1. The correction for absorption was performed by an empirical procedure [6] based on measurement of intensities during ψ scans [7].

Solution and refinement of the structures

The initial positions for the non-hydrogen atoms in the structure of $[(np_3)Ni(P_4Se_3)] \cdot 2C_6H_6$ were obtained from the structure of the isomorphous sulfur derivative [1]. The symmetry-independent part of the structure is formed by one third of the formula unit. In the full-matrix least-squares refinement the function $\Sigma w(|F_0| - |F_c|)^2$ was minimized with weights $w = 1/\sigma^2(F_0)$. Anisotropic thermal parameters were used for atoms heavier than carbon and isotropic parameters for the carbon atoms, overall values being applied within each benzene solvate molecule. Hydrogen atoms were introduced in calculated positions (C–H 1.00 Å) each with a temperature factor ca. 20% larger than that of the corresponding carbon atom. The scattering factors for the neutral atoms and anomalous dispersion corrections for Ni and Se were taken from ref. 8. Convergence was attained at $R = 0.050$. The largest shift/error ratio in the last cycle was 0.02. Peaks in the final ΔF map were all lower than 0.5 eÅ^{-3} and could not be assigned any chemical significance. Final positional and thermal parameters for the non-hydrogen atoms in $[(np_3)Ni(P_4Se_3)] \cdot 2C_6H_6$ are listed in Table 2.

The structure of P_4Se_3 was refined with input coordinates from the previous work [2], taking account of the different setting. There are four symmetry-independent molecules in the structure, each lying in special position *m*. Anisotropic thermal parameters were used for all atoms and the refinement converged at $R = 0.045$. The largest shift/error ratio in the final cycle was 0.06 and the highest peak in the final ΔF map was ca. 1.0 eÅ^{-3}. Final positional parameters with atomic labels corresponding with those in ref. 2, and thermal parameters are listed in Table 3. Listings of hydrogen atom coordinates for $[(np_3)Ni(P_4Se_3)] \cdot 2C_6H_6$ and of observed and calculated structure factors for both structures are available from the authors on request.

Results and discussion

The main properties of the compound $[(np_3)Ni(P_4Se_3)] \cdot 2C_6H_6$ have already been reported [1]. Its structure, which is closely similar to that of the isomorphous sulfur derivative [1], is formed by isolated $[(np_3)Ni(P_4Se_3)]$ and benzene molecules. All of these molecules lie in special positions of 3 symmetry; in the molecule of the nickel complex a threefold axis passes through the metal atom, the nitrogen atom of the tripod ligand, which is uncoordinated, and the apical phosphorus atom of the P_4Se_3 unit, which is bound to the metal atom. A view of the $[(np_3)Ni(P_4Se_3)]$ molecule is shown in Fig. 1. Values of bond lengths and angles are listed in Table 4.

The metal atom is coordinated by the three phosphorus atoms of the np_3 ligand

(Continued on p. 378)

TABLE 2

ATOMIC PARAMETERS FOR [(np$_3$)Ni(P$_4$Se$_3$)]·2C$_6$H$_6$ [a]

Fractional coordinates ($\times 10^4$) and isotropic thermal parameters ($\times 10^3$) [b]

Atom	x/a	y/b	z/c	U (Å^2)	Atom	x/a	y/b	z/c	U (Å^2)
Ni [c]	349(1)	349(1)	349(1)		C(15)	1268(9)	−2482(8)	1758(8)	65(3)
Se	−1547(1)	−668(1)	47(1)		C(16)	1254(8)	−1661(8)	1737(8)	62(3)
P(1)	519(2)	−207(2)	1500(2)		C(21)	−207(7)	−89(7)	2247(7)	45(2)
P(2) [c]	−339(2)	−339(2)	−339(2)		C(22)	−927(7)	157(7)	2064(7)	45(2)
P(3)	−1960(2)	−1396(2)	−929(3)		C(23)	−1510(8)	200(8)	2618(7)	59(3)
N [c]	1385(6)	1385(6)	1385(6)		C(24)	−1380(8)	−15(8)	3359(8)	71(3)
C(1)	1402(7)	115(7)	2003(7)	50(2)	C(25)	−663(9)	−250(9)	3559(10)	82(3)
C(2)	1431(8)	978(7)	2133(8)	55(3)	C(26)	−61(9)	−317(8)	3024(8)	64(3)
C(11)	579(7)	−1272(7)	1562(7)	40(2)	C(31)	3382(11)	2290(11)	2711(11)	92(3)
C(12)	−40(7)	−1695(7)	1415(7)	45(2)	C(32)	2862(11)	2211(10)	3309(10)	92(3)
C(13)	−55(9)	−2497(8)	1456(8)	66(3)	C(41)	4451(18)	3626(15)	4537(17)	174(3)
C(14)	645(9)	−2867(9)	1597(8)	71(3)	C(42)	3798(19)	3947(18)	4774(15)	174(3)

Anisotropic thermal parameters ($\times 10^3$) [d]

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ni [c]	52(1)	52(1)	52(1)	−1(1)	−1(1)	−1(1)
Se	44(1)	59(1)	60(1)	−5(1)	10(1)	1(1)
P(1)	37(2)	37(2)	33(2)	2(1)	0(1)	1(1)
P(2) [c]	32(1)	32(1)	32(1)	0(1)	0(1)	0(1)
P(3)	58(2)	73(2)	88(2)	−29(2)	−14(2)	−9(2)
N [c]	60(3)	60(3)	60(3)	−1(3)	−1(3)	−1(3)

[a] Atoms C(31)–C(42) belong to the two benzene solvate molecules. [b] Isotropic temperature factors are of the form $\exp[-8\pi^2 U(\sin^2\theta)/\lambda^2]$. [c] Atom in special position of 3 symmetry. [d] Anisotropic temperature factors are of the form $\exp[-2\pi^2(U_{11}h^2a^{*2} + \ldots + 2U_{12}hka^*b^* + \ldots)]$.

TABLE 3

FRACTIONAL ATOMIC COORDINATES[a] ($\times 10^4$) AND THERMAL PARAMETERS[b] ($\times 10^3$) FOR P_4Se_3

Mol.	Atom	x/a	y/b	z/c	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
I	P(1)	1272(3)	870(8)	2500	24(6)	105(8)	50(6)	12(5)	0	0
	P(2)	889(2)	2285(4)	3644(5)	52(4)	57(4)	33(3)	−24(3)	−4(3)	−5(3)
	P(4)	−23(3)	430(6)	2500	31(5)	40(5)	55(6)	−4(4)	0	0
	Se(1)	712(1)	−549(2)	2500	52(3)	46(2)	47(2)	22(2)	0	0
	Se(2)	137(1)	1606(2)	4262(2)	46(1)	44(1)	44(1)	−4(1)	18(1)	−7(1)
II	P(1)	2170(3)	324(6)	7500	60(6)	30(5)	40(5)	−17(4)	0	0
	P(2)	1437(2)	327(4)	8642(5)	65(4)	34(3)	37(3)	10(3)	2(3)	−9(3)
	P(4)	1672(3)	−2376(5)	7500	29(5)	22(4)	43(5)	0(4)	0	0
	Se(1)	2430(1)	−1479(3)	7500	24(2)	50(2)	43(2)	−2(2)	0	0
	Se(2)	1299(1)	−1469(2)	9257(2)	35(1)	42(1)	35(1)	4(1)	7(1)	10(1)
III	P(1)	3235(4)	−516(7)	2500	67(7)	46(6)	64(7)	33(5)	0	0
	P(2)	2526(2)	−94(4)	3640(5)	67(4)	35(3)	30(3)	−13(3)	5(4)	4(3)
	P(4)	3081(3)	2370(6)	2500	51(6)	37(5)	44(5)	−8(4)	0	0
	Se(1)	3699(1)	1069(3)	2500	25(2)	106(3)	53(2)	1(2)	0	0
	Se(2)	2612(1)	1715(2)	4259(2)	50(2)	43(1)	36(1)	−4(1)	12(1)	−13(1)
IV	P(1)	3967(4)	−1888(6)	7500	84(7)	28(5)	53(6)	−12(5)	0	0
	P(2)	3666(2)	−402(4)	8639(5)	35(3)	54(3)	39(3)	−15(3)	8(3)	−4(3)
	P(4)	4876(3)	232(7)	7500	34(5)	54(6)	42(5)	−4(4)	0	0
	Se(1)	4816(1)	−1681(3)	7500	74(3)	46(2)	51(2)	30(2)	0	0
	Se(2)	4351(1)	603(2)	9262(2)	36(1)	47(1)	43(1)	−1(1)	−2(1)	−17(1)

[a] Atomic labels as in ref. 2. The numerals I–IV refer to the four independent molecules in the structure of P_4Se_3. Atoms P(1), P(4), and Se(1) in I–IV lie in special position *m*. [b] Anisotropic temperature factors are of the form $\exp[-2\pi^2(U_{11}h^2a^{*2} + \ldots + 2U_{12}hka^*b^* + \ldots)]$.

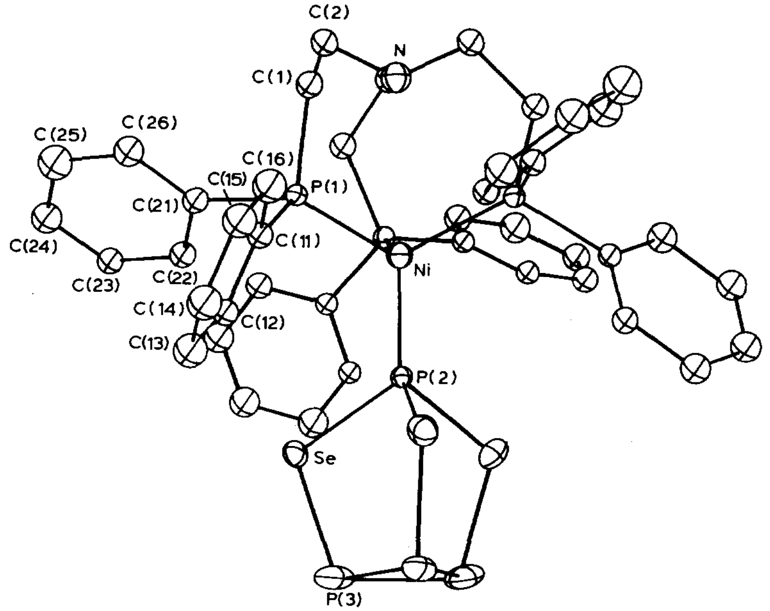

Fig. 1. Perspective view of the $[(np_3)Ni(P_4Se_3)]$ molecule with 20% probability ellipsoids. Symmetry-independent atoms are labeled.

and by the apical phosphorus atom of the P_4Se_3 unit, which form a distorted tetrahedral environment. The Ni–P(np_3) and Ni–P(P_4Se_3) distances (2.246(3), 2.075(5) Å) match those in the P_4S_3 derivative [1] (2.245(3), 2.072(5) Å). Although the values of the P(np_3)–Ni–P(np_3) and P(np_3)–Ni–P(P_4Se_3) angles (108.9(1)°, 110.1(1)°) are similar to those of the corresponding angles in the P_4S_3 derivative (108.5(1)°, 110.4(1)°), the former are closer to the tetrahedral value, indicating a smaller elongation [9] of the np_3 ligand in the P_4Se_3 adduct. As a consequence, the distance of the uncoordinated nitrogen atom from the metal is shorter in the P_4Se_3 derivative (3.124(8) Å) than in the P_4S_3 one (3.166(8) Å). Such small differences

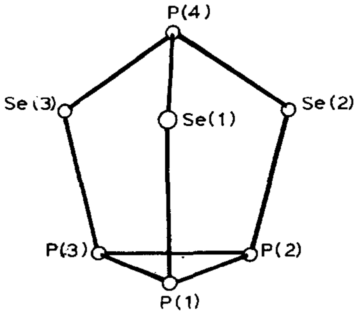

Fig. 2. Schematic representation of the P_4Se_3 molecule with labeling consistent with that in ref. 2. A symmetry mirror plane passes through atoms P(4), Se(1), and P(1) in each of the four symmetry-independent molecules in the structure of P_4Se_3.

between the two structures may be due to small differences in packing caused by the larger size of the P_4Se_3 cage compared to P_4S_3. There are no unusually short contacts in the structure of $[(np_3)Ni(P_4Se_3)] \cdot 2C_6H_6$. The mean distance between the parallel planes of the two benzene solvate molecules is 4.21 Å, compared to 4.11 Å in the P_4S_3 adduct. The short C–C bonds in both benzene molecules, particularly in that one with the larger overall *U* value, are ascribed to uncorrected effects of thermal motion.

Bond distances and angles in the four symmetry-independent P_4Se_3 molecules in the structure of tetraphosphorus triselenide are listed in Table 5. In Table 6 mean values of such bond distances and angles are shown along with corresponding values from the previous work [2] and with the dimensions of coordinated P_4Se_3 in the $[(np_3)Ni(P_4Se_3)]$ molecule. Although standard deviations on the mean values obtained from the present refinement, in which counter data were used, are much

TABLE 4

BOND LENGTHS (Å) AND ANGLES (°) FOR $[(np_3)Ni(P_4Se_3)] \cdot 2C_6H_6$ [a]

Ni–P(1)	2.246(3)	C(14)–C(15)	1.31(2)
Ni–P(2)	2.075(5)	C(15)–C(16)	1.43(2)
Ni···N	3.124(8)	C(16)–C(11)	1.39(2)
P(2)–Se	2.280(3)	C(21)–C(22)	1.36(2)
P(3)–Se	2.238(5)	C(22)–C(23)	1.40(2)
P(3)–P(3)′	2.204(8)	C(23)–C(24)	1.36(2)
P(1)–C(1)	1.85(1)	C(24)–C(25)	1.36(2)
P(1)–C(11)	1.86(1)	C(25)–C(26)	1.41(2)
P(1)–C(21)	1.83(1)	C(26)–C(21)	1.43(2)
N–C(2)	1.49(1)	C(31)–C(32)	1.39(2)
C(1)–C(2)	1.52(2)	C(31)–C(32)′	1.33(2)
C(11)–C(12)	1.33(2)	C(41)–C(42)	1.33(3)
C(12)–C(13)	1.40(2)	C(41)–C(42)′	1.21(3)
C(13)–C(14)	1.40(2)		
P(1)–Ni–P(1)′	108.9(1)	C(12)–C(11)–C(16)	117(1)
P(1)–Ni–P(2)	110.1(1)	C(11)–C(12)–C(13)	124(1)
Ni–P(2)–Se	120.4(1)	C(12)–C(13)–C(14)	117(1)
Se–P(2)–Se′	96.6(2)	C(13)–C(14)–C(15)	122(1)
P(2)–Se–P(3)	102.4(2)	C(14)–C(15)–C(16)	120(1)
Se–P(3)–P(3)′	105.5(2)	C(15)–C(16)–C(11)	120(1)
Se–P(3)–P(3)″	105.7(2)	P(1)–C(21)–C(22)	120(1)
P(3)′–P(3)–P(3)″ [b]	60	P(1)–C(21)–C(26)	121(1)
Ni–P(1)–C(1)	113.6(4)	C(22)–C(21)–C(26)	118(1)
Ni–P(1)–C(11)	119.2(4)	C(21)–C(22)–C(23)	121(1)
Ni–P(1)–C(21)	119.7(4)	C(22)–C(23)–C(24)	121(1)
C(1)–P(1)–C(11)	103.1(5)	C(23)–C(24)–C(25)	119(1)
C(1)–P(1)–C(21)	101.8(6)	C(24)–C(25)–C(26)	123(1)
C(11)–P(1)–C(21)	96.3(6)	C(25)–C(26)–C(21)	118(1)
C(2)–N–C(2)′	113.3(7)	C(32)′–C(31)–C(32)	120(1)
P(1)–C(1)–C(2)	113(1)	C(31)–C(32)–C(31)′	120(1)
N–C(2)–C(1)	110(1)	C(42)′–C(41)–C(42)	124(1)
P(1)–C(11)–C(12)	120(1)	C(41)–C(42)–C(41)′	115(1)
P(1)–C(11)–C(16)	123(1)		

---

[a] Primed and double-primed atoms are related to the corresponding unprimed ones by a threefold symmetry axis. [b] Symmetry-determined value.

TABLE 5

BOND DISTANCES (Å) AND ANGLES (°) FOR P_4Se_3 [a]

	I	II	III	IV
P(4)–Se(1)	2.251(8)	2.256(7)	2.235(8)	2.266(8)
P(4)–Se(2)	2.248(5)	2.245(5)	2.249(5)	2.246(6)
P(1)–Se(1)	2.231(10)	2.238(8)	2.236(10)	2.245(10)
P(2)–Se(2)	2.218(6)	2.234(5)	2.231(5)	2.239(5)
P(1)–P(2)	2.247(9)	2.226(9)	2.224(9)	2.222(8)
P(2)–P(3)	2.231(10)	2.226(11)	2.222(10)	2.220(10)
Se(1)–P(4)–Se(2)	99.1(2)	99.3(2)	99.3(2)	98.7(3)
Se(2)–P(4)–Se(3)	99.6(3)	99.4(3)	99.3(3)	99.8(3)
P(4)–Se(1)–P(1)	100.3(3)	100.2(3)	100.4(3)	100.2(3)
P(4)–Se(2)–P(2)	100.6(2)	100.2(2)	100.3(2)	100.5(2)
Se(1)–P(1)–P(2)	105.3(3)	105.4(3)	105.6(3)	105.5(3)
Se(2)–P(2)–P(1)	105.3(3)	105.9(3)	105.4(3)	105.6(3)
Se(2)—P(2)–P(3)	105.7(3)	105.6(3)	105.7(3)	105.7(3)
P(2)–P(1)–P(3)	59.5(3)	60.0(4)	59.9(4)	59.9(3)
P(1)–P(2)–P(3)	60.2(4)	60.0(4)	60.0(4)	60.0(4)

[a] I–IV denote the four independent molecules in the structure of P_4Se_3. Atomic labels are as in ref. 2 and Fig. 2.

smaller than those from the original structure determination of P_4Se_3 [2], which was based on two-dimensional film data, the mean values of bond distances and angles from the two investigations are in reasonable agreement, except for the mean P_{bas}–P_{bas} distance, which is now found to be significantly shorter than previously reported (Table 6). Comparison between the first and second rows in Table 6 shows that the P_4Se_3 molecule undergoes a compression upon coordination, as indicated by the decrease in the Se–P_{ap}–Se angle and P_{bas}–P_{bas} distance and by the increase in the P_{ap}–Se–P_{bas} angle. Moreover, elongation of the P_{ap}–Se bonds occurs. Analogous deformations of the cage molecule upon coordination were observed for the P_4S_3 adduct, and the possible steric or electronic factors which may give rise to such trends were briefly discussed [1]. In particular, the lengthening of the P_{ap}–Se bonds is ascribed to occupancy of antibonding orbitals which results from the interaction between the electron-rich P_4Se_3 molecule and the d^{10} metal moiety. Extended Hückel calculations performed on model compounds following published procedures [10] confirm the previous suggestion [1] that the main contribution to the bonding

TABLE 6

DIMENSIONS OF THE COORDINATED AND UNCOORDINATED P_4Se_3 MOLECULE [a,b]

	P_{ap}–Se	P_{bas}–Se	P_{bas}–P_{bas}	Se–P_{ap}–Se	P_{ap}–Se–P_{bas}	Se–P_{bas}–P_{bas}	P_{bas}–P_{bas}–P_{bas}
Coordinated [c]	2.280(3)	2.238(5)	2.204(8)	96.6(2)	102.4(2)	105.6(2)	60 [f]
Uncoordinated [d]	2.249(7)	2.233(8)	2.228(9)	99.2(3)	100.4(1)	105.6(2)	60.0(2)
Uncoordinated [e]	2.25(4)	2.23(3)	2.257(8)	99.9(1.8)	100.1(7)	105.3(1.1)	60.0(2)

[a] Standard deviations on mean values of bond distances (Å) and angles (°) were calculated by the formula $[\Sigma_i \Delta_i^2/(n-1)]^{1/2}$, where n is the number of observations and Δ_i are deviations of individual values from the mean. [b] P_{ap} is atom P(2) in Fig. 1 and P(4) in Fig. 2. [c] P_4Se_3 unit in $[(np_3)Ni(P_4Se_3)] \cdot 2C_6H_6$, present work. [d] Present work. [e] Ref. 2. [f] Symmetry-determined value.

between the two moieties is due to participation of 4*s* and 4*p* empty orbitals. The Ni–P_{ap} bond has predominantly σ character, since the bonding and antibonding π contributions largely cancel out.

References

1 M. Di Vaira, M. Peruzzini and P. Stoppioni, Inorg. Chem., 22 (1983) 2196.
2 E. Keulen and A. Vos, Acta Cryst., 12 (1959) 323.
3 K. Irgolic, R.A. Zingaro and M. Kudchadker, Inorg. Chem., 4 (1965) 1421.
4 G.M. Sheldrick, System of Computing Programs, University of Cambridge, 1976.
5 International Tables for X-Ray Crystallography, Volume I, Kynoch Press, Birmingham, England, 1965.
6 A.C.T. North, D.C. Phillips and F.S. Mathews, Acta Cryst. A, 24 (1968) 351.
7 Computer program kindly supplied by Dr. E. Cannillo, Centro Studio Cristallografia Strutturale, CNR, Pavia, Italy.
8 International Tables for X-Ray Crystallography, Volume IV, Kynoch Press, Birmingham, England, 1974.
9 C. Mealli and L. Sacconi, Inorg. Chem., 21 (1982) 2870 and refs. cited therein.
10 R. Hoffmann, Science, 211 (1981) 995 and refs. cited therein.