PERGAMON

Polyhedron 21 (2002) 1261–1265

POLYHEDRON

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Phosphine nickel(0) π-complexes with vinylcyclosilazane —synthesis and structure

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Received 8 January 2002; accepted 13 March 2002

Abstract

Synthesis, spectroscopic (^{1}H, ^{13}C, ^{31}P, ^{29}Si NMR) characterisation and X-ray structure of the first nickel(0) π-complex with vinylcyclosilazane [$\{Ni(PPh_3)\}_2\{\mu\text{-}(\eta\text{-}CH_2{=}CH(Me)Si(\mu\text{-}NH))_4\}$] (**1a**) have been presented. This is the first report on the structure of a TM complex with a vinylsilazane ligand. Complex **1a** is synthesised by the reaction of [$Ni(cod)_2$] with cyclotetra (vinylmethylsilazane). The complex occupies a special position of the space group $P2_1/c$ on the centre of symmetry that lies in the middle of the cyclotetrasilazane ring. The coordination of nickel is close to triangular one. © 2002 Elsevier Science Ltd. All rights reserved.

Keywords: Nickel(0); Vinylcyclosilazane complexes; Crystal structure

1. Introduction

The recent interest in the chemistry of transition metal olefin complexes containing vinyl derivatives of organosilicon compounds as π-ligands is fully justified not only by the particular nature of the bond between vinylsilicon derivatives and transition metals, but mainly by the possible application of this kind of complex as catalysts in hydrosilylation of olefins [1]. Hydrosilylation is also used to a much greater extent in industry to produce crosslinked products [2]. Such processes generally require the use of highly active platinum—containing catalysts. One class of platinum compounds used as hydrosilylation catalysts includes Pt(0) complexes containing vinyl-siloxane ligands. An example is the Karstedt's catalyst, which contains both bridging and chelating divinyl ligands [$\{Pt(\eta\text{-}CH_2{=}CHSiMe_2)_2O\}_2\{\mu\text{-}(\eta\text{-}CH_2{=}CHSiMe_2)_2O\}$] [3,4]. Besides showing a high catalytic activity, this complex is also a good starting compound for synthesis of other platinum(0) π-complexes. The treatment of the Karstedt's catalyst with maleic anhydride, styrene or divinyltetramethyldisiloxane gave [$\{Pt(\eta\text{-}CH_2{=}CHSiMe_2)_2O\}\{\eta^2\text{-}CHC(O)OC(O)CH\}$] [4], [$\{Pt(\eta\text{-}CH_2{=}CHSiMe_2)_2O\}\text{-}(\eta^2\text{-}CH_2{=}CHPh)$] [4] and [$\{Pt(\eta\text{-}CH_2{=}CHSiMe_2)_2O\}\text{-}\{\eta^2\text{-}(CH_2{=}CHSiMe_2)_2O\}$] [5].

Due to the fact that tertiary phosphine ligands play a crucial role in many industrially important metals—catalysed organic reactions, phosphine metal—vinylsilicon complexes were also synthesised [6,7]. The reaction between Karstedt's catalyst [$\{Pt(LL)\}_2(\mu\text{-}LL)$] (where $LL = \{CH_2{=}CHSiMe_2\}_2O$) and mono-, bis- or tris(tertiary phosphine) led to complexes of the type [$Pt(LL)(PR_3)$] [6], [$\{Pt(LL)\}_2(DIPHOS)$] or [$\{Pt(LL)\}_3(TRIPHOS)$] [7], respectively. Moreover there is arousing interest in complexes with cyclic π-vinylsilicon ligands. Cyclotetrakis[vinyl(methyl)siloxane] has been used to generate the platinum complex [$\{Pt(PPh_3)\}_2\{\mu\text{-}(\eta\text{-}CH_2{=}CH(Me)Si(\mu\text{-}O))_4\}$] acting as a hydrosilylation catalyst equivalent to Karstedt's catalyst [2,5,9].

The cost of platinum compounds has stimulated much research to check other transition metal compounds as potential hydrosilylation catalysts. By analogy to Karstedt's catalyst, the nickel complex [$\{Ni(\eta\text{-}CH_2{=}CHSiMe_2)_2O\}_2\{\mu\text{-}(\eta\text{-}CH_2{=}CHSiMe_2)_2O\}$] was obtained and showed high catalytic activity in the hydrosilylation and related reactions (dehydrogenative coupling especially of vinyl derivatives) [10]. In analogy to platinum

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PII: S0277-5387(02)01001-X

complexes, those of the type [Ni(LL)(PR$_3$)] were also prepared [6,7]. Very recently, nickel complexes with cyclotetrasiloxane [{Ni(PR$_3$)}$_2${μ-(η-CH$_2$=CH(Me)Si-(μ-O))$_4$}] were synthesised and the X-ray structure of one of them was determined [11]. There are also well known platinum or nickel complexes with vinylsilanes (i.e. vinyltrimethylsilane [12,13] or divinyldimethylsilane [7]) used as ligands, however, the application of vinyl derivatives of silazane as ligands in organometallic chemistry has been rather rare. Hitherto only two examples of the synthesis of nickel complexes with linear divinyltetramethyldisiloxane, i.e. [{Ni(η-CH$_2$=CHSiMe$_2$)$_2$NH}$_2$ {μ-(η-CH$_2$=CHSiMe$_2$)$_2$NH}] and [Ni(PR$_3$)(η-CH$_2$=CHSiMe$_2$)$_2$NH}], have been reported in Ph.D. Theses [8,14], however, they have not been fully characterised and their crystallographic structures have not been resolved.

In this paper, we present a synthetic procedure, spectroscopic characterisation and X-ray structure of phosphine–nickel complexes with vinylcyclosilazane ligands. To the best of our knowledge this is the first example of a fully characterised TM complex with a vinylsilazane ligand.

2. Experimental

2.1. General procedures

All reagents were dried and purified before use by the usual procedures. [Ni(cod)$_2$] was prepared as described in literature [15]. Other chemicals were purchased from Fluka. The NMR spectra (^{1}H, ^{13}C, ^{31}P, ^{29}Si) were recorded on a Varian XL 300 spectrometer. In all cases C$_6$D$_6$ was used as a solvent.

2.2. Synthesis of complexes

2.2.1. [{Ni(PPh$_3$)}$_2${μ-(η-CH$_2$=CH(Me)Si(μ-NH))$_4$}] (**1a**)

Cyclotetrakis [vinyl(methyl)silazane] (2 ml) was added slowly to a stirred red suspension of [Ni(cod)$_2$] (0.5 g, 1.8 mmol) and PPh$_3$ (0.47 g, 1.8 mmol) in diethyl ether (10 ml) at ambient temperature, yielding a yellow solution. The reaction mixture was allowed to stir overnight. The volatiles were removed in vacuo to give a yellow oil, which was extracted into pentane (2 × 2 ml). The extract was filtered through Celite. The filtrate was concentrated, then set aside at −30 °C to yield a yellow solid compound (0.66 g, 0.72 mmol, 79%). Yellow crystals of **1a** suitable for X-ray diffraction, were grown from benzene at ambient temperature over a period of 24 h.

^{1}H NMR (C$_6$D$_6$, 298 K, 300 MHz,) δ 0.09 (s, 12H), 2.68 (td, 4H), 3.04 (dd, 8H), 7.08–7.76 (m, 30H); $^3J(^1H_1-^1H_2) = 12.0$ Hz, $^3J(^1H_1-^1H_3) = 16.0$ Hz, $^3J(^1H_1-^{31}P) = 4.6$ Hz, $^3J(^1H_2-^{31}P) = 7.7$ Hz, $^3J(^1H_3-^{31}P) = 6.0$ Hz.

^{13}C NMR (C$_6$D$_6$, 298 K, 125.8 MHz); δ 2.66 (s, CH$_3$), 60.66 (d, =CH−), 62.93 (s, =CH$_2$), 129.00–134.00 (m, Ph), $^2J(^{13}C_1-^{31}P) = 6.7$ Hz.

^{29}Si NMR (C$_6$D$_6$, 298 K, 99.4 MHz); δ −12.07(d), $^3J(^{29}Si-^{31}P) = 3.8$ Hz.

^{31}P NMR (C$_6$D$_6$, 298 K, 101.2 MHz); δ 38.59.

2.2.2. [{NiP(C$_6$H$_4$Me-4)$_3$}$_2${μ-(η-CH$_2$=CH(Me)Si(μ-NH))$_4$}] (**1b**)

Compound **1b** (0.76 g, 0.74 mmol, 82%) was prepared in a similar manner to **1a**, except that P(C$_6$H$_4$Me-4)$_3$ was used in the place of PPh$_3$. Yellow crystals of **1b** were obtained by recrystalisation from benzene, at ambient temperature.

^{1}H NMR (C$_6$D$_6$, 298 K, 300 MHz); δ 0.15 (s, 12H), 2.10 (s, 9H), 2.72 (td, 4H), 3.12 (dd, 8H), 6.90–7.34 (m, 24H).

^{13}C NMR (C$_6$D$_6$, 298 K, 125.8 MHz); δ 2.39 (s, CH$_3$), 21.58 (s, CH$_3$), 58.28 (s, =CH−), 61.86 (s, =CH$_2$), 127.90–139.22 (m, tol).

^{29}Si NMR (C$_6$D$_6$, 298 K, 99.4 MHz); δ −12.57 (d).

^{31}P NMR (C$_6$D$_6$, 298 K, 101.2 MHz); δ 39.24.

2.2.3. [{NiP(C$_6$H$_{11}$-c)$_3$}$_2${μ-(η-CH$_2$=CH(Me)Si(μ-NH))$_4$}] (**1c**)

Compound **1c** (0.65 g, 0.66 mmol, 72%) was prepared in a similar manner to **1a**, except that P(C$_6$H$_{11}$-c)$_3$ was used in the place of PPh$_3$. Yellow crystals of **1c** were obtained by recrystalisation from benzene at ambient temperature.

^{1}H NMR (C$_6$D$_6$, 298 K, 300 MHz); δ 0.17 (s, 12H), 1.21–1.82 (m, 33H), 2.63 (td, 4H), 3.08 (dd, 8H).

^{13}C NMR (C$_6$D$_6$, 298 K, 125.8 MHz); δ 2.54 (s, CH$_3$), 26.23–35.54 (m, C$_6$H$_{11}$-c), 59.76 (s, =CH−), 61.34 (s, =CH$_2$).

^{29}Si NMR (C$_6$D$_6$, 298 K, 99.4 MHz); δ −12.94(d).

^{31}P NMR (C$_6$D$_6$, 298 K, 101.2 MHz); δ 38.27.

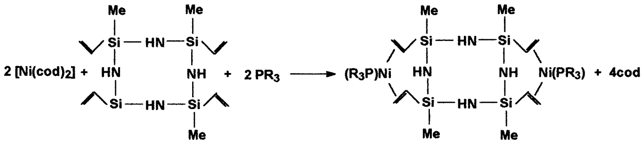
2.3. Crystal structure determination of **1a**

Diffraction data from a yellow, prism-shaped crystal with dimensions 0.2 × 0.2 × 0.15 mm, sealed in a glass capillary, were collected on a KUMA KM4CCD diffractometer [16] at room temperature (r.t.), using graphite-monochromated Mo Kα radiation ($\lambda = 0.71073$ Å). The data collection was performed in six separate runs in order to cover the symmetry-independent part of the reciprocal space. The ω-scan was used with a step of 0.75°, two reference frames were measured after every 50 frames, they did not show any systematical changes either in the peaks positions or in their intensities. A total of 782 frames was collected, giving 22 024 reflections up to $2\theta = 60°$. The unit-cell parameters were determined by the least-squares treatment of the setting angles of 2400 highest-intensity reflections, chosen from the whole experiment. The Lorentz and polarisation corrections were applied [17], and then the reflexes were merged and corrected for absorption ($A_{min} = 0.747$, $A_{max} = 0.767$) with Sortav [18]. The merging procedure produced 6003 unique data with R_{int} of 0.13. The structure was solved by direct methods with SHELXS-97 program [19], and refined with full-matrix least-squares by SHELXL-97 [20]. Non-hydrogen atoms were refined anisotropically, hydrogen atoms were generated geometrically and were refined as a 'riding model' with their U_{iso} parameters set at 1.2 times U_{eq} of the appropriate carrier atom. Selected geometric parameters are listed in Table 1.

Crystallographic data for $[Ni_2(PPh_3)_2(Si_4N_4C_{12}H_{28})]$: empirical formula $Ni_2P_2Si_2N_4C_{48}H_{58}$, monoclinic, space group $P2_1/c$, $a = 13.5628(17)$ Å, $b = 9.7290(12)$ Å, $c = 18.605(2)$ Å, $\beta = 90.349(10)°$, $V = 2454.9(5)$ Å^3, $Z = 2$, $d_x = 1.332$ g cm^{-3}, μ(Mo Kα) $= 0.97$ mm^{-1}, $F(000) = 1032$, $R(F)$ $[F_o > 4(F_o)] = 0.057$, $wR(F^2) = 0.127$, where $w^{-1} = [\sigma^2(F_o)^2 + (0.01P)^2 + 5P]^{-1}$, $P = (\mathrm{Max}(F_o{}^2,0) + 2F_c{}^2/3)$, $S = 1.29$, $(\Delta/\sigma)_{max} = 0.001$ in the last cycle of refinement, $\Delta\rho_{max} = 0.42$ e Å^{-3}, $\Delta\rho_{min} = -0.35$ e Å^{-3}.

3. Results and discussion

The reaction of $[Ni(cod)_2]$ with cyclotetra(vinylmethylsilazane) in the presence of tertiary phosphine was effective for the preparation of the corresponding phosphine nickel–vinylcyclosilazane complexes according to the following equation.



1a R = C_6H_5, **1c** C_6H_{11}-c

1b C_6H_4Me-4,

The above reaction seems to be a very simple and convenient route to synthesise various phosphine complexes with vinylsilicon ligands. As mentioned earlier, the nickel phosphine complexes with cyclotetra(vinylmethylsiloxane) $[\{Ni(PR_3)\}_2\{\mu$-$(\eta$-$CH_2{=}CH(Me)Si(\mu$-$O))_4\}]$ (**2**) (where $R = C_6H_5$ (**a**), C_6H_4–Me (**b**) and c-C_6H_{11} (**c**)) were synthesised previously [11].

Table 1
Selected bond lengths (Å), bond angles (°) and torsion angles (°) with e.s.d.'s in parentheses

Bond lengths			
Ni(1)–X(1)	1.890(10)	Ni(1)–X(2)	1.920(10)
Ni(1)–P(1)	2.175(3)	P(1)–C(21)	1.836(10)
P(1)–C(31)	1.850(12)	P(1)–C(11)	1.859(12)
Si(1)–N(2)	1.710(9)	Si(1)–N(1) [a]	1.722(8)
Si(1)–C(1A)	1.840(10)	Si(1)–C(1)	1.873(10)
C(1A)–C(1B)	1.364(14)	C(2A)–C(2B)	1.417(15)
C(2A)–Si(2)	1.841(11)	N(2)–Si(2)	1.724(9)
Si(2)–N(1)	1.722(7)	Si(2)–C(2)	1.872(11)
Bond angles			
X(1)–Ni(1)–X(2)	127.5(4)	X(1)–Ni(1)–P(1)	113.6(3)
X(2)–Ni(1)–P(1)	117.1(4)	C(21)–P(1)–Ni(1)	123.1(4)
C(31)–P(1)–Ni(1)	116.8(4)	C(11)–P(1)–Ni(1)	108.1(4)
N(2)–Si(1)–N(1) [a]	111.0(4)	N(2)–Si(1)–C(1A)	108.9(5)
N(1)^{i}–Si(1)–C(1A)	111.3(5)	N(2)–Si(1)–C(1)	112.1(5)
N(1)^{i}–Si(1)–C(1)	105.4(5)	C(1A)–Si(1)–C(1)	108.1(4)
C(1B)–C(1A)–Si(1)	121.6(8)	C(2B)–C(2A)–Si(2)	121.2(8)
Si(1)–N(2)–Si(2)	129.7(5)	N(1)–Si(2)–N(2)	111.3(4)
N(1)–Si(2)–C(2A)	111.1(5)	N(2)–Si(2)–C(2A)	109.1(4)
N(1)–Si(2)–C(2)	105.8(4)	N(2)–Si(2)–C(2)	110.7(5)
C(2A)–Si(2)–C(2)	108.9(5)	Si(1) [a]–N(1)–Si(2)	132.7(5)
Torsion angles			
N(2)–Si(1)–C(1A)–C(1B)	−134.2(8)	N(1) [a]–Si(1)–C(1A)–C(1B)	−11.5(10)
C(1)–Si(1)–C(1A)–C(1B)	103.8(9)	N(1) [a]–Si(1)–N(2)–Si(2)	−99.9(7)
C(1A)–Si(1)–N(2)–Si(2)	23.1(8)	C(1)–Si(1)–N(2)–Si(2)	142.6(6)
Si(1)–N(2)–Si(2)–N(1)	101.8(7)	Si(1)–N(2)–Si(2)–C(2A)	−21.1(8)
Si(1)–N(2)–Si(2)–C(2)	−140.8(6)	C(2B)–C(2A)–Si(2)–N(1)	7.4(10)
C(2B)–C(2A)–Si(2)–N(2)	130.4(9)	C(2B)–C(2A)–Si(2)–C(2)	−108.8(9)
N(2)–Si(2)–N(1)–Si(1) [a]	−29.2(9)	C(2A)–Si(2)–N(1)-Si(1) [a]	92.6(8)
C(2)–Si(2)–N(1)–Si(1) [a]	−149.4(8)		

X1 and X2 denote the middlepoints of the C1A–C1B and C2A–C2B bonds, respectively.

[a] Symmetry transformations used to generate equivalent atoms: $-x+1$, $-y$, $-z+1$.

All kinds of analyses of the crystalline yellow complexes **1a**–**c** have brought satisfactory results. The NMR spectra taken for complexes **1a**–**c**, were very similar to those for complexes **2a**–**c**. The NH signal was not observed in the spectra which is consistent with the ^{1}H spectrum obtained for hexamethyldisilazane [21].

The ^{1}H NMR spectra of **1a**–**c** reveals one signal from the methyl group and two signals from the vinyl group in the region appropriate for vinylsilicon complexes of this type. The ^{13}C NMR spectra also confirm the structure of complexes **1a**–**c**. These spectra showed one signal assigned to the methyl carbons and the signals corresponding to the vinyl carbons at frequencies lower than those observed for the free ligand. The ^{31}P NMR spectra of each of the complexes obtained revealed a single signal. The signals in these spectra showed some differences in positions, depending on the type of phosphine used, however, they always appeared in the region typical of a coordinated phosphine. The values of spectroscopic chemical shifts in ^{1}H, ^{13}C and ^{31}P NMR spectra for complexes **1a**–**c** are quite similar to those for complexes **2a**–**c**, but some differences are observed in the ^{29}Si NMR spectra. For the complexes with vinylsiloxane ligands, the ^{29}Si NMR spectrum showed signals at -24 ppm but in the spectra of complexes with vinylsilazane these signals were observed at -12 ppm. The difference depends on the nature of the vinylsilicon compounds used.

The crystal structure of **2b** is very similar to that one established for **1a.**

The complex **1a** lies in a special position of the space group $P2_1/c$, on the centre of symmetry that lies in the middle of the cyclotetrasilazane ring. The coordination of nickel is close to a triangular (regarding the middlepoints of the double bonds as coordination centres), however, some degree of pyramidalization can be noticed. The sum of the angles around nickel is 358.2(3)° and the Ni1 atom is displaced by 0.156(5) Å from the plane defined by P1, X1 and X2 (X1 and X2 denote the middlepoints of C1A–C1B and C2A–C2B double bonds, respectively). The Ni–P bond length of 2.175(3) Å is within the range for short Ni–P bonds (the Ni–P lengths distribution is bimodal [22]).

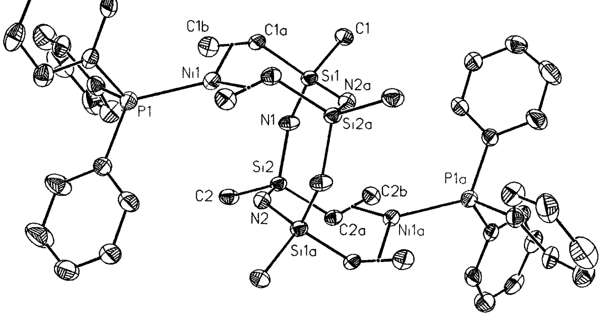


Fig. 1. The thermal ellipsoid view of the complex **1a** (at 50% probability level) with the numbering scheme [23]. The hydrogen atoms are omitted for clarity.

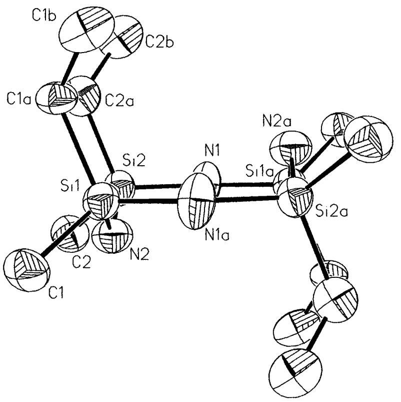


Fig. 2. The conformation of the tetrasilazane ring. The thermal ellipsoids are drawn at 50% probability level, only symmetry-independent atoms are labelled.

The eight-membered silazane ring adopts the chair conformation; six atoms N1, Si2, Si1, N1′, Si2′ and Si1′ are almost perfectly coplanar (the prime denotes the atoms created by the centre of symmetry), with the largest deviation from the least-square plane of 0.012(5) Å, while the N2 and N2′ atoms are tilted out of this plane by + and $-0.729(9)$ Å, respectively, (Figs. 1 and 2). This different character of N1 and N2 nitrogen atoms is reflected in differences of the Si–N–Si angles: 132.7(5)° for N1 and 129.7(5)° for N2, while the Si–N bond lengths are almost equal with the mean value of 1.720(2) Å. The methyl substituents are in quasi-equatorial positions, and the vinyl ones in quasi-axial ones (cf. Table 1).

The phenyl rings are planar within experimental error, the mean value of the C_{ar}–C_{ar} bond length is 1.380(7) Å, and agrees well with the typical value. The mutual disposition of these rings can be described by the dihedral angles between their least-squares planes A/B 85.1(4)°, B/C 72.6(4)°, A/C 82.8(3)°, where A, B, and C denote the rings with C11, C21, and C31 atoms, respectively. The crystal packing is determined mainly by van der Waals interactions.

4. Conclusions

1) Nickel complexes of the type $[\{Ni(PR_3)\}_2\{\mu\text{-}(\eta\text{-}CH_2{=}CH(Me)Si(\mu\text{-}NH))_4\}]$ are the first examples of TM complexes with vinylsilazane whose crystal structure has been determined.
2) The NMR and crystal structure data obtained have proved that the compounds **1a**–**c** and **2a**–**c** are similar. They both are binuclear nickel(0) complexes with a inversion centre at the mid-point of the chair-shaped $(SiO)_4$ or $(SiNH)_4$ ring.

3) Phosphine nickel(0) π-complexes with vinylcyclosilazane ligands seem to be promising potential catalysts for hydrosilylation and related reactions; the catalytic aspects will be considered in a separate paper.

5. Supplementary data

Supplementary data are available from the CCDC, 12 Union Road, Cambridge, CB2 1EZ (fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk; or www: http://www.ccdc.cam.ac.uk) on request, quoting the deposition number CCDC 168038.

Acknowledgements

This work was supported by funds from the State Committee for Scientific Research, Poland, Project No. 2 T09A 122 17.

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