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THERMAL DECOMPOSITION OF η^5-CYCLOPENTADIENYL-η^2-PROPENEALKYLNICKEL COMPLEXES *,**

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Summary

Thermolysis of compounds of the type CpNiR (η^2-propene) (R = CH_3 (**1**), CD_3 (**2**), CH_2CH_3 (**3**), $CH_2CH_2CH_3$ (**4**) and CH_2SiMe_3 (**5**)) was studied. The methyl complex **1** gives mainly methane, ethane and $(CpNi)_3CH$. Toluene-d_8 has no effect on the composition of the gaseous products and does not yield gases containing deuterium. In the CD_3 complex **2** a H–D exchange reaction between the complexed propene and the CD_3 group takes place. Decomposition of the trimethylsilylmethyl complex **5** gave tetramethylsilane. The complexes **3** and **4** containing a β-hydrogen atom, decompose via a β-elimination reaction giving the corresponding olefin. The unstable {CpNiH} formed in the reaction reacts with {CpNiR} giving the corresponding alkane and a mixture of non-isolated cyclopentadienylnickel clusters. The complexes studied undergo insertion of the coordinated olefin into the Ni–C σ-bond, which results in the formation of higher hydrocarbons.

Introduction

Thermal decomposition reactions of alkyl-transition metal compounds have been intensively studied in recent years because of the importance of these types of compounds in organometallic chemistry and in various organic reactions catalyzed by transition metals. Understanding the behaviour of these transition-metal complexes is of fundamental importance in the elucidation of metal-promoted catalytic processes.

The decomposition of alkyl-transition metal compounds has been studied for Ti, V, Cr, Mn, Co, Pt and other metals. However, no general theory for decomposition, or even a clear understanding of specific cases has resulted. Wilkinson [1], Baird [2],

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* Dedicated to Professor Günter Wilke on the occasion of his 60th birthday.

** This work has been carried out between November 1981 and July 1982 in Mülheim.

Davidson [3]. Whitesides [4] and Schrock [5], among others, discuss the decomposition extensively.

The thermolysis of compounds containing a nickel–carbon σ-bond was not studied in detail until now. The preparation and thermal decomposition of $[(2\text{-biph-O})_3P]_2NiMe_2$was described by Wilke [6]. He found evolution of 1 mol of ethane per gram-atom of Ni. The thermal decomposition of the $Et_2Ni(dipy)$ complex was studied by Yamamoto et al. [7]. The complex began to decompose at 110°C under vacuum. Ethane, ethene and n-butane were formed as gaseous products. The heating of $Me_2Ni(PMe_3)_3$ and $Me_2Ni(PMe_3)_2$ above 60°C gives ethane, nickel(0) and trimethylphosphine [8]. Similarly the $R_2Ni(PPh_3)_2$ complex ($R =$ n-Pr, n-Bu) eliminates predominantly the 1-olefins [9]. Yamamoto et al. [10] prepared a series of dialkyl(dipyridyl)nickel compounds with $R = CH_3$, C_2H_5, n-C_3H_7 and i-C_4H_9 and studied their thermal stabilities. The stabilities of the alkylnickel bonds are indicated by the decomposition temperatures of the complexes, which are in the sequence $CH_3 > C_2H_5 >$ n-$C_3H_7 >$ i-C_4H_9 (160°C, 97°C, 94°C, −15°C, respectively). Pyrolysis of di-n-propyl(dipyridyl)nickel gives propane and propene as products in approximately equal amounts.

The paper mentioned above described bis-alkyl-nickel compounds. Thomson and Baird studied the thermal decomposition reactions of mono-alkyl-nickel compounds. The compounds η^5-$C_5H_5Ni(PPh_3)R$ (R = Me, Et, n-Pr, iso-Pr, n-Bu, s-Bu, iso-Bu, $PhCH_2$, Me_3SiCH_2) have been prepared and characterized, and their thermal decompositions studied. Compounds containing a β-hydrogen atom decompose via a β-elimination reaction, the others apparently via a unimolecular reaction which does not involve free radicals [11,12]. Nearly all the thermolyzed alkylnickel compounds decompose at 60–160°C. At such high temperatures a number of side reactions can take place, which complicate the picture of nickel–carbon σ-bond scission. Therefore, alkylnickel compounds of lower thermal stability and the general formula $CpNiR(\eta^2\text{-}CH_2{=}CHCH_3)$ where $R = CH_3$, CD_3, C_2H_5, C_3H_7, $(CH_3)_3SiCH_2$ were chosen for our studies, since their decomposition begins already at temperatures above −20°C.

Results

η^5-Cyclopentadienylmethyl-η^2-propenenickel (**1**) is relatively stable at temperatures below −20°C or at room temperature under equilibrium propene pressure. Above −20°C it slowly decomposes with the evolution of gases. After 14 days at 24°C half the volume of gases is evolved, the second half evolves at 24–70°C. When the complex is heated from −40°C to room temperature during 3–4 h, ca. 20% of the total volume of gases evolves. The evolution of gases is the fastest at 40–45°C and amounts to ca. 60% of the total volume. Above 70°C the thermal decomposition of the complex with evolution of gases does not proceed. The gases evolved were mainly propene, methane and ethane (Table 1, exp. 1, 2), smaller amounts of C_4 and very little C_5 gases. The thermal decomposition of the methyl complex **1** in a closed vessel at room temperature during 14 days (Table 1, exp. 4) results in an increase in the amount of propene evolved and a considerable decrease in that of methane from ca. 30 to 17%. Under these conditions the amount of C_4 gases increases from 2 to 4%. The composition of the gases evolved depends only to a small extent on the reaction temperature (Table 1, exp. 5). When the thermal decomposition of complex

TABLE 1

THERMAL DECOMPOSITION OF CpNi(CH_2=$CHCH_3$)Me (Thermolysis temperature −20 to 80°C)

Exp.	R	Solvent	Gases formed (%)											
			CH_3	C_2H_6	C_3H_6	C_3H_8	i-C_4H_{10}	n-C_4H_{10}	2-C_4H_8 *trans*	1-C_4H_8	i-C_4H_8	2-C_4H_8 *cis*	C_3H_4	C_5
1	CH_3	–	31.44	5.46	61.13	–	0.65				1.31			
2	CH_3	toluene-d_8	29.92	3.88	63.98	2.22								
3	CD_3	–	18.0	8.37	63.0	1.24	0.25	1.04	1.84	0.82	2.77	0.76	0.19	1.17
4 [a]	CH_3	–	16.6	5.72	74.2	1.14	0.15	1.00	0.31	0.08	0.58	0.11	0.06	–
5 [b]	CH_3	–	13.6	3.9	77.5	1.05	0.21	1.28	0.49	0.26	1.09	0.22	0.13	0.18

[a] Thermolysis time 14 days, room temperature. [b] The sample in exp. 4 was heated to 80°C.

1 dissolved in toluene-d_8 was carried out, no deuterium was found in the gases evolved. Also, no effect of the solvent on the rate and composition of the gases evolved was found (Table 1, exp. 2).

After thermal decomposition of the methyl complex a black-green paramagnetic solid was obtained. At 80°C a small amount of Cp_2Ni sublimes from it, which was confirmed by mass spectroscopy. This solid dissolves in THF, toluene and benzene, partly dissolves in Et_2O and is insoluble in pentane. The mass spectrum of the solid at 140°C contains signals at *m/e* 188, 125, 123, 97, 66, 60, 58 and 39. The signals are of high intensity and they correspond to the typical fragmentation products of Cp_2Ni (188). Additionally an intense signal at *m/e* 66 (C_5H_6) and a number of signals corresponding to the organic products being formed (ca. 40) occur. Besides the signals mentioned above, a number of signals of low intensity occur in the spectrum, the *m/e* values of which correspond e.g. to $CpNi_3$ (239), $(CpNi)_2$ (246), Cp_2Ni_3 (304). The signals at *m/e* 382, 384, 386, 388, corresponding to $(CpNi)_3CH$, are also present but no signals corresponding to $(CpNi)_2CH_2$ are present in the spectrum.

At higher temperatures (240 and 280°C) the number of $(CpNi)_2$ fragmentation products increases.

In order to purify the thermal decomposition products from Cp_2Ni and other oligomeric products $(CpNi)_n$, they were washed many times with Et_2O, until the green colouring disappeared, and dissolved in toluene. Then the precipitate was filtered and the solvent was distilled off. A solid was obtained, which did not melt up to 340°C. During heating the appearance of the substance changes, probably due to its decomposition. The compound is paramagnetic. Anal. Found: C, 50.51; H, 4.20; Ni, 45.16. $C_{16}H_{16}Ni_3$ calcd.: C, 50.00; H, 4.17; Ni, 45.83%.

The thermolysis of the $CpNi\text{-}\eta^2\text{-}(CH_2{=}CHCH_3)CD_3$ complex **2** results in the evolution of higher amounts of C_4 and C_5 gases (~ 10%), a lower amount of methane (~ 10%) and a higher amount of ethane (~ 4%) (Table 1, exp. 3). Complex **2** is more stable than complex **1**. When heated for 2.5 h at −40 to 22°C it decomposes evolving ca. 4% of gases. The fastest decomposition takes place from 50 to 60°C. The analysis of the gases obtained during the decomposition of complex **2** showed the presence of 53% of CD_4 and 47% of CD_3H but less deuterated methane is practically absent. Almost completely deurated ethane (CD_3CD_3) was found and about 10% of propene evolved was monodeuterated.

The η^5-cyclopentadienyl-η^2-propeneethylnickel complex **3** slowly decomposes at room temperature (19 cm^3 of gases during 2 h) and quickly at 50–60°C (127 cm^3 of gases at 22–80°C during 1 h). At temperatures above 80°C further decomposition with the evolution of gases does not take place. The composition of the gases evolved

TABLE 2

THERMAL DECOMPOSITION OF $CpNi(CH_2{=}CHCH_3)Et$ (Thermolysis temperature −20 to 80°C)

Gases	(%)
Ethane	21.4
Ethylene	17.8
Propene	56.0
1-Butene	4.7

TABLE 3

THERMAL DECOMPOSITION OF CpNi((CH_2=$CHCH_3$)Pr. (Thermolysis temp. −20 to 80°C)

Exp.	Solvent	Gases (%			
		C_3H_6	C_3H_8	1-C_5H_{10}	1-C_6H_{12}
1	–	82.8	16.4	0.34	0.45
2	toluene	82.2	17.8	–	–
3	toluene-d_8	83.3	16.7	–	–
4	–	85.0	14.3	0.3	0.5

during the thermolysis is shown in Table 2. The residue after decomposition is a black-green solid.

The η^5-cyclopentadienyl-η^2-propenepropylnickel complex **4** was thermolyzed from −20 to 80°C. From −20°C to room temperature the decomposition of the complex proceeds very slowly. The fastest evolution of gases is observed in the range 50–70°C. Above 80°C no gases evolve. The course of thermolysis is independent of the reaction time, but it depends on the temperature (Table 3, exp. 1, reaction time 25 min, exp. 3, reaction time 6 h) and the amount of gases evolved is ca. 80% of that calculated. The composition of the gases evolved is independent of decomposition in the absence of solvent or in toluene solution (Table 3, exp. 1 and 2). When the thermolysis is carried out in a solution of deuterated toluene (Table 3 exp. 3) no gases containing deuterium are formed.

The residue after thermal decomposition is a black-green solid and it partly dissolved in THF, toluene and benzene. The main part of the solid does not dissolve in any of those solvents.

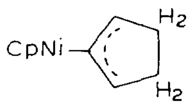
The thermolysis of complex **4** carried out in an autoclave for 3 h at 120°C in the presence of an excess of propene gave about 90% of propene dimerization products. Lower and higher hydrocarbons than C_6 are the compounds found (Table 4).

The black-red residue remaining in the autoclave after thermolysis did not decompose further. Autoclaving took place in the presence of excess propene at 90°C under atmospheric pressure. From the residue, a small amount of a red, viscous liquid was distilled off at 100°C in oil pump vacuum. On the bases of the ^{1}H NMR spectrum this liquid was found to be complex **5**.

TABLE 4

THERMAL DECOMPOSITION OF CpNi(CH_2=$CHCH_3$)Pr IN AN AUTOCLAVE IN THE PRESENCE OF PROPYLENE (Temperature 120°C, time 3 h)

Dimerization products	(%)
2-Methylpent-3-ene	11.4
2-Methylpent-1-ene	17.4
Not identified	4.88
1-Hexene	14.9
3-Hexene *cis*	0.054
3-Hexene *trans*	0.22
2-Hexene *trans*	34.7
2-Hexene *cis*	9.76

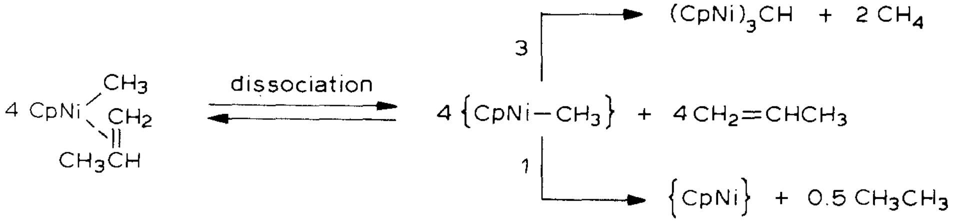


(5)

The η^5-cyclopentadienyl-η^2-propenesilylmethyl complex **6** was thermally decomposed at 20–80°C. A clear decomposition with the evolution of gases starts at 28°C, with very rapid thermolysis at 75–80°C, at 80°C the evolution of gases stops. Propene was found in the gases, and tetramethylsilane was distilled off in an oil pump vacuum from the residue after thermolysis.

Discussion

The reaction course of the thermal decomposition of complex **1** is shown in Scheme 1. The coordinated propene stabilizes the complex. At temperatures above −20°C a decoordination reaction of propene proceeds with the formation of the unstable compound $\{CpNiCH_3\}$. The rate of decoordination increases with rise of temperature. The reaction is reversible. Thus, in the presence of an excess of propene at room temperature and equilibrium pressure in the autoclave no decomposition takes place. The unstable compound $\{CpNiCH_3\}$ easily decomposes giving methane, ethane, isolated tris(η^5-cyclopentadienylnickel)methane and unisolated $\{CpNi\}_n$ oligomers.



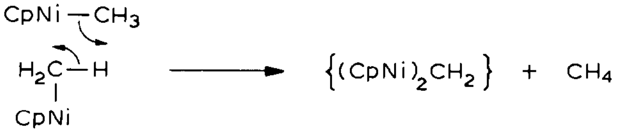
	Found (%)	Calcd. (%)
CH_4	32.0	30.8
CH_3CH_3	5.6	7.7
$CH_2{=}CHCH_3$	62.4	61.5

SCHEME 1

Ustynyuk et al. [14] described compounds of structures $(CpNi)_3CC_6H_5$ and $(CpNi)_3CC_6H_4CH_3$, which are analogous to $(CpNi)_3CH_3$ and Dahl et al. [15] have shown the existence of five different kinds of cyclopentadienylnickel clusters, among them $Ni_3(C_5H_5)_4$ and $Ni_6(C_5H_5)_6$.

A good correlation between the amounts of gases found and calculated favour Scheme 1. From four mol of $\{CpNiCH_3\}$, three mol react giving methane and one mol gives ethane.

The probable mechanism of methane formation consists in α-hydrogen abstraction in a bimolecular reaction with the simultaneous formation of bis(η^5-cyclopentadienylnickel)methane.

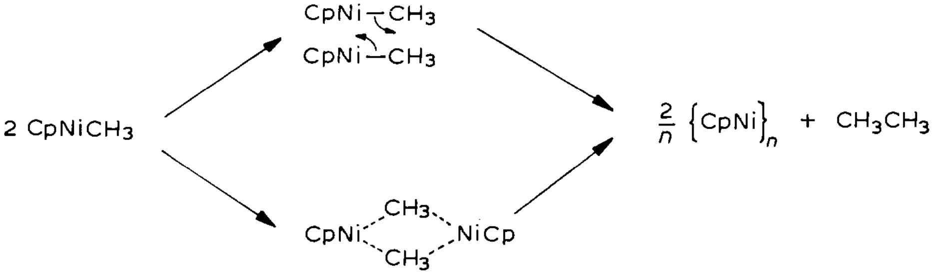


Under favourable conditions then β-hydrogen abstraction takes place giving $(CpNi)_3CH$:

$$\{(CpNi)_2CH_2\} + \{CpNiCH_3\} \rightarrow (CpNi)_3CH + CH_4$$

The positioning of two molecules of $CpNiCH_3$ could result in the methyl groups in an unexpectedly close proximity to each other, resulting in interaction between the groups which would not take place if they were further apart.

Similarly the mechanism for ethane formation can be explained by one of the two ways shown in Scheme 2. The reaction can be considered as reductive elimination, which may proceed via a concerted reaction and can provide low energy pathways.



SCHEME 2

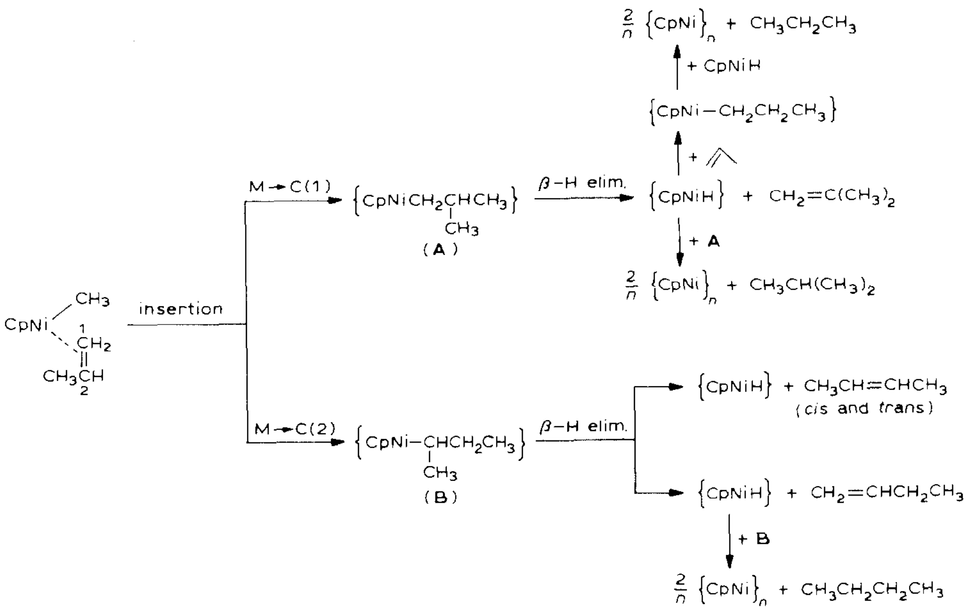
α-Hydrogen abstraction and coupling reactions can proceed via heterocyclic or homolytic scission of the Ni–C or C–H bonds. It seems possible that homolytic cleavage generates short-lived radicals (to be distinguished from "free" radicals) which react with hydrogen or a methyl group. Due to steric hindrances α-hydrogen abstraction should proceed easier than coupling. The results obtained confirm this, since from four mol of $\{CpNiCH_3\}$ three mol give methane and one gives ethane (Scheme 1).

When the $\{CpNiCH_3\}$ compound is decomposed under normal pressure and with continuous removal of the gases, propene, methane and ethane form up to ca. 98% of the gaseous products. The remaining 2% are mainly butenes and butanes. The decomposition of the complex under the pressure of the gases being evolved (mainly propene within approximately a fortnight causes an increase of ca. 4%. in the amount of side products.

The formation of higher hydrocarbons can be explained by the course, of the complexed propene molecule side insertion reaction into the Ni–CH_3 bond, besides the main reaction of dissociation of complex **1**. The insertion can proceed at the C(1)–C(2) bond of propene.

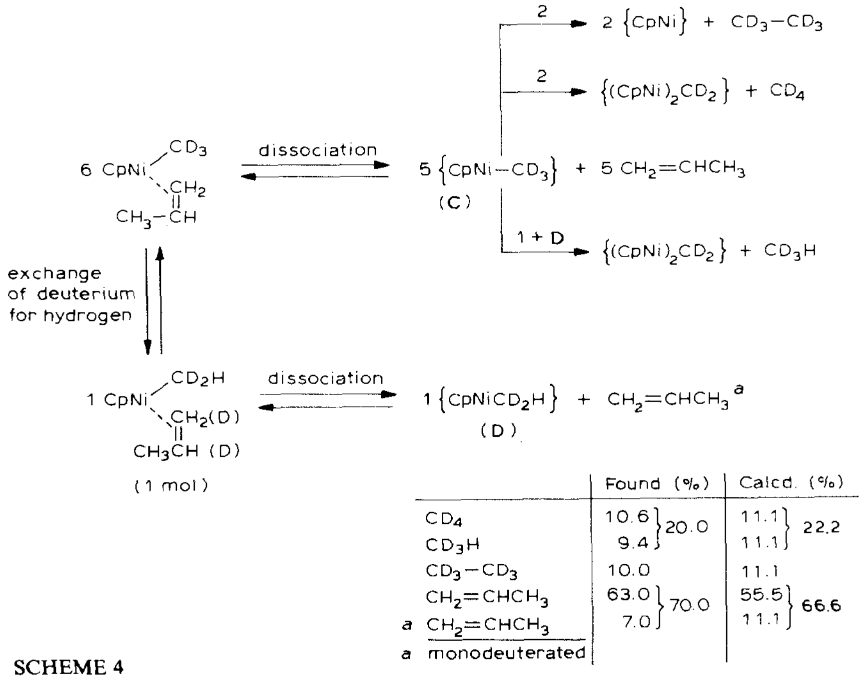
The unstable complexes **A** and **B** formed (Scheme 3) undergo further β-hydrogen elimination reactions giving butenes, butane, isobutane and propane.

The solvent has no remarkable effect on the thermolysis of complex **1** for thermolysis of **1** in toluene-d_8 did not give deuterated gases. This means that methane is not formed as a result of hydrogen abstraction from the solvent.



SCHEME 3

The thermal decomposition of CpNi-η^2-(CH_2=CHCH)$_3$$CD_3$ (**2**) takes place at a higher temperature than that of complex **1**. Besides the main gaseous products (propylene, methane and ethane, 90%), also ca. 10% of higher hydrocarbons are formed (Table 1, exp. 3). This indicates that the increased stability of complex **2** favours the insertion shown in Scheme 3.



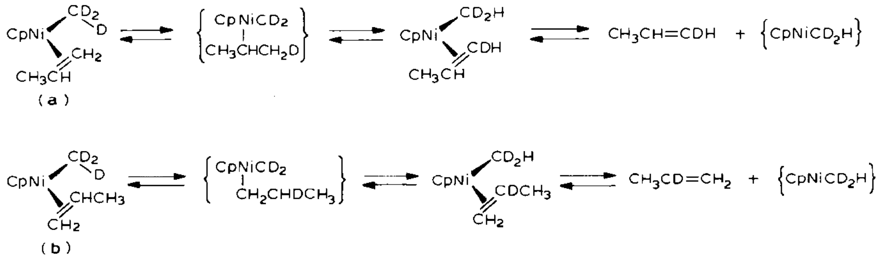
	Found (%)		Calcd. (%)	
CD_4	10.6	20.0	11.1	22.2
CD_3H	9.4		11.1	
CD_3–CD_3	10.0		11.1	
CH_2=$CHCH_3$	63.0	70.0	55.5	66.6
a CH_2=$CHCH_3$	7.0		11.1	

a monodeuterated

SCHEME 4

Scheme 4 presents the course of dissociation and explains clearly the formation of deuterated gases. It is suggested that the lower amount of methane evolved (~ 20%) in comparison to that in the case of complex **1** (~ 30%) is caused by the lower reactivity of $(CpNi)_2CD_2$ compared to its hydrogen derivative.

In order to explain the formation of CD_3H (~ 10%) and monodeuterated propene (~ 7%) it must be assumed, that in complex **2** an exchange of deuterium for hydrogen takes place. This reaction is presented in Scheme 5. As was shown by Lehmkuhl et al. [16], the ratio of rotamers **a**/**b** = 9/1 in methyl derivatives is probably the reason why $CH_3CH{=}CDH$ is in excess in the products.



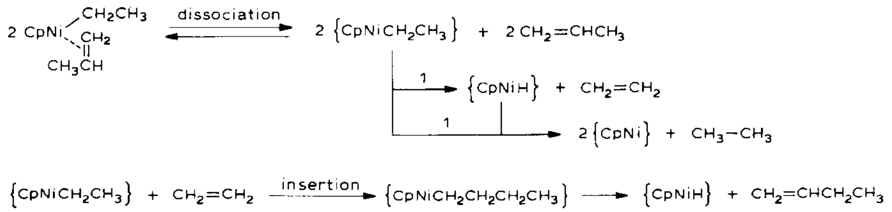
SCHEME 5

Hydrogen abstraction from the CH_3 group of the $\{CpNiCH_3\}$ complex, proceeds in our experiments more readily than deuterium abstraction from the CD_3 group. Hence the following reaction leads mainly to CD_3H:

$$CpNCD_3 + \{CpNiCD_2H\} \rightarrow \{(CpNi)_2CD_2\} + CD_3H$$

Thus, despite the high excess of $\{CpNiCD_3\}$ to $\{CpNiCD_2H\}$, 5/1, almost equimolar amounts of CD_4 and CD_3H are evolved.

The thermolysis of the ethyl complex **3**, similar to that of the methyl complex **1** starts from dissociation of the coordinated propene molecule. The unstable compound, $\{CpNiCH_2CH_3\}$, thus formed, yields, via β-hydrogen elimination, ethene and very reactive $\{CpNiH\}$ (Scheme 6).



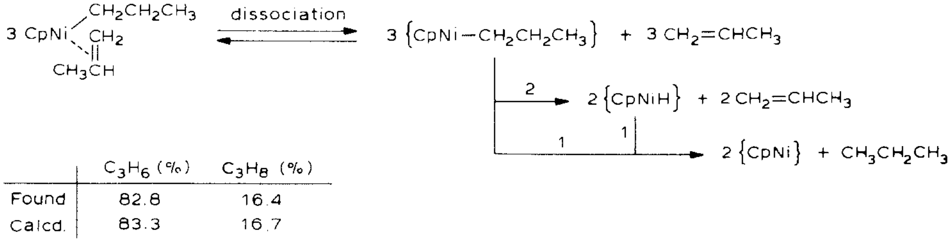
	C_3H_6 (%)	C_2H_4 (%)	C_2H_6 (%)	1-C_4H_8 (%)
Found	56.0	17.8	21.4	4.7
Calcd.*	52.6	18.4	23.7	5.3

*Assuming that 5% of $\{CpNiCH_2CH_3\}$ reacts with ethylene yielding 1-butene

SCHEME 6

The reaction of $\{CpNiH\}$ with $\{CpNiCH_2CH_3\}$ yields ethane and a cyclopentadienylnickel cluster. Assuming that ca. 5% of $CpNiCH_2CH_3$ reacts with ethylene (insertion reaction) yielding 1-butene, the agreement in the found and calculated amounts of gases is good. The absence of butane in the gaseous products indicates that compounds containing a β-hydrogen do not undergo a bimolecular C–C bond formation reaction.

From the thermolysis of the propene complex **4** propene and propane are formed almost exlusively, 99.2% (Scheme 7). Not taking into account propene formation from dissociation of complex **4**, the decomposition of $\{CpNiCH_2CH_2CH_3\}$ leads to the formation of propene and propane (1/2 mole ratio). This indicates that the $\{CpNiH\}$ formed easily hydrogenates the propyl group bonded to nickel. The agreement in the experimentally and theoretically calculated amounts of gases as can be seen in Scheme 7 is very good.



	C_3H_6 (%)	C_3H_8 (%)
Found	82.8	16.4
Calcd.	83.3	16.7

SCHEME 7

The thermolysis of complex **4** carried out in an autoclave at 120°C at equilibrium pressure of excess propene gives mainly propene dimerization products (~ 73%), similar to the case of complex **1** (Scheme 3), but with a considerably higher yield resulting from the higher temperature and propene pressure. The first reaction step is propene insertion into the Ni–C bond (Scheme 8). The next steps are β- and γ-hydrogen elimination and the formation of respective hexenes. In contrast to the methyl complex, no olefin hydrogenation products are formed, since the excess of propene and its pressure favour the synthesis of complex **4**.

$$\{CpNiH\} + CH_2{=}CHCH_3 \rightarrow \{CpNiCH_2CH_2CH_3\} \xrightarrow{+CH_2=CHCH_3} \mathbf{4}$$

The formation of 3-hexene (*trans* and *cis*) and 2-methylpent-3-ene can be explained by γ-hydrogen elimination. Complex **5**, not having a β-hydrogen gives mainly tetramethylsilane. Similarly as in the case of the methyl complex **1**, tetramethylsilane results mainly from α-hydrogen abstraction.

Experimental

Reagents

η^5-Cyclopentadienyl-η^2-propenealkylnickel complexes were obtained by methods described earlier [13]. Their purity was determined by means of ^{1}H NMR. All the solvents used were carefully deoxygenated and dried. Toluene-d_8, THF-d_8 and CD_3I were dried by means of sodium tetraethylaluminate and distilled.

All the reactions were carried out under dry, deoxygenated argon.

Example of a typical thermal decomposition reaction

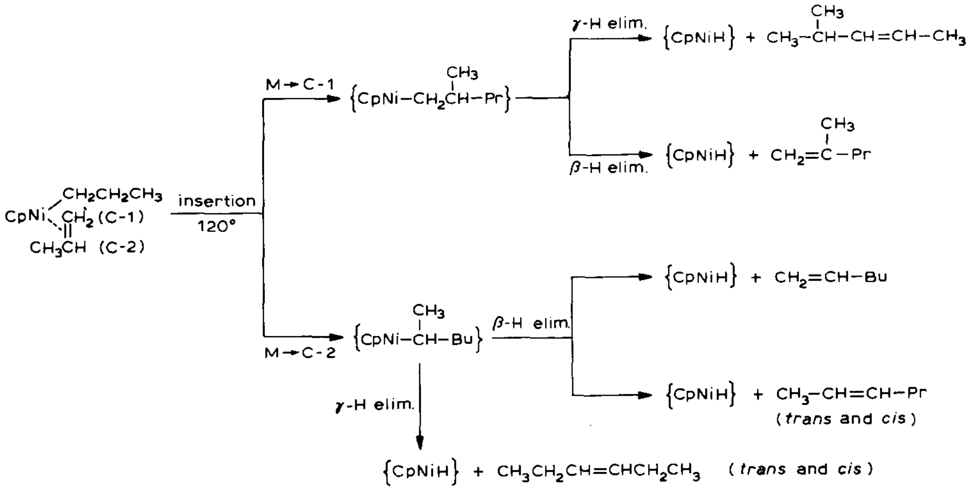
0.5-1 g of the complex under investigation was placed into a weighed Schlenk vessel cooled to −60°C. The vessel was weighed again and connected to a gas burette filled with mercury. Thermolysis was carried out under argon with oil pump vacuum slowly increasing the temperature from −60 to 80°C. The rate of thermolysis was determined by the volume of gases being evolved. After reaching 80°C and finding that no more gases are evolved, a sample of gases was taken into an ampoule under high vacuum. The gases evolved were identified by gas chromatography and/or mass spectroscopy. If the thermolysis was carried out with longer reaction times and at room temperature a thick-wall 150 cm^3 Schlenk vessel was used. After the required time the vessel was connected to a gas-burette and the volume of gases evolved was measured. A sample of the gases was taken for analysis. The results are given in Tables 1–3.

Thermolysis of $CpNi(CH_2{=}CHCH_3)Pr$ at 120°C in the presence of propene

To 50 cm^3 of liquid propene 0.90 g (4.32 mmol) of $CpNi(CH_2{=}CHCH_3)Pr$ was introduced. The solution obtained was poured into a cooled (−40°C) and flushed with argon (100 cm^3) autoclave. The autoclave was closed and the temperature was allowed to rise to 25°C within 3 h. The content was stirred by a magnetic stirrer. The autoclave was then heated to 120°C and the reaction was carried out at that temperature for 3 h. Then the autoclave was cooled to −40°C and its content was transferred to a Schlenk vessel. Propene was distilled off from the vessel under a slight over-pressure. The residue was distilled at −20°C under an oil pump vacuum and 0.9 g of a colourless liquid was obtained, which contained 80% of propene dimerization products (Table 4). The residue in the vessel is a red-black solid. Heating of this solid to 90°C does not cause any gas evolution. At 100°C under oil pump pressure a small amount of a deep-red viscous liquid was distilled off. The ^1H NMR spectrum showed it to be the hydrogenation product of Cp_2Ni (probably via CpNiH) of the formula $CpNiC_5H_7$.

Isolation of $(CpNi)_3CH$

The solid after thermolysis, $CpNi(CH_2{=}CHCH_3)Me$, was washed many times on



SCHEME 8

the filtration funnel with small portions of Et_2O until the green colour of the filtrate disappeared. The insoluble residue was dried under oil pump vacuum and then 5 cm^3 of toluene was added. The toluene was filtered off, and the residue was washed twice with 5 cm^3 of toluene. The filtrates were distilled under oil pump vacuum and a black crystalline substance was obtained. Found: C, 50.58; H, 4.22; N, 45.12. $C_{16}H_{16}Ni_3$ calcd.: C, 50.00; H, 4.17; N, 45.83%. The compound is paramagnetic, and mass spectral data are given in the Results section.

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