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Rearrangement processes of bis-(η^5-alkylcyclopentadienyl)-zirconium dichlorides under electron impact

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Abstract

Rearrangement processes occurring in compounds $(C_5H_4R)_2ZrCl_2$ (R = H, Me, ^{n}Pr, ^{i}Pr, ^{n}Bu, ^{t}Bu) and $(C_5Me_5)_2ZrCl_2$ under electron impact have been studied. The structure of an alkyl substituent in a cyclopentadienyl ligand has been shown to determine the dissociative ionization direction. The availability of a hydrogen atom at a substituent α-carbon atom alone gives the possibility of preceding the rearrangement process with the elimination of an HCl molecule from a molecular ion. The role of the process above increase with increasing carbon atom number in the alkyl substituent of the normal structure. The complete substituent of hydrogen atoms by methyl groups in a cyclopentadienyl ring reduces the probability of the rearrangement process with HCl elimination occurring due to steric hindrances. On the basis of the decomposition of the compound $[C_5H_4C(D)Me_2]_2ZrCl_2$, direct evidence of the participation of the hydrogen atoms at the substituent α-carbon atom in the rearrangement processes has been obtained. The elimination of a DCl molecule has been demonstrated to be one of the directions of molecular ion decomposition. The subsequent abstraction of an HCl molecule is suggested to result from deutero-hydrogen exchange or the participation of methyl groups in the substituent.

Introduction

At present, high-activity catalytic systems based on zirconocene derivatives are widely studied for in organic synthesis [1] and olefin polymerization processes [2–5]. The catalytic activity of such systems is determined by several factors including the ability of zirconium derivatives to be rearranged in a reaction complex with the formation of hydride centers [6]. Moreover, in some works [7,8], it was suggested that on generating catalytic centers, the cation complexes of zirconocene were obtained. Studies carried out in the gaseous phase under electron impact showed that the reactions of the cation complexes formed are of common character with separate stages of the catalytic process [6,9]. Regarded in

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Table 1

Mass spectra of compounds **I–VIII**

Ion	Relative intensity (%) [a]							
	I	**II**	**III**	**IV**	**V**	**VI**	**VII**	**VIII**
M^+	47.2	68.1	26.0	21.8	10.7	17.8	33.0	32.1
$(M-Cl)^+$	12.3	13.2	6.0	5.5	5.4	6.1	4.1	2.0
$(M-HCl)^+$	–	100.0	100.0	100.0	100.0	100.0	–	15.1
$(M-HCl-H)^+$	–	13.9	6.1	13.5	6.5	2.1	–	0.3
$(M-2HCl)^+$	–	15.2	27.3	44.0	29.3	18.2	–	–
$(M-Me)^+$	–	–	–	–	–	–	40.6	–
$(M-Me\text{-}H)^+$	–	–	–	–	–	–	6.0	–
$(M-Me-HCl)^+$	–	–	–	–	–	–	100.0	–
$(M-L)^+$	100.0	85.3	39.9	58.0	22.6	59.1	46.2	100.0
$(M-L-H)^+$	–	5.3	2.3	0.3	2.5	–	–	–
$(M-L-2H)^+$	–	4.6	19.2	24.7	13.4	12.5	1.0	–
$(M-2L)^+$	5.7	15.3	15.6	16.1	1.9	12.2	0.9	0.1

[a] The relative intensity of the line for each ion is the sum of the line intensities of its isotopic varieties referred to the similar sum of intensities of ion lines taken as 100%. M^+, molecular ion; L, π-ligand.

that light, knowledge of zirconocene properties appearing under electron impact is of interest.

The rearrangement processes of bis(cyclopentadienyl)metal dichlorides have been insufficiently investigated. The fragmentation directions for the compounds $(C_5H_4Alk)_2TiCl_2$ [10] and $Me_2Si(C_5H_4)_2TiX_2$ (X = Cl, Br, I), and $(C_5H_4EMe_3)_2$-$TiCl_2$ (E = C, Si, Ge) [11] have been described. The data on zirconium and hafnium derivatives are entirely unavailable.

In the present work, the rearrangement processes of $(C_5H_4Alk)_2ZrCl_2$ under electron impact are considered and the important role of alkyl substituents in the fragmentation character of these compounds is shown. To obtain direct evidence for the participation of hydrogen atoms at the substituent α-carbon atom in the rearrangement processes, the dissociative ionization of the compound $[C_5H_4C(D)Me_2]_2ZrCl_2$ was also studied.

Results and discussion

Directions of dissociative ionization of bis-(η^5-alkylcyclopentadienyl)zirconium dichlorides

Mass spectrometric investigation of bis-(η^5-alkylcyclopentadienyl)zirconium dichlorides $(C_5H_4R)_2ZrCl_2$ (R=H (**I**), Me (**II**), Et (**III**), ${}^nPr$ (**IV**), ${}^iPr$ (**V**), ${}^nBu$ (**VI**), ${}^tBu$ (**VII**)) and $(C_5Me_5)_2ZrCl_2$ (**VIII**) was carried out. The results are given in Table 1. According to the lines of the corresponding ions in the mass spectra and the conversion $M^+ \rightarrow (M-HCl)^+$ for the compounds under study, three different schemes of fragmentation are proposed.

$$\begin{tikzcd}
(M\text{-}L)^+ & M^+ \arrow[l, "-L"'] \arrow[r, "-HCl"] \arrow[d, "-Cl"] & (M\text{-}HCl)^+ \arrow[r, "-HCl"] & (M\text{-}2HCl)^+ \\
 & (M\text{-}Cl)^+ & &
\end{tikzcd} \tag{1}$$

$$\begin{tikzcd}
(M\text{-}L)^{+} & M^{+} \arrow[l, "-L"'] \arrow[r, "-Me"] \arrow[d, "-Cl"] & (M\text{-}Me)^{+} \arrow[r, "-HCl"] & (M\text{-}Me\text{-}HCl)^{+} \\
 & (M\text{-}Cl)^{+} & &
\end{tikzcd} \tag{2}$$

$$(M\text{-}Cl)^{+} \xleftarrow{-Cl} M^{+} \xrightarrow{-L} (M\text{-}L)^{+} \xrightarrow{-L} (M\text{-}2L)^{+} \tag{3}$$

Scheme 1 shows the dissociative ionization of compounds **II–VI**, **VIII**. Comparing of this scheme with scheme 2 for compound **VII** and scheme 3 for compound **I**, it is seen that the elimination of the HCl molecule from the molecular ion takes place only for the compounds that contain substituents with hydrogen atoms at the α-carbon atom in the cyclopentadienyl ligand.

One can suggest that the rearrangement processes with the HCl molecule abstraction are accompanied by the migration of the hydrogen atom at the α-carbon atom in the alkyl group. This process results in the formation of the ion with two ligands ($C_5H_4CR'R''$ ($R{=}CHR'R''$):

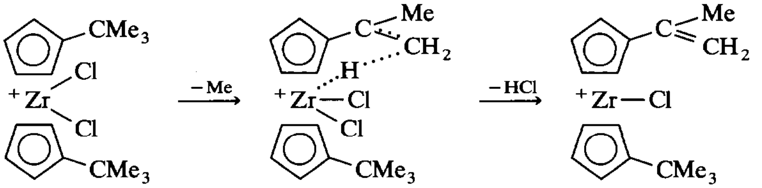
$$(C_5H_4R)_2ZrCl_2^{+} \xrightarrow{-HCl} (C_5H_4CR'R'')(C_5H_4R)ZrCl^{+} \xrightarrow{-HCl} (C_5H_4CR'R'')_2Zr^{+} \tag{4}$$

The role of the hydrogen atoms at the α-carbon atom of the alkyl group was ascertained by us earlier for the fragmentation of the molecular ions $(C_5H_4Alk)_2Ni^{+}$ and $(C_5Me_5)_2M^{+}$ (M = V, Mn, Ni) [12,13]. Moreover, it is known that the rearrangement processes proceeding on heating or photolysis of $(C_5Me_5)_2MR_2$ (M = Ti, Zr; R = Me, Ph) are accompanied by the formation of RH due to the hydrogen atoms in methyl groups [14–17].

Part of the rearrangement process with elimination of the HCl molecule increases with the growing number of carbon atoms in the alkyl substituent of the normal structure, and the largest is seen for the compound with an isopropyl substituent in the ring.

With complete substitution of hydrogens in the cyclopentadienyl ligand by methyl groups, the steric hindrances in low-energy processes with migration of the hydrogen atom to the metal increase sharply. As a result, the rearrangement process with elimination of the HCl molecule is much weaker than expected. The ligand abstraction becomes the predominant route of the fragmentation of compound **VIII** and the line of the ion $(M\text{-}L)^{+}$ is the main line in the spectrum.

The molecular ion of compound **VII** does not eliminate the HCl molecule. The elimination becomes possible only after abstraction of the methyl group from the molecular ion (reaction 2), the α-carbon atom of the substituent leaving the cyclopentadienyl ring plane and the ion with the ligand η^6-$C_5H_4CMe_2$ being formed. The elimination of the HCl molecule with the participation of the hydrogen atom of the methyl group becomes sterically possible. Similar processes with a participating hydrogen atom of the tert-butyl group are observed on the thermolysis of $(C_5H_4{}^tBu)_2ZrPh_2$ [18] and the fragmentation of $Me_2Si(C_5H_4)_2Ti^{+}Br$ [11].

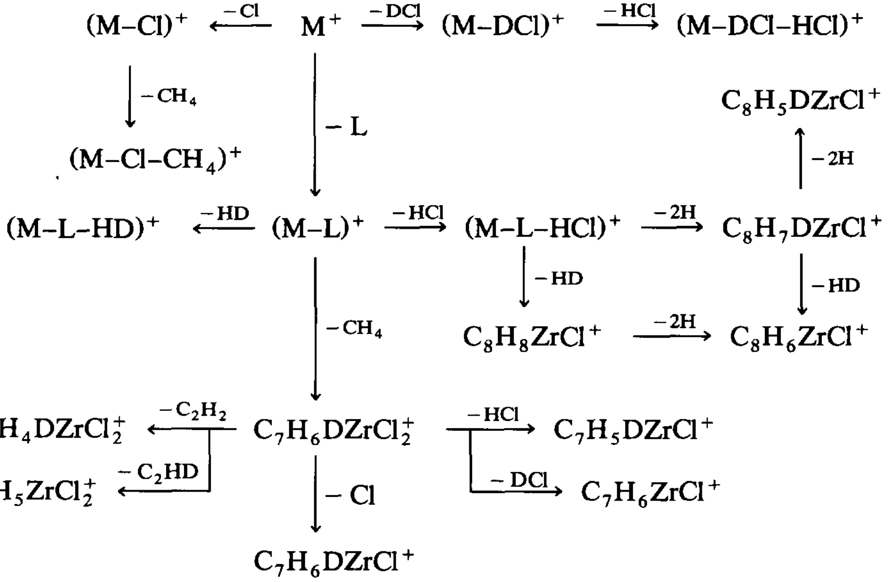


(5)

As the substituent is absent in the ligand, elimination of the HCl molecule in compound **I** does not take place at any stage (scheme 3).

Direct evidence for participation of the hydrogen of the alkyl group at α-carbon atom in rearrangement processes

The mass spectrometric study of the compound $[C_5H_4C(D)Me_2]_2ZrCl_2$ (**IX**) was carried out to solve unambiguously the problem of participation of the hydrogen atoms at the substituent α-carbon atom of the cyclopentadienyl ring in the rearrangement processes. The character of the decomposition processes of compound **IX** and the analogy with the fragmentation of compound **V** are discussed. A more complete spectrum is given in Table 3. Table 4 presents experimental and calculated values of the total content of deuterium in the ions in order to establish a regular change of its dependence on the ion origin. From the data obtained, the fragmentation scheme of compound **IX** was suggested:



The main route of the molecular ion decomposition of compound **IX** is a sequential loss of a DCl molecule and then an HCl molecule (Table 2, *m/z* 339, 303). The elimination of a DCl molecule and the deuterium content in the ion $(M-DCl)^+$, essentially equal to half its content in the molecular ion (Table 4), indicate definitively that the deuterium atom, directly linked with the α-carbon atom relative to the cyclopentadienyl ring, takes part in the formation of the DCl molecule. This is direct proof of the mechanism for the main fragmentation process of alkylzirconocene dichlorides containing the hydrogen at the α-carbon atom. At the first stage, the elimination of the DCl molecule on the molecular ion fragmentation suggests the coordination of the deuterium atom from the substituent α-carbon atom onto the metal ("agostic" bonding Zr · · · D–C). The α-position is sterically convenient for mutual overlap of valent orbitals of metal and hydrogen.

The fact that the ion $(M-DCl)^+$ loses the HCl molecule can be explained in two different ways. The first is the participation of the methyl group of the ligand

Table 2

Mass spectrum of $[C_5H_4C(D)Me_2]_2ZrCl_2$

Ion	m/z	I (%) [a]	Ion	m/z	I (%)
M^+	376	9.4	$(M-L)^+$	268	21.0
	375	0.2		267	0.4
$(M-Cl)^+$	341	7.1	$(M-L-2H)^+$	266	0.8
	340	1.9	$(M-L-HD)^+$	265	12.4
$(M-DCl)^+$	339	100.0	$C_7H_6DZrCl_2^+$	252	14.5
	338	2.7		251	0.2
	336	0.7		250	0.7
$(M-Cl-CH_4)^+$	335	0.3		249	1.9
	325	7.9	$(M-L-HCl)^+$	232	3.2
	324	2.6	$C_8H_7DZrCl^+$	230	14.8
	323	7.2	$C_8H_8ZrCl^+$	229	6.7
	322	1.2	$C_8H_5DZrCl^+$	228	4.3
	321	0.3	$C_8H_6ZrCl^+$	227	5.6
$(M-DCl-HCl)^+$	303	38.9	$C_5H_4DZrCl_2^+$	226	3.4
	302	0.8	$C_5H_4ZrCl_2^+$	225	6.1
	302	3.3		224	0.9
	300	1.9	$C_7H_6DZrCl^+$	217	6.2
	299	0.6	$C_7H_5DZrCl^+$	216	15.2
	298	2.4	$C_7H_6ZrCl^+$	215	4.0
	297	3.1		214	2.6
	296	2.8		213	2.2
	295	7.9			

[a] The relative intensity of each ion line (I, %) is the sum of the line intensities of its isotopic varieties referred to the similar sum of lines for the ion $(M-DCl)^+$ expressed as percentages. Therefore the values of m/z are related arbitrarily to ions containing the most frequently found elements. M^+, molecular ion; L, π-ligand.

Table 3

Mass spectrum of $[C_5H_4C(H)Me_2]_2ZrCl_2$

Ion	m/z	I (%)	Ion	m/z	I (%)
M^+	374	10.7		266	2.5
$(M-Cl)^+$	339	5.4	$(M-L-2H)^+$	265	13.4
$(M-HCl)^+$	338	100.0		264	1.0
	337	6.5		263	0.7
	336	1.2	$C_7H_7ZrCl_2^+$	251	14.1
	335	1.3		250	0.6
	323	8.1		249	1.1
	322	2.0	$(M-L-HCl)^+$	231	2.1
	321	1.0		230	1.3
$(M-2HCl)^+$	302	29.3	$C_8H_8ZrCl^+$	229	20.7
	301	1.0		228	2.2
	300	2.4	$C_8H_6ZrCl^+$	227	9.5
	299	1.8	$C_5H_5ZrCl_2^+$	225	10.0
	298	0.7		224	0.9
	297	4.0	$C_7H_7ZrCl^+$	216	7.0
	296	0.3	$C_7H_6ZrCl^+$	215	18.6
	295	7.8		214	1.9
	267	22.6		213	2.2

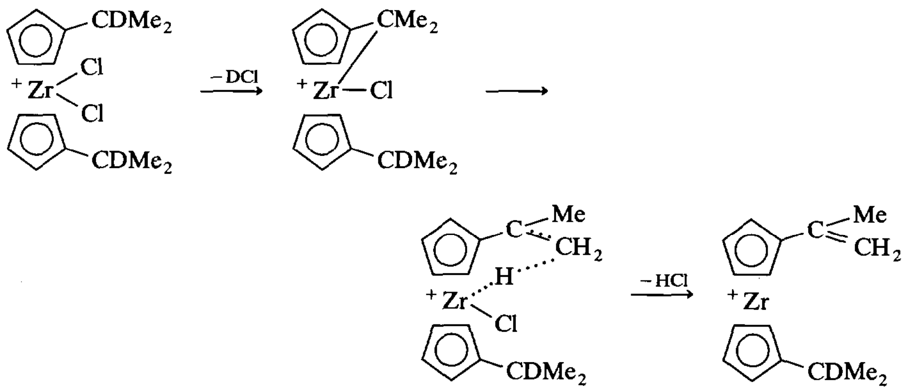
Table 4

The total content of deuterium in ions (D, %)

Ion type	Ion composition	m/z	D (%)	
			Exp.	Calc. [a]
M^+	$C_{16}H_{20}D_2ZrCl_2^+$	376	9.0	9.0
$(M\text{-}Cl)^+$	$C_{16}H_{20}D_2ZrCl^+$	341	9.0	9.0
$(M\text{-}DCl)^+$	$C_{16}H_{20}DZrCl^+$	339	4.6	4.7
$(M\text{-}Cl\text{-}CH_4)^+$	$C_{15}H_{16}D_2ZrCl^+$	325	9.6	11.0
$(M\text{-}DCl\text{-}HCl)^+$	$C_{16}H_{19}DZr^+$	303	4.9	4.9
$(M\text{-}L)^+$	$C_8H_{10}DZrCl_2^+$	268	9.0	9.0
$(M\text{-}L\text{-}2H, HD)^+$	$C_8H_{8-n}D_nZrCl_2^+$	266, 265	0.7	11.0-0.0
$(M\text{-}L\text{-}CH_4)^+$	$C_7H_6DZrCl_2^+$	252	14.1	14.1
$(M\text{-}L\text{-}HCl\text{-}2H, HD)^+$	$C_8H_{8-n}D_nZrCl^+$	230, 229	8.6	12.4-0.0
$(M\text{-}L\text{-}CH_4\text{-}C_2H_2, C_2HD)^+$	$C_5H_{5-n}D_nZrCl_2^+$	226, 225	7.5	19.8-0.0
$(M\text{-}L\text{-}CH_4\text{-}Cl)^+$	$C_7H_6DZrCl^+$	217	14.1	14.1
$(M\text{-}L\text{-}CH_4\text{-}HCl, DCl)^+$	$C_7H_{6-n}D_nZrCl^+$	216, 215	13.2	16.4-0.0

[a] Calculated data for the ions formed on the abstraction of 2H and HD, C_2H_2 and C_2HD, HCl and DCl are the magnitudes within the limits of which the total content of deuterium can change.

liberated D atom to form the DCl molecule as in the case of the fragmentation process observed with compound **VII**.

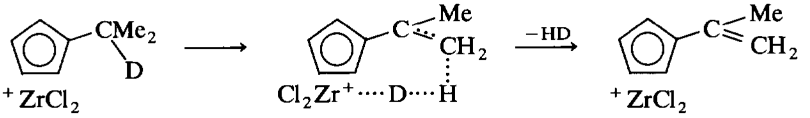


(6)

Another explanation for the elimination of HCl assumes deuterium–hydrogen exchange in the ion (M–DCl)$^+$ followed by abstraction of the hydrogen chloride molecule with participation of the hydrogen atom at the substituent α-carbon atom of the other cyclopentadienyl ring. This mechanism, however, seems to be more probable but does not exclude the contribution of direction 6 into the fragmentation processes.

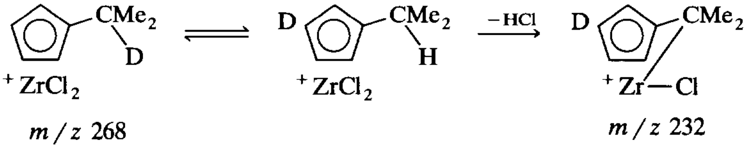
With the deuterium–hydrogen exchange, the ion (M–2DCl)$^+$ has to form to a small degree (Table 2, m/z 302) along with the ion (M–DCl–HCl)$^+$ but registration of the line intensity of this ion is not possible because of the superposition on it of ion lines (Table 2, m/z 302–295) corresponding to a vast successive abstraction of hydrogen atoms from the ion (M–DCl–HCl)$^+$ with m/z 303. The deuterium–hydrogen exchange is completely excluded for the molecular ion and becomes possible only after elimination of the first molecule of hydrogen chloride. The exchange of a deuterium (hydrogen) atom is carried out by interaction of the hydrogen atom with the coordination sphere of the metal followed by its (hydrogen) migration onto the other ligand.

The second significant direction of the molecular ion decomposition is ligand abstraction with the formation of the ion $(M{-}L)^+$ with m/z 268 (Table 2). The ion $(M{-}L)^+$ can be further subjected to fragmentation in three ways. The first is the abstraction of the HD molecule instead of H_2 with the formation of the ion $(M{-}L{-}HD)^+$ with m/z 265. Elimination of the HD molecule is accompanied by the migration of deuterium onto the metal and interaction with the methyl group:



(7)

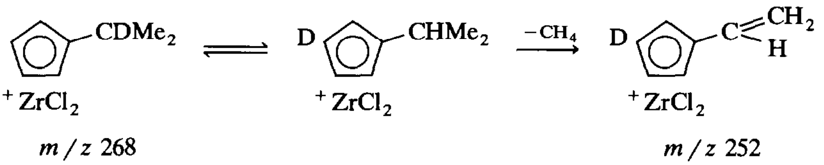
The formation of the ion $(M{-}L{-}HD)^+$ testifies in favour of an initial position of deuterium in the ion $(M{-}L)^+$. But by another route of decomposition, the ion $(M{-}L)^+$ eliminates the HCl molecule rather than DCl as expected. The ions $(M{-}L{-}HCl)^+$ with m/z 232 are apparently formed from the ions $(M{-}L)^+$ with deuterium–hydrogen rearrangement. Because of very low intensity of the ion line with m/z 232, it is impossible to take into account the negligible intensity of the line with m/z 231 corresponding to the DCl molecule abstraction from the ion $(M{-}L)^+$. The ion $(M{-}L{-}HCl)^+$, successively losing the molecules of H_2 and HD, changes into the ions $C_8H_7DZrCl^+$ with m/z 230, $C_8H_8ZrCl^+$ with m/z 229 and $C_8H_5DZrCl^+$ with m/z 228, $C_8H_6ZrCl^+$ with m/z 227 (Table 2). The summary relative intensity of the lines for the ions with m/z 230 and 229 (Table 2) is essentially equal to the relative intensity of the line of the ion $C_8H_8ZrCl^+$ with m/z 229 in the mass spectrum of compound **V** (Table 3). Similarly, the summary relative intensity of the lines for the ions with m/z 228 and 227 (Table 2) is almost equal to the relative intensity of the line for the ion $C_8H_6ZrCl^+$ with m/z 227 (Table 3). The ratio of the relative intensities of the lines for the ions with m/z 230 and 229 is 2:1 and these relative intensity values suggest that the molecules of H_2 or HD are formed due to the abstraction of hydrogen atoms in the cyclopentadiene ring if the ion $(M{-}L{-}HCl)^+$ was formed from the ion $(M{-}L)^+$ with deuterium–hydrogen rearrangement in the ring, including into the exchange sphere also the position at the α-carbon atom. The structures of the ions (m/z 268 and 232) can be presented as follows:



(8)

According to eq. 8, the ion with m/z 232 must contain three hydrogen atoms and one deuterium atom in the ring. It follows that the probability of the formation of H_2 or HD molecules is the same but due to the isotopic-kinetic effect, the abstraction of the HD molecule proceeds twice as slow as that of H_2. As a result, the amount of the ions $C_8H_7DZrCl^+$ formed with m/z 230 is twice that of the ions $C_8H_8ZrCl^+$ with m/z 229.

By the third direction of decomposition, the ion $(M-L)^+$ eliminates the CH_4 molecule, changing into the ion $C_7H_8DZrCl_2$ with *m/z* 252 (Table 2). For compound V, a similar process leads to the formation of the ion $C_7H_7ZrCl_2^+$ (Table 3), *m/z* 251). The actual equality of the relative intensities of the lines for the ions with *m/z* 252 (Table 2) and 251 (Table 3) and the regular change in the total content of deuterium on the conversion of ion $(M-L)^+$ with *m/z* 268 into the ion with *m/z* 251 (Table 3) points to the formation of a methane molecule due to methyl groups in the substituent:



(9)

When eliminating the HCl molecule, the ion with *m/z* 252 having the structure shown in eq. 9, changes into the ion $C_7H_7DZrCl^+$ with *m/z* 216, and while losing the DCl molecule, it changes into the ion $C_7H_6ZrCl^+$ with *m/z* 215. The ratio of the relative intensities of the lines for the ions with *m/z* 216 and 215 (Table 2) meets the condition of deuterium–hydrogen exchange in the ring including the hydrogen (deuterium) at the α-carbon atom. In a mass spectrum of compound V, the total relative intensity of the lines for the ions with *m/z* 216 and 215 is approximately equal to the value of the relative intensity of the line for the ion $C_7H_6ZrCl^+$ with *m/z* 215 (Table 3).

The ion with *m/z* 252 can also decompose with the abstraction of a C_2H_2 or C_2HD molecule giving the ions $C_5H_4DZrCl_2^+$ or $C_5H_5ZrCl_2^+$ (Table 2, *m/z* 226, 225). These decomposition products are also indicative of deuterium–hydrogen exchange in the parent ion.

It should be noted that the ion $C_7H_6DZrCl^+$ with *m/z* 217 has its origin from the ion $C_7H_6DZrCl_2^+$ (*m/z* 252). Both ions have the same content of deuterium (Table 4).

The third direction of the molecular ion decomposition is accompanied by the successive loss of a Cl atom and a CH_4 molecule with the formation of the ion $(M-Cl-CH_4)^+$ with *m/z* 325. The CH_4 molecule is formed due to the substituent methyl groups.

Conclusion

Thus, the alkyl substituent structure determines the direction of the rearrangement processes of the molecular ion. The availability of mobile hydrogen atoms in the substituent at the α-carbon atom accounts for the processes with elimination of the HCl molecule from the molecular ion. This part of the process is the largest for a compound with an isopropyl substituent in the ring.

A straightforward analogy between catalytic processes and the decomposition processes under electron impact cannot always be observed. Nevertheless, in the study of ethylene and propylene polymerization with the system bis(alkylcyclopentadienyl)zirconium dichloride–methylalumoxane, it was established that compounds with an isopropyl substituent in the ring show the largest catalytic activity [6,19,20]. These results require further study.

Experimental

Compounds **I–VIII** were synthesized from the corresponding lithium derivatives of cyclopentadiene and $ZrCl_4$ and purified by double recrystallization from hexane or toluene.

Compound **IX** was prepared via the reaction of $ZrCl_4$ with $[C_5H_4C(D)Me_2]Li$ and purified by recrystallization from heptene. Isopropylcyclopentadiene $C_5H_5C(D)Me_2$ was obtained by the interaction of dimethylfulvene with $LiAlD_4$. The isotopic purity of compound **IX** is (%): d_2 from 98.2; d_1 form, 1.8. The total content of deuterium in the position above is 99.1.

Mass spectra were recorded with a MI-1305 device at electron ionization energy of 70 eV, an emission current of 1.5 mA and accelerating voltage of 2 kV. The introduction of the substance was carried out by placing it directly into the ion source. The sample temperature was 80°C.

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