Inorganic salts: one of the controlling factors of reaction mechanism and complex compound formation
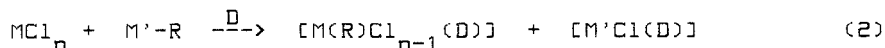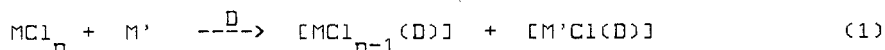
Piotr Sobota

Institute of Chemistry, University of Wrocław,
14, Joliot-Curie, 50-383 Wrocław (Poland)

Abstract –Many chemical reactions, besides the main product, yield also an inorganic salt , e.g $MgCl_2$ or $AlCl_3$. Such a salt was found to react with commonly used transition metal chlorides. Depending on molar ratio, different ionic or molecular complex compounds are formed, so what essentially influences the reaction mechanism? The results of the effect of $MgCl_2$ on Grignard reagent composition, its reactivity and activition of N_2 molecule are presented.

INTRODUCTION

In the synthesis of transition metal compounds in low oxidation state, the most frequently used initial substrates are the transition metal chlorides MCl_n, while the Main Group metals M'or their organometallic compounds are the reducing agents. Ethers or other polar compounds are used as solvents (D).

$$MCl_n + M' \xrightarrow{\ \ D\ \ } [MCl_{n-1}(D)] + [M'Cl(D)] \qquad (1)$$

$$MCl_n + M'-R \xrightarrow{\ \ D\ \ } [M(R)Cl_{n-1}(D)] + [M'Cl(D)] \qquad (2)$$

Depending on the substrates molar ratio different products can be obtained. In reactions 1 and 2, beside transition metal compounds, M'Cl also is formed. Such inorganic salts were thought to be the side-products, not involved in the reaction. So the question arose, whether an inogranic salt formed in reactions 1 or 2 and analogous reactions, is chemically neutral or whether it reacts with either substrate or the products or both. If the answers were affirmative, such a salt could essentially influence the reaction mechanism and composition of its products. The problem will be discussed basing on selected examples of our studies, which comprise mainly the reactions between $MgCl_2$, $AlCl_3$ and some commonly used transition metal chlorides. Magnesium dichloride was chosen because it is formed in reduction reaction of MCl_n with metallic magnesium, and is a component of the Grignard reagent. Another reason was the fact, that catalysts based on magnesium dichloride are very important in the field of polymerization of ethylene and propylene process (ref.1). Instead, $AlCl_3$ as a Lewis acid is used in many reactions and is a component of some Ziegler–Natta catalysts.

## REACTIONS BETWEEN $MgCl_2$ AND MOLYBDENUM CHLORIDES

We have initiated our studies with molybdenum pentachloride $MoCl_5$, used as an initial substrate in synthesis of many complexes e.g. trans-$[Mo(N_2)_2(dppe)_2]$ and other low valent compounds. In such reactions the magnesium turnings are used as a reducing agent (ref.2). It is known that dissolving of $MoCl_5$ in THF can produce $[MoOCl_3(THF)_2]$ and for that reason this compound is used as a substrate.

$$HgCl_2 + Mg \xrightarrow{\ THF\ } [MgCl_2(THF)_2] + Hg \qquad (3)$$
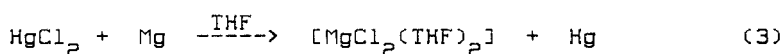
Since $MgCl_2$ is insoluble in THF we have used $[MgCl_2(THF)_2]$, obtained in the reaction 3 (ref.3). The reaction between $[MgCl_2(THF)_2]$ and $[MoOCl_3(THF)_2]$ in in THF proceeds easily. Two different compounds can be obtained depending on the molar ratio of the substrates (ref.4)

$$2[MoOCl_3(THF)_2] + [MgCl_2(THF)_2] \xrightarrow{THF} [Mg(THF)_6][MoOCl_4(THF)]_2 \quad (4)$$
$$\underset{I}{}$$
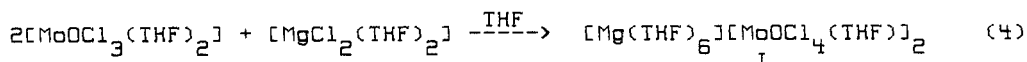
Reaction 4, at 2 : 1 gives rise to the formation of the compound, I. In this reaction the molybdenum atom is a more powerful Lewis acid and removes both chloride atoms from $[MgCl_2(THF)_2]$ to produce $[Mg(THF)_6]^{2+}$ and $[MoOCl_4(THF)]^-$ ions. Compound I reacts further with $[MgCl_2(THF)_2]$ to produce compound II.

$$I + 3[MgCl_2(THF)_2] \xrightarrow{THF} 2[Mg_2(\mu-Cl)_3(THF)_6][MoOCl_4(THF)] \quad (5)$$
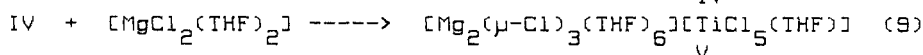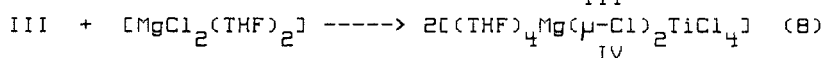$$\underset{II}{}$$
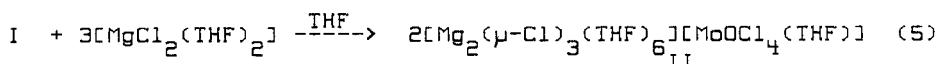
I and II contain the same $[MoOCl_4(THF)]^-$ anions. The difference lies in cations. In reaction 5, the hexakis(tetrahydrofuran)magnesium(II) reacts with three equivalents of $[MgCl_2(THF)_2]$ to yield two $[Mg_2(\mu-Cl)_3(THF)_6]^+$ cations, and $[MoOCl_4(THF)]^-$ anion remains probably unchanged.

$$[Mg(THF)_6]^{2+} + 3[MgCl_2(THF)_2] \longrightarrow 2[Mg_2(\mu-Cl)_3(THF)_6]^+ \quad (6)$$
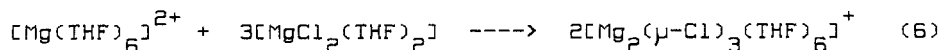
## REACTIONS BETWEEN MgCl₂ AND TITANIUM CHLORIDES

We have carried out also the series of reactions between $[TiCl_4(THF)_2]$ and $[MgCl_2(THF)_2]$ to explain the unique role of $MgCl_2$ as a component of catalyst in α-olefin polymerization process. Three different complex compounds may be obtained depending upon molar ratio in which they are combined (refs.5 & 6).

$$[MgCl_2(THF)_2] + 2[TiCl_4(THF)_2] \xrightarrow{THF} [Mg(THF)_6][TiCl_5(THF)]_2 \quad (7)$$
$$\underset{III}{}$$

$$III + [MgCl_2(THF)_2] \longrightarrow 2[(THF)_4Mg(\mu-Cl)_2TiCl_4] \quad (8)$$
$$\underset{IV}{}$$

$$IV + [MgCl_2(THF)_2] \longrightarrow [Mg_2(\mu-Cl)_3(THF)_6][TiCl_5(THF)] \quad (9)$$
$$\underset{V}{}$$

Equation 7, with $MgCl_2: TiCl_4 = 1 : 2$ gives compound III. The titanium atom exhibits the acid properties because it removes chlorine atoms from magnesium dichloride to give the $[Mg(THF)_6]^{2+}$ cation and $[TiCl_5(THF)]^-$ anions. The molecular compound IV, is formed in reaction 8. The accumulation of negative charge associated with the coordination of six chlorides around Ti^{4+} causes its acid properties decrease. Consequently, the chlorides became more basic and dislodge the THF molecules from the coordination sphere of solvated magnesium cation. This allows the formation of a heterobimetallic molecular compound IV. In this context the acid and basic properties of both metal atoms in IV have to be similar. In eq. 9, between compound IV and $[MgCl_2(THF)_2]$ the magnesium dichloride also shows basic properties because it removes the $MgCl^+$ cation from $[(THF)_4Mg(\mu-Cl)_2TiCl_4]$ to produce the $[Mg_2(\mu-Cl)_3(THF)_6]^+$ cation and $[TiCl_5(THF)]^-$ anion (ref.6)

$$[MgCl_2(THF)_2] + [MgCl(THF)_5]^+ \xrightarrow{-THF} [Mg_2(\mu-Cl)_3(THF)_6]^+ \quad (10)$$
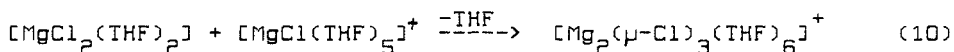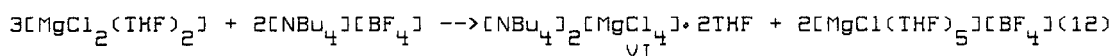
The above results comprise mainly the mutual interaction of $MgCl_2$ and $TiCl_4$. However, in the polymerization process the active catalyst center is widely regarded as Ti^{3+}. For that reason we have examined the series of reactions between $[MgCl_2(THF)_2]$, $AlCl_3$ and $[TiCl_3(THF)_3]$, under various conditions. These compounds do not react mutually. Instead, $[TiCl_3(THF)_3]$ in reaction with $ZnCl_2$ forms $[$ trans-$TiCl_2(THF)_4][ZnCl_3(THF)]$ compound (ref.7).

$$[TiCl_3(THF)_3] + ZnCl_2 \xrightarrow{THF} [trans-TiCl_2(THF)_4][ZnCl_3(THF)] \quad (11)$$
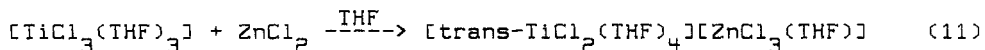
In a similar reaction with $SnCl_4$ the $[trans-TiCl_2(THF)_4][SnCl_5(THF)]$ compound is
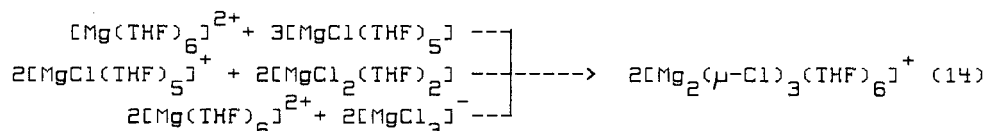
formed (ref.8). The understanding of the unusual role of $MgCl_2$ and other in-organic salts required explanation, why $[TiCl_3(THF)_3]$ reacted with $ZnCl_2$ or $SnCl_4$ but not with $MgCl_2$ and $AlCl_3$? $AlCl_3$ is more powerful Lewis acid than $ZnCl_2$. Nevertheless ,it does not form the compound with $[TiCl_3(THF)_3]$. It seems most likely, that the course of the reactions discussed here depends first of all upon equilibria formed in a solution. The $[TiCl_3(THF)_3]$ and $ZnCl_2$ or $SnCl_4$ undergo ionization in THF. The ions in solution would react mutually. The equilibrium in solution would depend upon the least soluble species formed in solution. From our data and others (ref.9) it follows that the $[MCl_5(THF)]^-$ (M =Ti, Zr, Sn or Hf) type anions form with cations in THF the least soluble compounds which settled down in a solution and cause the shift of the reaction equilibrium. Instead, in the $[TiCl_3THF)_3]$, $[MgCl_2(THF)_2]$ or $AlCl_3$ tetrahy-drofuran mixture the least soluble is $[TiCl_3(THF)_3]$. Additional confirmation of this conclusion is the reaction between $[MgCl_2(THF)_2]$ and $[NBu_4][BF_4]$ salts (ref.10a).

$$3[MgCl_2(THF)_2] + 2[NBu_4][BF_4] \longrightarrow [NBu_4]_2[\underset{VI}{MgCl_4}] \cdot 2THF + 2[MgCl(THF)_5][BF_4] \quad (12)$$

$[NBu_4]^+$ cation exists only in the ionic form and its direct interaction with bis(tetrahydrofuran)magnesium dichloride was not expected. Theoretically, the $[BF_4]$ anion could form different compounds with Mg^{2+}. Such a possibility, how-ever, was ^{19}F NMR excluded . This indicates that the course of reaction 12 can thus be explained only as the ionization of $[MgCl_2(THF)_2]$ in THF (ref 10a).

$$3[MgCl_2(THF)_2] \underset{\overleftarrow{}}{\overset{THF}{\rightleftharpoons}} 2[MgCl(THF)_5]^+ + [MgCl_4]^{2-} \quad (13)$$
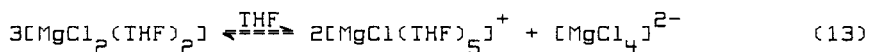
The conductivity of $[MgCl_2(THF)_2]$ in THF is low and equal to $0.5\mu S$. This is indicative of small amount of ions in solution. Addition to bis(tetrahydro-furan)magnesium dichloride $[NBu_4][BF_4]$ in THF results in precipitation of the least soluble ionic compound VI (eq.12) followed by a shift in equilibrium of reaction to the right. Equation 13 shows the simplified situation. The ions which remain in solution would then react mutually and/or with substrate.

$$\left. \begin{array}{r} [Mg(THF)_6]^{2+} + 3[MgCl(THF)_5] \longrightarrow \\ 2[MgCl(THF)_5]^+ + 2[MgCl_2(THF)_2] \longrightarrow \\ 2[Mg(THF)_6]^{2+} + 2[MgCl_3]^- \longrightarrow \end{array} \right\} \longrightarrow 2[Mg_2(\mu\text{-}Cl)_3(THF)_6]^+ \quad (14)$$

The $[MgCl_4]^{2-}$ anion was found to react easily with $TiCl_4$ and $TiCl_3$.

$$2[TiCl_4(THF)_2] + VI \xrightarrow{THF} [NBu_4]_2[\underset{VII}{TiCl_6}] + [MgCl_2(THF)_2] \quad (15)$$
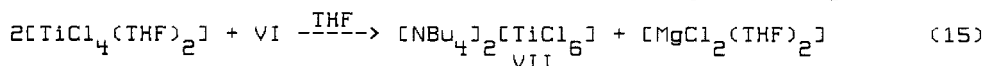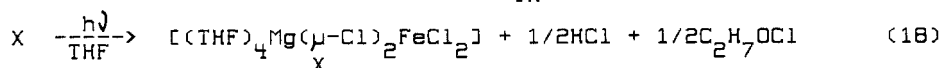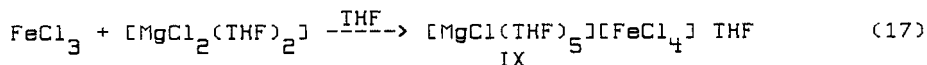
Yellow compound VII is unstable and under influence of light in THF solution it undergoes reduction to blue $[NBu_4][trans\text{-}TiCl_4(THF)_2] \cdot THF$, $VIII$, and simul-taneously a white $[NBu_4][Cl]$ salt precipitates.

$$VII \xrightarrow[THF]{h} [NBu_4][trans\text{-}TiCl_4(THF)_2] + [NBu_4][Cl] + 1/2HCl + 1/2C_4H_7OCl \quad (16)$$
$$\underset{VIII}{}$$
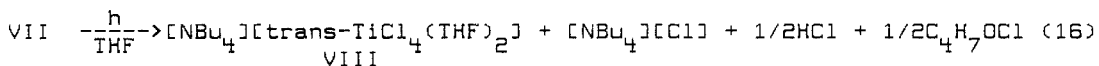
In post-reaction mixture, HCl and C_4H_7OCl were MS/GL detected. In $VIII$, titanium atom exists as Ti^{3+}, which points to the reduction of titanium Ti^{4+} (in VII) to Ti^{3+} (in $VIII$) under influence of light and THF. Similar effect was observed during reaction 18.
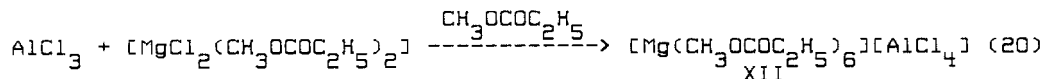
REACTIONS BETWEEN MgCl₂ AND FeCl₃ OR AlCl₃

It was found, that among the trivalent transition metal chlorides only $FeCl_3$ reacted with $MgCl_2$ to form an ionic compound IX. This compound is unstable. Under influence of light the Mg-Cl bond undergoes splitting and the iron Fe^{3+} is reduced to Fe^{2+}. As a result the molecular complex compound X is formed. The existence of $MgCl^+$ cation was earlier frequently postulated. However, only now it was evidenced by X-ray method in compound,IX,(refs.11 &12).To find out whether the $MgCl^+$ cation would arise also in other reactions, the similar re-

$$FeCl_3 + [MgCl_2(THF)_2] \xrightarrow{THF} \underset{IX}{[MgCl(THF)_5][FeCl_4]} \; THF \qquad (17)$$

$$X \xrightarrow{\frac{h\nu}{THF}} \underset{X}{[(THF)_4Mg(\mu-Cl)_2FeCl_2]} + 1/2HCl + 1/2C_2H_7OCl \qquad (18)$$

action was carried out with $AlCl_3$. Aluminium trichloride, like $FeCl_3$, easily produced $[AlCl_4]^-$. During this reaction, the new compound XI, similar with IX

$$AlCl_3 + [MgCl_2(THF)_2] \xrightarrow{THF} \underset{XI}{[MgCl(THF)_5][AlCl_4]} \cdot THF \qquad (19)$$
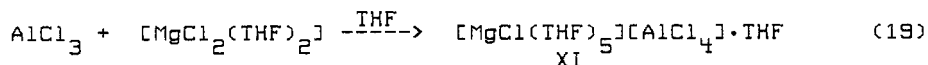
compound was obtained, in which $[MgCl(THF)_5]^+$ cation also existed. Interesting was the formation of compound, XII, when the same reaction proceeded in ethyl acetate. In that compound beside of $[AlCl_4]^-$ exists also $[Mg(CH_3OCOC_2H_5)_6]^{2+}$ cation (ref.13).

$$AlCl_3 + [MgCl_2(CH_3OCOC_2H_5)_2] \xrightarrow{CH_3OCOC_2H_5} \underset{XII}{[Mg(CH_3OCOC_2H_5)_6][AlCl_4]} \quad (20)$$

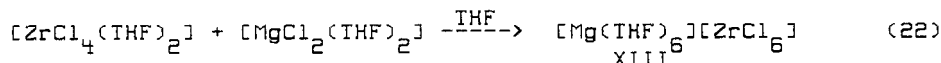## REACTION BETWEEN [ZrCl₂(THF)₄] AND THE GRIGNARD REAGENT

The reported results proved that the inorganic salts could react with substrates and with the reaction products. Now are presented some examples to show how $[MgCl_2(THF)_2]$ could control the reaction course. It is well known that in reaction between the Grignard reagent and $[ZrCl_4(THF)_2]$ the organometallic compound ZrR_4 is formed, along with magnesium dichloride (refs.14-16).

$$4[Mg(R)Cl(THF)_x] + [ZrCl_4(THF)_2] \xrightarrow[258K]{THF} ZrR_4 + 4[MgCl_2(THF)_2] \qquad (21)$$
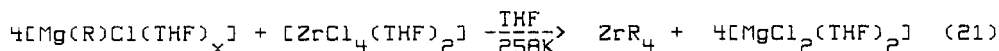
When Grignard reagent is slowly added to $[ZrCl_4(THF)_2]$ at a 4/1 molar ratio in THF compound $[Mg(THF)_6][ZrCl_6]$, XIII, separates out (yield 58%). The composition of XIII suggests that it is formed by a secondary reaction between the substrate $[ZrCl_4(THF)_2]$ and the product $[MgCl_2(THF)_2]$, (ref. 17).

$$[ZrCl_4(THF)_2] + [MgCl_2(THF)_2] \xrightarrow{THF} \underset{XIII}{[Mg(THF)_6][ZrCl_6]} \qquad (22)$$

It follows then, that eq.21 describes only the first stage of the reaction between the Grignard reagent and $[ZrCl_4(THF)_2]$ but does not include the formation of compound XIII. When the yield of compound XIII is taken into account, the reaction can be written as follows;

$$4[Mg(R)Cl(THF)_x] + 2[ZrCl_4(THF)_2] \xrightarrow[258K]{THF} ZrR_4 + XIII + 3[MgCl_2(THF)_2] \quad (23)$$
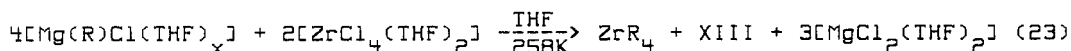
The yield of XIII and the course of reaction depend on the sequence of addi -
tion of substrates, e.g. when Grignard reagent is replaced by $[MgR_2(THF)_2]$ in the reaction XIII is formed with 54% yield, only when $[MgR_2(THF)_2]$ is added to $[ZrCl_4(THF)_2]$.

$$2[MgR_2(THF)_2] + 2[ZrCl_4(THF)_2] \xrightarrow[258K]{THF} 2ZrR_4 + XIII + [MgCl_2(THF)_2] \quad (24)$$
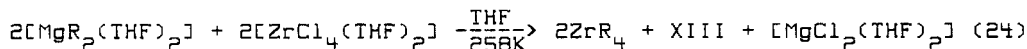
However, when zirconium tetrachloride is added to $[MgR_2(THF)_2]$, ZrR_4 is formed, and compound XIII is not.

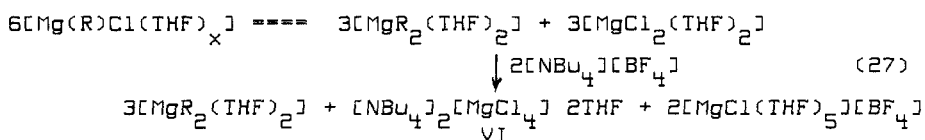$$[ZrCl_4(THF)_2] + 2[MgR_2(THF)_2] \xrightarrow[258K]{THF} ZrR_4 + [(THF)_4Mg(\mu-Cl)_2MgR_2] \quad (25)$$
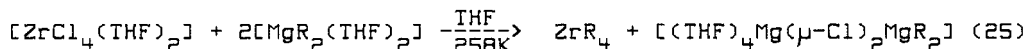
This is probably due to the fact that $[MgCl_2(THF)_2]$ formed in this reaction, reacts immediately with $[MgR_2(THF)_2]$ to produce compound $[(THF)_4Mg(\mu-Cl)_2MgR_2]$ This compound has not been isolated in the pure state although its formation has been postulated (refs. 18-20).

INFLUENCE OF MgCl₂ ON GRIGNARD REAGENT COMPOSITION

The nature of the Grignard reagents "RMgX" in solution is not simple. They exist in an equilibrium mixture which may involve various mono-,bi- and poly-nuclear solvated compounds (refs.18-20), e.g. in tetrahydrofuran the equilibrium is expressed simply, as follows:

$$2[Mg(R)X(THF)_x] \rightleftharpoons [MgR_2(THF)_2] + [MgX_2(THF)_2] \qquad (26)$$
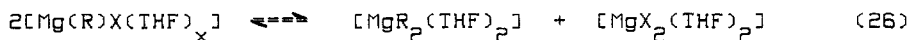
Ease of formation of $[MgCl(THF)_5]^+$, $[Mg(THF)_6]^{2+}$, $[Mg_2(\mu-Cl)_3(THF)_6]^+$ and $[MgCl_4]^{2-}$ ions and molecular compounds from $[MgCl_2(THF)_2]$ by the action of acids in THF, suggests that such species and similar one could be also formed by reaction of Grignard reagent with other substrates leading to the intermidiates in the formation of MgX_2 (eq.26).It was interesting to explain whether $[MgCl_2(THF)_2]$, a component of the Grignard reagent, undergoes ionization and thus behaves as in reaction 12. It was expected that addition of $[NBu_4][BF_4]$ to the Grignard reagent in THF would result in liberation of compound VI and that only $[MgR_2(THF)_2]$ and $[MgCl(THF)_5][BF_4]$ would remain in post-reaction mixture.

$$6[Mg(R)Cl(THF)_x] \rightleftharpoons 3[MgR_2(THF)_2] + 3[MgCl_2(THF)_2]$$

$$\downarrow 2[NBu_4][BF_4] \qquad (27)$$

$$3[MgR_2(THF)_2] + \underset{VI}{[NBu_4]_2[MgCl_4]} \cdot 2THF + 2[MgCl(THF)_5][BF_4]$$

It was found, that compound VI was really formed (ref.21). The reaction 27 with $[NBu_4][BF_4]$ salt confirmed the new equilibrium in Grignard reagent:

$$6[Mg(R)Cl(THF)_x] \rightleftharpoons 3[MgR_2(THF)_2] + [MgCl_4]^{2-} + 2[MgCl(THF)_5]^+ \quad (28)$$
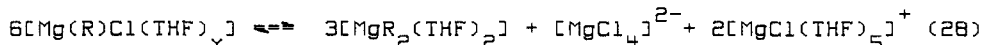
Evidence for the existence of the Schlenk equlibrium 26 is given by the reaction with dioxane leading to the formation of $[MgCl_2(C_4H_8O_2)_2]$, together with $[MgR_2(THF)_2]$ (ref.22).The Schlenk equilibrium and equilibrium 28 should be considerted as particular example. The equilibrium attained in solution would depend upon the least soluble species formed in solution with given substrate.

INFLUENCE OF MgCl₂ ON THE N₂ REDUCTION PROCESS

When $[TiCl_4(THF)_2]$ is reduced by magnesium turnings under N_2 in THF, a material formulated as $TiNMg_2Cl_2(THF)$, XIV, is formed (refs.5 & 23).

$$2[TiCl_4(THF)_2] + 6Mg + N_2 \xrightarrow{THF} 2\underset{XIV}{TiNMg_2Cl_2(THF)} + 2[MgCl_2(THF)_2] \quad (29)$$
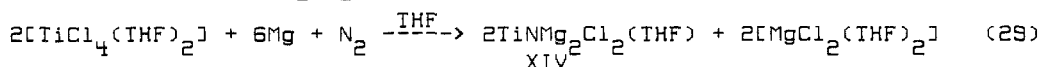
Hydrolysis of species XIV liberates a stoichiometric amount of NH_3. This is indicative of splitting of nitrogen-nitrogen triple bond and the formation of mononitrogen species. Hence it should be expected that the nitrogen atom in XIV would appear in one of three possible forms: nitride, amide, imide or as a mixture of all three. In reaction 29 also the magnesium dichloride arises, what indicates that this reaction in reality, proceeds in Lewis acid medium and it would be expected that the nitrogen atom in species XIV has amide character $Ti-N(MgCl)_2(THF)$. If it really is so,it could have been expected that the insertion reaction of carbon monoxide into the titanium-nitrogen bond would be possible.
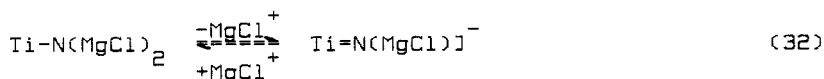
$$\underset{XIV}{Ti-N(MgCl)_2(THF)} + CO \xrightarrow{THF} [A] \xrightarrow{CH_3I} \underset{73\%}{(CH_3)_2NCOCH_3} \quad (30)$$

Species XIV in reaction 30 yields the product [A] which in subsequent reaction with CH_3I produced 73% $(CH_3)_2NCOCH_3$, i.e.the expected compound with amide group. To obtain additional confirmation of the amide character of the nitrogen atom, species XIV was oxidized with stoichiometric amount of I_2,to transform the amide character of the nitrogen atom into imide one. The reaction

between diiodine and species XIV proceeds as follows:

$$Ti-N(MgCl)_2 + 1/2I_2 \xrightarrow[-MgClI]{} T=N(MgCl) \xrightarrow{CO} Ti(NCO)MgCl \quad (31)$$
$$XV$$
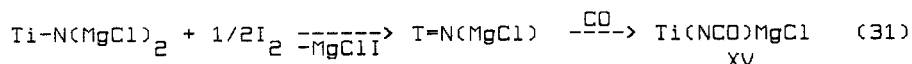
For simplification, the coordinated THF molecules were omitted. Compound XV entered into stoichiometric reaction with CO to yield the isocyanate compoud XV with (ν(NCO) 2197cm^{-1}) what is indicative of imide character of nitrogen atom in XV. The oxidation reaction of XIV by I_2 follows most probably the pattern of amide deprotonation or imide protonation (ref.24)

$$Ti-N(MgCl)_2 \underset{+MgCl^+}{\overset{-MgCl^+}{\rightleftharpoons}} Ti=N(MgCl)]^- \quad (32)$$

$$M-NH_2 \underset{+H^+}{\overset{-H^+}{\rightleftharpoons}} M=NH]^- \quad (33)$$

The presented results proved that the N_2 fixation process proceeds in the acid medium created by the magnesium dichloride (reaction 29) and it is now obvious that the nitrogen atom in $TiNMg_2Cl_2(THF)$ has to have amide character.

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