

Ionic Hydrogenation of Carbonyl Groups With Molybdenum and Tungsten Complexes

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몰리브덴과 텅스텐 착물을 이용한 카르보닐기의 이온성 수소화 반응

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Abstract Metalhydrides such as $\text{Cp}(\text{CO})_2(\text{L})\text{MH}$ ($\text{L} = t\text{-butylisocyanide}$ and $2,6\text{-dimethylphenylisocyanide}$ $\text{M} = \text{Mo}$ and W) have been synthesized and used for ionic hydrogenation of the carbonyl groups in the presence of triflic acid. When these complexes have also used as catalyst precursors for hydrogenation of 3-pentanone under mild conditions (23°C , $<4.1\text{ atm H}_2$). The turnover rates were very slow, with the fastest initial rate of about 2 turnovers per 1 day for the $[\text{Cp}(\text{CO})_2(\text{ArNC})\text{Mo}][\text{BAR}^{\text{F}}_4]$ system.

요약 CO와 유사한 분자궤도함수를 갖는 isocyanide 리간드가 포함된 $\text{Cp}(\text{CO})_2(\text{L})\text{MH}$ ($\text{L} = t\text{-butylisocyanide}$ and $2,6\text{-dimethylphenylisocyanide}$ $\text{M} = \text{Mo}$ and W)를 합성하고, 이 착물과 triflic acid를 사용하여 카르보닐기를 알코올로 전환시키는 실험을 하였다. 또한, 이들 착물을 촉매 전구체로 이용하여 3-pentanone의 수소화 반응을 온화한 조건 (23°C , $<4.1\text{ atm H}_2$)에서 수행하였다. 촉매 활성은 매우 낮았으며, $[\text{Cp}(\text{CO})_2(\text{ArNC})\text{Mo}][\text{BAR}^{\text{F}}_4]$ 의 경우에 2 turnovers/day의 결과를 나타내었다.

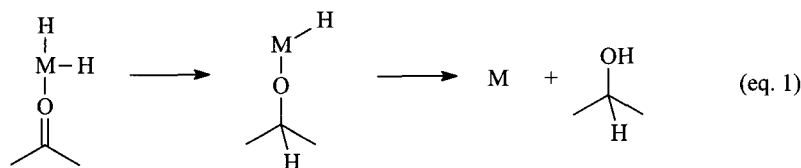
Key Words : Metal Hydride Complex, Ionic Hydrogenation, Isocyanide Ligand.

1. Introduction

Hydrogenation of carbonyl groups to alcohol is a reaction of fundamental importance and practical utility, being used in the synthesis of fine chemicals as well as compounds used in the pharmaceutical and agricultural industries. Main group hydride reagents such as NaBH_4 and LiAlH_4 are often used for the reduction of $\text{C}=\text{O}$ bonds; these reactions are well developed and immensely

useful, but they consume stoichiometric amounts of the hydride reagent and produce waste by-products.

Many transition metal homogeneous hydrogenation catalysts have been developed. The traditional mechanism of these hydrogenations involves coordination of the carbonyl group to the metal, followed by insertion of the carbonyl into the metal-hydrogen bond as shown in generalized in eq. 1 for the case of ketone hydrogenation. The alcohol product is released from the metal through a



reductive elimination reaction involving the second metal hydride bond.^[1]

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An alternative hydrogenation method was reported Kursanov and coworkers over 30 years ago. An authoritative review published in 1974 documented the utility of this process for stoichiometric hydrogenation of a variety of C=C and C=O bonds.^[2] They also showed that delivery of H₂ to the carbonyl could be achieved through ionic hydrogenation that did not require coordination of the carbonyl group to the metal. The versatile reactivity patterns of metal hydrides suggest the possibility of using a metal hydride bond as a source of protons, and a second metal hydride bond as a hydride donor in ionic hydrogenations.^[3] The development of [CpM(CO)₂(PR₃)(n¹-ketone)]⁺ (M = Mo, W; R = Cyclohexyl, Phenyl, Methyl) as catalyst precursors for the homogeneous hydrogenation of ketones was presaged by studies of two of the key individual steps of the reaction transfer^[4] and hydride transfer reactions of metal hydrides,^[5] as well as demonstrations of stoichiometric hydrogenations using metal hydrides.^[6] This demonstrates a connection between kinetic and mechanistic studies of fundamental reactivity patterns and the evolution of the knowledge gained from such studies into the development of new catalysts.

Improved catalysts will be sought that exhibit higher activities and longer lifetimes than those studied thus far. Mechanistic information implies that an improved catalyst should have sufficient steric bulk of the phosphine ligand to destabilize ketone binding. At the same time, increasing the acidity of the cationic metal dihydride is desired since protonation of a ketone by the dihydrides [CpM(CO)₂(PR₃)(H)₂]⁺ is thermodynamically unfavorable.^[7] A comprehensive study of the kinetics of hydride transfers showed that substitution of one CO ligand by a phosphine can dramatically affect the kinetic hydricity of the metal hydride. The electronic effect of the phosphine is to make the metal center more electron-rich, and this effect is manifested in the greatly enhanced kinetic hydricity of the phosphine-substituted metal hydride. The electronic effect clearly predominates over the steric effect of the phosphine.^[8]

We had studied ionic hydrogenations of ketones with new series of complexes such as CpM(CO)(R₂PCH₂CH₂PR₂)H (M = Mo, W; R = 3,5-(CF₃)₂C₆H₃, C₆F₅) because partially phosphine ligands appeared to have precisely the properties that are

required: they would be sterically demanding to suppress ketone binding to the metal, yet concurrently the electron-withdrawing substituents would help to enhance the acidity of the complex. We expected to find that ketones and aldehydes could be easily hydrogenated by HOTf and Cp(CO)(R₂PCH₂CH₂PR₂)MH to give free alcohol and the metal triflate complex Cp(CO)(R₂PCH₂CH₂PR₂)MOTf. However, the reaction of propionaldehyde with HOTf and Cp(CO)(R₂PCH₂CH₂PR₂)MH gave Cp(CO)(R₂PCH₂CH₂PR₂)MOTf and H₂. The hydrogenated organic product, 1-propanol, was not observed, indicating that the reaction of protonated aldehyde with Cp(CO)(R₂PCH₂CH₂PR₂)MH was sterically hindered.^[9]

Since isocyanide and CO are isoelectronic, their molecular orbital descriptions are very similar and more sterically demanding substituents can also be introduced to the isocyanide carbon. We report here the preparation of isocyanide-substituted hydride complexes of molybdenum and tungsten, and a study of their use in hydrogenation of carbonyl groups by an ionic mechanism.

2. Experimental Section

General Procedures. All manipulations were carried out under an atmosphere of argon using Schlenk or vacuum-line techniques, or in a Vacuum Atmospheres drybox. Organic reagents were obtained commercially and were used without purification. Cp(CO)₃MoH and Cp(CO)₃WH were prepared by literature methods.^[10] ¹H and ¹³C NMR spectra were recorded on a Bruker AM-3000 spectrometer; chemical shifts were referenced to the residual proton peak of CD₂Cl₂ at δ 5.32. IR spectra were recorded on a Mattson Polaris FT-IR spectrometer with 0.1 mm CaF₂ solution cells. Elemental Analyses were measured by using EA 1108 Elmer CHNS-O.

Preparation of Cp(CO)₂(ArNC)MoH. Cp(CO)₃MoH (492 mg, 2 mmol) was dissolved in 5 mL of hexane and 2,6-dimethylphenylisocyanide (262 mg, 2 mmol) was added under vigorous stirring, and an immediate CO evolution was observed. The reaction mixture was stirred for 30 min at room temperature. The resulting yellowish white precipitate was isolated by filtration and washed

with hexane (3 mL x 2). The product was dried at room temperature for 6 hours in vacuo to Cp(CO)₂(ArNC)MoH (635 mg, 91%).

¹H NMR (CD₂Cl₂): δ 7.03 (m, 3H, aryl), 5.28 (s, 5H, Cp), 2.32 (s, 6H, 2 CH₃), -5.52 (s, 1H, MoH).

¹³C{¹H} NMR (CD₂Cl₂): δ 231.4 (CO), 186.9 (N≡C), 134.4, 129.7, 128.3, 128.0 (aryl), 89.4 (Cp), 19.1 (CH₃).

IR (CD₂Cl₂): ν 2087 (N≡C), 1958(s), 1906(vs) cm⁻¹.

Anal. Calcd for C₁₆H₁₅NO₂Mo: C, 55.02; H 4.33. Found: C, 55.13; H, 4.21.

Preparation of Cp(CO)₂(t-BuNC)MoH. Cp(CO)₃WH (520 mg, 2.11 mmol) was dissolved in 5 mL of hexane.

To this was added t-butyliisocyanide (250 μL, 2.21 mmol) was added under vigorous stirring in the absence of light. The reaction mixture was stirred for 10 min at room temperature. The resulting yellowish white precipitate was isolated by filtration and washed with hexane (3 mL x 2). The product was dried at room temperature for 6 hours in vacuo to Cp(CO)₂(t-BuNC)WH (564 mg, 89%).

¹H NMR (CD₂Cl₂): δ 5.21 (s, 5H, Cp), 1.47 (s, 9H, t-Bu), -5.70 (s, 1H, MoH).

¹³C{¹H} NMR (C₆D₆): δ 232.3 (CO), 174.1 (N≡C), 88.7(Cp), 58.3 (ipso- C of t-Bu), 30.8 (CH₃).

IR (hexane): ν 2071 (N≡C), 1958(s), 1900(vs) cm⁻¹.

Anal. Calcd for C₉H₁₅NO₂Mo: C, 35.89; H 5.02. Found: C, 35.80; H, 5.13.

Preparation of Cp(CO)₂(ArNC)WH. Cp(CO)₃WH (167 mg, 0.5 mmol) was dissolved in 5 mL of hexane. To this was added 2,6-dimethylphenylisocyanide (66 mg, 0.5 mmol) was added under vigorous stirring, and an immediate CO evolution was observed. The reaction mixture was stirred for 15 hours at room temperature. The solvent was reduced to ~ 1 mL and the resulting yellowish white precipitate was isolated by filtration and washed with hexane (2 mL x 2). The product was dried at room temperature for 6 hours in vacuo to Cp(CO)₂(ArNC)WH (160 mg, 73%).

¹H NMR (CD₂Cl₂): δ 7.09 (s, 3H, aryl), 5.41 (s, 5H, Cp), 2.39 (s, 6H, 2 CH₃), -7.09 (s, 1H, WH).

¹³C{¹H} NMR (CD₂Cl₂): δ 231.4 (CO), 186.9 (N≡C), 134.4, 129.7, 128.3, 128.0 (aryl), 89.4 (Cp), 19.1 (CH₃).

IR (hexane): ν 2076 (N≡C), 1949(s), 1900(vs), 1872(W-H) cm⁻¹.

Anal. Calcd for C₁₆H₁₅NO₂W: C, 43.96; H 3.46. Found: C, 44.32; H, 3.23.

Preparation of Cp(CO)₂(t-BuNC)WH. Cp(CO)₃WH (340 mg, 1.02 mmol) was dissolved in 5 mL of hexane. To this was added t-butyliisocyanide (120 μL, 1.06 mmol) was added under vigorous stirring in the absence of light. The reaction mixture was stirred for 3 hours at room temperature. The solvent was reduced to ~ 1 mL and the resulting yellowish white precipitate was isolated by filtration and washed with hexane (5 mL). Sublimation at 0.5 mtorr/70°C yielded Cp(CO)₂(t-BuNC)WH (370 mg, 93%).

¹H NMR (CD₂Cl₂): δ 5.27 (s, 5H, Cp), 1.46 (s, 9H, t-Bu), -7.36 (s, 1H, WH).

¹³C{¹H} NMR (C₆D₆): δ 232.3 (CO), 174.1 (N≡C), 87.1(Cp), 58.6 (ipso- C of t-Bu), 31.0 (CH₃).

IR (CH₂Cl₂): ν 2059 (br, w, N≡C), 1948(s), 1893(vs) cm⁻¹.

Anal. Calcd for C₉H₁₅NO₂W: C, 27.78; H 3.89. Found: C, 27.55; H, 3.93.

Ionic Hydrogenation of propionaldehyde using Cp(CO)₂(ArNC)MoH and HOTf. Cp(CO)₂(ArNC)MoH (12 mg, 34 μmol) and propionaldehyde (3 μL, 41 μmol) were placed in an NMR tube and CD₂Cl₂ (0.67 mL) was added. After adding HOTf (4 μL, 46 μmol), the tube was shaken for 5 min at room temperature. The ¹H NMR spectrum of the resulting red-brown solutions showed the formation of an alcohol complex (~25%) along with 1-propanol (~33%) and the unreacted propionaldehyde (~42% based on the starting material).

¹H NMR of [Cp(CO)₂(ArNC)Mo(1-propanol)]OTf in CD₂Cl₂: δ 5.79 (s, 5H, Cp), 3.82 (t, J = 6.8Hz, OCH₂), 1.68 (m, CH₂), 0.94 (t, J = 7.4Hz, CH₃).

Ionic Hydrogenation of propionaldehyde using Cp(CO)₂(t-BuNC)MoH and HOTf. Cp(CO)₂(t-BuNC)MoH (10 mg, 33 μmol) and propionaldehyde (3 μL, 41 μmol) were placed in an NMR tube and CD₂Cl₂ (0.70 mL) was added. After adding HOTf (4 μL, 46 μmol), the tube was shaken for 5 min at room temperature. The ¹H NMR spectrum of the resulting red-brown solution showed the formation of an alcohol complex (~29%) along with 1-propanol (~16%) and the unreacted

propionaldehyde (~55% based on the starting material). *Thet*-BuNC ligand was also protonated by HOTf. After 25 hours at room temperature 42% of free 1-propanol and 46% of aldehyde were observed.

Ionic Hydrogenation of 3-pentanone using $\text{Cp}(\text{CO})_2(\text{ArNC})\text{WH}$ and HOTf. $\text{Cp}(\text{CO})_2(\text{ArNC})\text{WH}$ (17 mg, 39 μmol) and 3-pentanone (4 μL , 38 μmol) were placed in an NMR tube and CD_2Cl_2 (0.65 mL) was added. After adding HOTf (3.5 μL , 40 μmol), the tube was shaken for 5 min at room temperature. The ^1H NMR spectrum of the resulting red-brown solution showed the *cis*-alcohol complex (31%), free 3-pentanol (4%), and the unreacted 3-pentanone (65% based on the starting material). After 14 hours at room temperature 13% of the *cis*-alcohol complex, 54% of 3-pentanol, and 33% of 3-pentanone were observed.

^1H NMR of $[\text{Cp}(\text{CO})_2(\text{ArNC})\text{W}(\text{3-pentanol})]\text{OTf}$ in CD_2Cl_2 : δ 6.51 (d, $J = 8.0\text{Hz}$, 1H, OH), 6.00 (s, 5H, Cp), 3.16 (m, 1H, OCH), 1.47 (m, 4H, 2 CH_2), 0.89 (t, $J = 7.4\text{Hz}$, 3H, CH_3), 0.83 (t, $J = 7.4\text{Hz}$, 3H, CH_3).

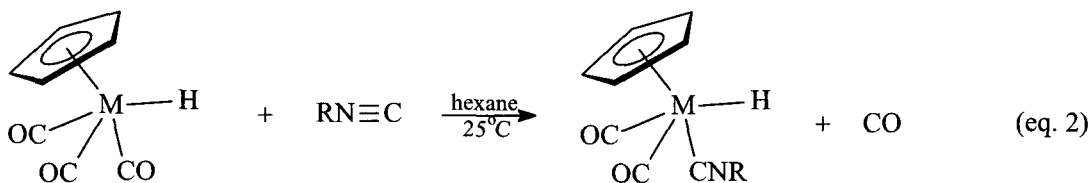
Catalytic Hydrogenation of 3-pentanone using $[\text{Cp}(\text{CO})_2(\text{ArNC})\text{Mo}][\text{BAr}^F_4]$. $\text{Cp}(\text{CO})_2(\text{ArNC})\text{MoH}$ (7.3 mg, 21 μmol) and $[\text{Ph}_3\text{C}][\text{BAr}^F_4]$ (19 mg, 21 μmol) was placed in a 5 mm NMR tube equipped with a J. Young valve. Then, 0.70 mL of the standard 300 mM solution of 3-pentanone in CD_2Cl_2 was added, giving a catalyst concentration of 30 mM. The ^1H NMR spectrum of the resulting red-brown solution showed the two Cp peaks at δ 5.91 and 5.74 indicating the formation of *cis/trans* ketone complexes. The tube was filled with H_2 (1.1atm) while frozen in liquid nitrogen, giving < 4.1atm H_2 when warmed to 23°C. The pressure was maintained by periodically refilling with H_2 as needed. The tube was wrapped with an aluminum foil to avoid any possible photochemical reaction and spun slowly using a mechanical stirring motor.

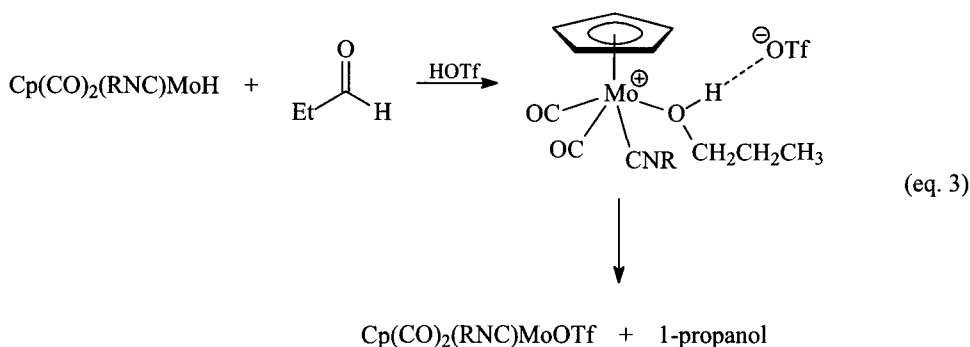
3. Results and Discussions

Synthesis and Characterization of $\text{Cp}(\text{CO})_2(\text{RNC})\text{MH}$ ($M = \text{Mo}, \text{W}$; $R = 2,6\text{-dimethylphenyl}, t\text{-butyl}$). The reaction of $\text{Cp}(\text{CO})_3\text{MH}$ ($M = \text{Mo}, \text{W}$) with RNC ($R = 2,6\text{-dimethylphenyl}, t\text{-butyl}$) at room temperature formed $\text{Cp}(\text{CO})_2(\text{RNC})\text{MH}$ in high yield (eq. 2).

The highly air-sensitive hydride complexes were identified by their characteristic IR bands, and two IR bands were observed in the IR spectrum between 1960-1890 cm^{-1} , higher in energy than values reported for the more electron-rich systems the more electron-rich systems $\text{Cp}(\text{CO})_2(\text{PCy}_3)\text{MoH}$ (1927 and 1850 cm^{-1}) and $\text{Cp}(\text{CO})_2(\text{PCy}_3)\text{WH}$ (1915 and 1821 cm^{-1}).¹ The $\text{N}\equiv\text{C}$ stretching band of the coordinated isocyanide ligand was observed between 2090-2050 cm^{-1} , indicating that the metal to ligand $d\pi\text{-}p\pi$ back bonding is considerably strong. In free isocyanides, ν_{NC} values are about 2130 cm^{-1} . It is noteworthy that the $d\pi\text{-}p\pi$ back bonding donation in the W-isocyanide complex (ν_{NC} 2059 cm^{-1} for $\text{Cp}(\text{CO})_2(t\text{-BuNC})\text{WH}$) is slightly stronger than that in the Mo-isocyanide complex (ν_{NC} 2071 cm^{-1} for $\text{Cp}(\text{CO})_2(t\text{-BuNC})\text{MoH}$). This observation is consistent to the fact that third row metals tend to form stronger bonds to ligands than second row metals.

Ionic Hydrogenation of propionaldehyde and 3-pentanone using $\text{Cp}(\text{CO})_2(\text{RNC})\text{MH}$ ($M = \text{Mo}, \text{W}$; $R = 2,6\text{-dimethylphenyl}, t\text{-butyl}$) in the presence of HOTf. Several examples of were known in which stoichiometric ionic hydrogenations of C=O bonds could be carried out by molybdenum metal hydrides upon reaction with acids.⁽¹¹⁾ Addition of triflic acid ($\text{CF}_3\text{SO}_3\text{H}$, abbreviated as HOTf) to a solution of propionaldehyde and $\text{Cp}(\text{CO})_2(\text{RNC})\text{MoH}$ ($R = 2,6\text{-dimethylphenyl}, t\text{-butyl}$) resulted in rapid hydrogenation of the aldehyde (eq. 3).





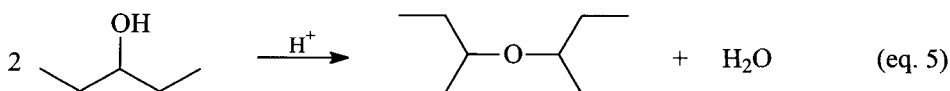
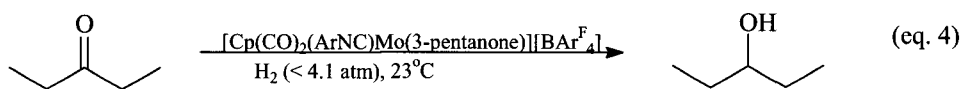
The formation of an alcohol complex was confirmed by ^1H NMR spectrum, and an alcoholic OH resonance at δ 6.51 for $[\text{Cp(CO)}_2(\text{ArNC})\text{Mo(3-pentanol)}]\text{OTf}$ suggested that the alcohol OH and an oxygen on the triflate counterion were attached via a hydrogen bond in solution.^[12]

Catalytic Hydrogenations of 3-pentanone using $[\text{Cp(CO)}_2(\text{ArNC})\text{Mo(3-pentanone)}][\text{BAR}^{\text{F}}_4]$ (Ar = 2,6-dimethylphenyl; $\text{Ar}^{\text{F}} = \text{C}_6\text{F}_5$). Bullock and Voges showed that the ketone complexes, $[\text{Cp(CO)}_2(\text{PR}_3)(\text{Et}_2\text{C}=\text{O})\text{M}][\text{BAR}^{\text{F}}_4]$ (M = Mo, W; R = phenyl, cyclohexyl, methyl), can hydrogenate ketones such as 3-pentanone at room temperature under <4 atm H_2 .^[8] Similarly, the catalyst formed *in situ* by the abstraction of hydride from by $\text{Cp(CO)}_2(\text{ArNC})\text{MoH}$ was studied under same reaction conditions used for by $[\text{Cp(CO)}_2(\text{PR}_3)(\text{Et}_2\text{C}=\text{O})\text{M}][\text{BAR}^{\text{F}}_4]$. When the ketone complex in CD_2Cl_2 was reacted under H_2 with 3-pentanone, hydrogenation of the C=O bond occurs, leading to the alcohol product, 3-pentanol (eq. 4).

The experiment was carried out in a 5 mm NMR tube sealed under 1 atm H_2 at liquid nitrogen temperature. When warmed to room temperature, this leads to an actual pressure in the tube of almost 4 atms. Under this

condition the concentration of H_2 dissolved in the CD_2Cl_2 is about 15 mM, as determined by integration of the resonance for dissolved H_2 at δ 4.60.^[13] The total amount H_2 in the tube is sufficient to hydrogenate all of the ketone, but most of it is in the gas phase above the solution. The reaction was monitored by ^1H NMR spectroscopy, indicating the appearance of the product alcohol as well as smaller amounts of its ether (Et_2CH) $_2\text{O}$ arising from condensation of two alcohols (eq. 5).

[Fig. 1] shows the time profile of the concentrations from the catalytic hydrogenation of 3-pentanone by $[\text{Cp(CO)}_2(\text{ArNC})\text{Mo(3-pentanone)}][\text{BAR}^{\text{F}}_4]$. Slow hydrogenation occurs, producing 3-pentanol, giving the fastest initial rate of about 2 turnovers per 1 day. After 5 days, the concentration of 3-pentanol was 219mM and that of the ether (Et_2CH) $_2\text{O}$ was 17 mM. As the catalytic reaction proceeds, the formation of $[\text{Cp(CO)}_2(\text{ArNC})\text{Mo(3-pentanol)}]^+$ is accompanied by other organometallic product, such as $\text{Cp(CO)}_2(\text{ArNC})\text{MoH}$. Most revealing is the formation of protonated isocyanide ligand which suggests that isocyanide ligand dissociates from the metal to reduce the catalytic activity.



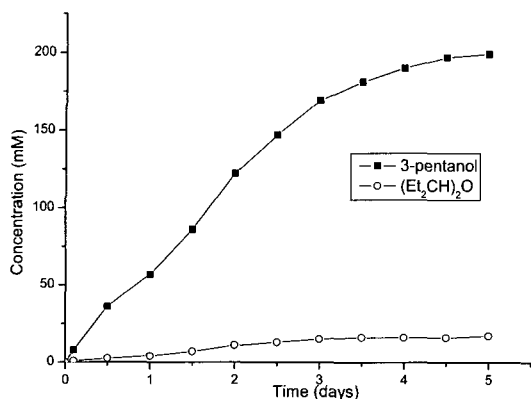


Fig 1. Time profile of the catalytic hydrogenation of 3-pentanone by $[\text{Cp}(\text{CO})_2(\text{ArNC})\text{Mo}(\text{3-pentanone})][\text{BAr}^F_4]$ under H_2 (<4.1 atm) in CD_2Cl_2 at 23°C .

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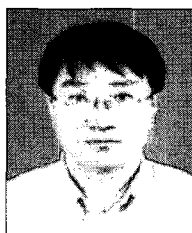
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[13] The amount of H_2 in solution determined by ^1H NMR integration is less than the actual amount because of the presence of 25% para- H_2 present in H_2 , so a correction factor has to be applied to account for this, multiplying the integrated amount by 1.33 to determine the actual concentration.

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