Supporting Information

for

Ruthenium-catalyzed deaminative redistribution of primary and secondary amines

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1 General Considerations

All manipulations were performed under an argon atmosphere using standard Schlenk techniques. All air-sensitive compounds and reactions were carried out under an inert atmosphere of argon using rotaflow stopcock. Glassware was stored in an oven or flame-dried prior to use. Routine solvents were purchased from Aldrich and were deoxygenated and dried using a Solvent Purification System. The reagents used for experiments were purchased from Sigma-Aldrich and fleshly distilled before use and stored under argon atmosphere. Toluene-d_8 was degassed and stored over activated 4-Å molecular sieves prior to use. ^{1}H NMR (300 MHz) and ^{13}C NMR (75 MHz) spectra were recorded on a Varian Mercury 300 MHz spectrometer using C_7D_8 as a solvent. NMR measurements were performed on Bruker Avance III DRX 600, operating at frequencies of 600.200 MHz (^{1}H) and 242.965 MHz (^{31}P). NMR spectra were recorded at 298 K. ^{1}H resonance was observed using signals of deuterated toluene (2.08 ppm) as internal standard. Chemical shifts are reported in ppm, relative to deuterated toluene at 2.08 ppm (^{1}H NMR). ^{31}P NMR spectra were referred to 85% H_3PO_4 at 0 ppm, the ^{31}P chemical shifts were uncorrected. Mass spectra of the products were determined by GC-MS analysis on a Varian Saturn 2100T, equipped with a BD-5 capillary column (30 m) and a Finigan Mat 800 ion trap detector.

2 Experimental section

2.1 Preparation of Products

2.1.1 A general procedure for redistribution of dibutylamine in the presence of ruthenium(0) and ruthenium(II) catalysis

Reagents and solvent were dried and deoxygenated, the syntheses were carried out in a closed system under argon atmosphere. The ruthenium catalyst $[Ru(H)(Cl)(CO)(PCy_3)_2]$ (**1**), $[Ru(H)(Cl)(CO)(PPh_3)_3]$ (**2**), $[Ru(Cl)_2(PPh_3)_3]$ (**3**), $[Ru_3(CO)_{12}]$ (**4**), $[RuH(CO)(MeCN)_2(PCy_3)_2]^+[BF_4]^-$ (**5**) and $[RuHCl(PPh_3)_3]$ (**6**) (0.0069 mmol of ruthenium complexes) and anthracene (0.069 mmol), placed into Schlenk vessel equipped with rotaflo stopcock and magnetic stirring bar, was dissolved in toluene (2 mL). Then dibutylamine (0.138 mmol) was added. The reaction mixture was stirred and heated at 120 °C for 24 h. The progress of the reaction was monitored by GC-MS.

2.1.2 A general procedure for redistribution of amines homo-coupling

Reagents and solvent were dried and deoxygenated, the syntheses were carried out in a closed system under argon atmosphere. The ruthenium catalyst [$RuHCl(CO)(PCy_3)_2$] (5 mg, 0.0069 mmol) and anthracene (0.069 mmol) or dodecane (0.069 mmol), placed into Schlenk vessel equipped with rotaflo stopcock and magnetic stirring bar, was dissolved in toluene (2 mL). Then amine (0.138 mmol) was added. The reaction mixture was stirred and heated at 120 ^oC for 24-72 h. The progress of the reaction was monitored by GC-MS. Products were isolated by fractional distillation.

2.1.3 A general procedure for redistribution of amine cross-coupling

Reagents and solvent were dried and deoxygenated, the syntheses were carried out in a closed system under argon atmosphere. The ruthenium catalyst [$RuHCl(CO)(PCy_3)_2$] (5 mg, 0.0069 mmol) was dissolved in toluene (2 mL) and introduced into a Schlenk vessel equipped with rotaflo stopcock and magnetic stirring bar. Then amine 1 (0.138 mmol), amine 2 (0.138 mmol) and anthracene (0.069 mmol) were added. The reaction mixture was stirred and heated at 120 ^oC and maintained at that temperature for 24 h. The progress of the reaction was monitored by GCMS.

2.1.4 A general procedure for stoichiometric and catalytic reactions monitored by ^1H NMR

0.01 g (0.013 mmol) of [$RuHCl(CO)(PCy_3)_2$] and 0.6 mL of toluene-d_8 were placed under argon in an Young NMR tube. Next the benzylamine (0.0015 g, 0.014 mmol for stoichiometric and 0.0295 g, 0.275 mmol for catalytic reaction) or diethylamine (0.020 g, 0.275 mmol for catalytic process) was added and the reaction was heated at 120 ^oC. As an internal standard was used mesitylene (s, 1.7 ppm). The progress of the reaction was monitored by NMR.

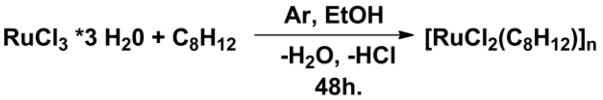
2.1.5 A general procedure for stoichiometric diethylamine and [$RuCl_2(PPh_3)_3$]

0.02 g (0.02 mmol) of [$RuCl_2(PPh_3)_3$] and 0.6 mL of toluene-d_8 were placed under argon in an Young NMR tube. Next the diethylamine (0.0015 g, 0.02 mmol) was added and the reaction was heated at 120 ^oC. As an internal standard was used mesitylene (s, 1.7 ppm). The progress of the reaction was monitored by NMR.

2.1.6 Preparation of $[RuHCl(CO)(PCy_3)_2]$

2.1.6.1 Preparation of polymer [dichloro(4-1,5-cyclooctadiene)ruthenium(II)] $[\{RuCl_2(4\text{-}C_8H_{12})\}]_n$

In a 100 mL Schlenk vessel equipped with a reflux condenser and an oil intake cap and magnetic stirrer, 2.4 g (9.2 mmol) of RuCl3x3H2O, 50 mL of dried and deoxygenated ethyl alcohol were charged. 2.49 g (23.0 mmol) of freshly distilled 1,5-cyclooctadiene were added dropwise. The whole was heated to a boiling point maintained for 48 hours under constant argon flow. The brown precipitate formed by cooling the solution was centrifuged and decanted (Scheme 1). The resulting compound was washed successively with ethanol (4x15-20 mL) and water (3x10 mL) and diethyl ether (2x15 mL), then dried under reduced pressure. As a result of synthesis, 2.45 g of $[RuCl_2(C_8H_{12})_n]$ polymer were obtained in a yield of 95% on the ruthenium atom. The complex was characterized by ^1H NMR analysis.



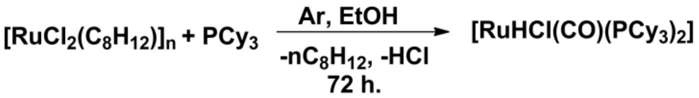
$$RuCl_3 \,{*}3\, H_20 + C_8H_{12} \xrightarrow[\substack{-H_2O,\ -HCl\\ 48h.}]{Ar,\ EtOH} [RuCl_2(C_8H_{12})]_n$$

Scheme 1 Preparation of polymer $[RuCl_2(C_8H_{12})_n]$

Polymer [dichloro(4-1,5-cyclooctadiene)ruthenium(II)] $[\{RuCl_2(4\text{-}C_8H_{12})\}]_n$ (Analysis one mer of polymer). ^1H NMR (600 Mz, C_6D_6) δ (ppm): 1.24 (m, 4H, $-CH_2-$); 1.94 (m, 4H, $-CH_2-$); 4.32 (m, 4H, $=CH-$).

2.1.6.2 Preparation of [chlorohydridocarbonylbis(tricyclohexylphosphine)ruthenium(II)] $[RuHCl(CO)(PCy_3)_2]$

In a evaporated 100 mL Schlenk vessel equipped with a magnetic stirrer, 1.0 g (3.57 mmol) of polymer $[RuCl_2(C_8H_{12})_n]$, 2.05 g (7.32 mmol) of tricyclohexylphosphine and 40 mL of dried and deoxygenated ethanol (all operations were carried out under argon!) sealing the system. The reaction was carried out at the boiling point of ethanol for 72 hours with intense stirring. During the reaction the color of the mixture changed from brown to orange (Scheme 2). The system was then cooled to room temperature, whereby the product appeared as a pale yellow fine crystalline solid. Excess of ethanol along with cyclooctadiene was decanted. The complex was washed three times with ethanol (15 mL) and then dried in vaccum. As a result of the synthesis, 2.54 g of the formulation was obtained in 85% yield, calculated as a metal atom. The complex was characterized by ^1H, ^{13}C, ^{31}P NMR analysis.



$$[RuCl_2(C_8H_{12})]_n + PCy_3 \xrightarrow[-nC_8H_{12},\ -HCl\ \ 72\ h.]{Ar,\ EtOH} [RuHCl(CO)(PCy_3)_2]$$

Scheme 2 Preparation of [RuHCl(CO)(PCy_3)$_2$]

[Chlorohydridocarbonylbis(tricyclohexylphosphine)ruthenium(II)]. H NMR (600 Mz, C_6D_6) δ (ppm): -24.16 (t, Ru-H); 1.26-1.54; 1.72-2.02 (m, $P(C_6H_{11})_3$); 2.32-2.57 (m, $P(C_6H_{11})_3$); **^{13}C NMR (600 Mz, C_6D_6)** δ (ppm): 27.20 (*p*-$P(C_6H_{11})_3$); 28.35 (*o*-$P(C_6H_{11})_3$); 30.45 (*m*-$P(C_6H_{11})_3$); 34.96 (t, *J* = 9.6Hz, $P(C_6H_{11})_3$); 202.10 (t, J_{PC} = 13.6Hz, CO); **^{31}P NMR (600 Mz, C_6D_6)** δ (ppm): 44.85 (s, 2PCy_3).

2.1.7 Preparation of [RuH(CO)(MeCN)$_2$(PCy_3)$_2$]$^+$[BF_4]$^-$

In a evaporated 25 mL Schlenk vessel equipped with a magnetic stirrer, 0.1 g (0.14 mmol) [RuHCl(CO)(PCy_3)$_2$] and 16 mg $NaBF_4$ were dissolved in 5mL of degassed acetonitrile. The reaction mixture was heated at 80 °C and was sttired for 8 h. The solution was filtrated through a celite after cooling to room temperature and washed by acetonitrile (15 mL). The combinated filtrate was evaporated under vaccum to give 0.103 g of product (89% yield). The complex was characterized by ^{1}H, ^{13}C, ^{31}P NMR analysis.

$$[RuHCl(CO)(PCy_3)_2] + NaBF_4 \xrightarrow[-\ NaCl]{MeCN} [RuHCl(CO)(MeCN)_2(PCy_3)_2]^+[BF_4]^-$$

The results are consistent with literature data[1].

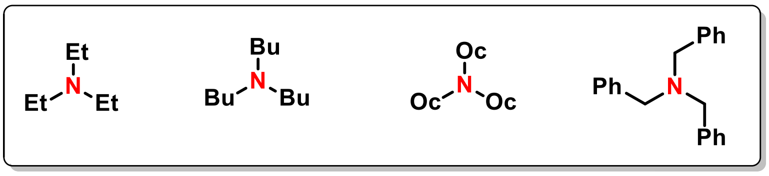
[Tetrafluoroborate diacetonitrilehydridocarbonylbis(tricyclophosphine)ruthenium(II)]. ^{1}H NMR (600 Mz, CD_2Cl_2) δ (ppm): -14.16 (t, Ru-H); 2.35 (s, 6H, CH_3); 1.10-2.10 (m, $P(C_6H_{11})_3$); **^{13}C NMR (600 Mz, CD_2Cl_2)** δ (ppm): 4.0 (CH_3); 27.20 (*p*-$P(C_6H_{11})_3$); 28.35 (*o*-$P(C_6H_{11})_3$); 30.45 (*m*-$P(C_6H_{11})_3$); 35.90 (t, *J* = 10.5Hz, $P(C_6H_{11})_3$); 125.4 (CN); 204,10 (t, J_{PC} = 13.4Hz, CO); **^{31}P NMR (600 Mz, CD_2Cl_2)** δ (ppm): 43.65 (s, PCy_3).

2.1.8 Preparation of [RuHCl(PPh_3)$_3$]

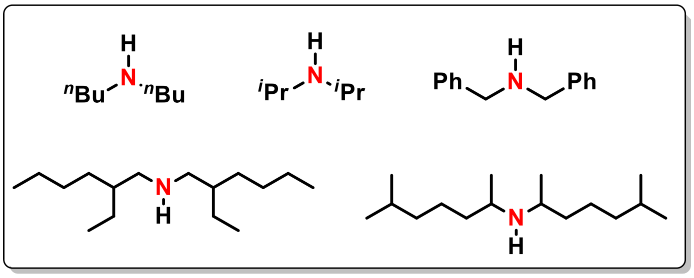
[RuHCl(PPh_3)$_3$] was prepared according to a literature method [2].

2.2 Obtained compound in reaction with one substrate

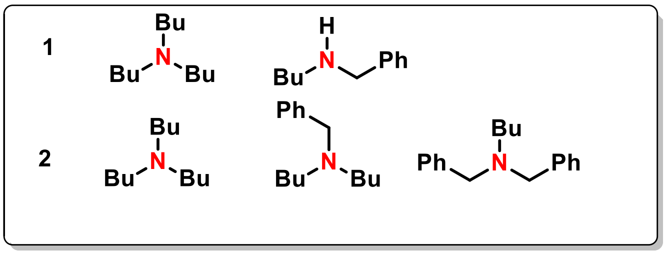
2.2.1 Products of redistribution of secondary amines



2.2.2 Products of redistribution of primary amines

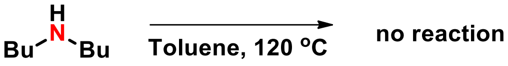


2.2.3 Products of redistribution of two different amines



3 Test reactions

3.1 Reaction of amine without catalyst



Scheme 3 Reaction of dibutylamine without ruthenium catalyst

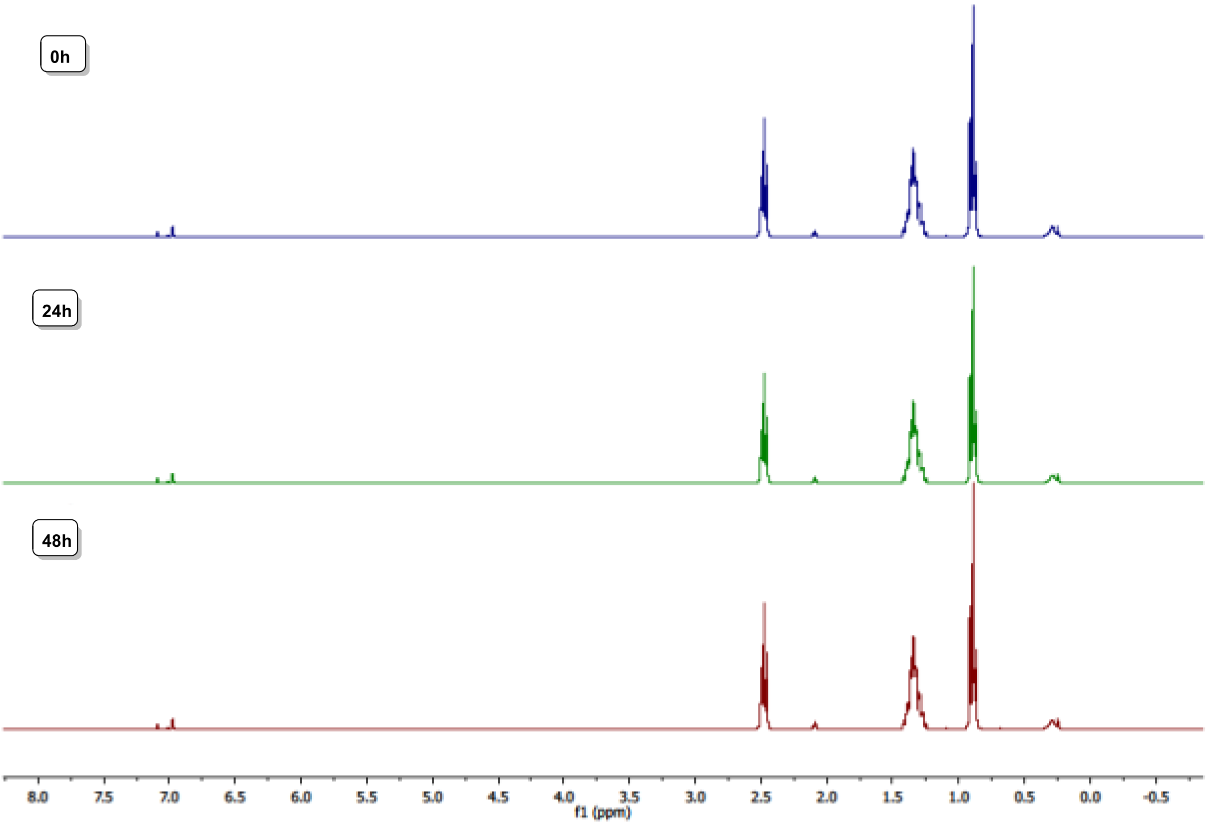
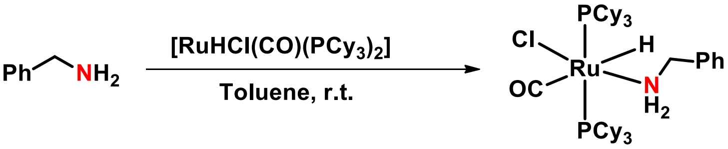


Fig. 1 Spectra ^{1}H NMR of reaction of dibutylamine without presence of ruthenium catalyst

Dibutylamine without catalyst was heated for 24 h at 120 °C (Scheme 3). Comparing ^{1}H NMR spectra of mixture before and after heating does not show tributylamine formation (Fig. 1). The experiment confirms necessity of ruthenium catalyst presence for redistribution to occur.

3.2 The coordination of benzylamine to complex [RuHCl(CO)(PCy3)2]



Scheme 4 The coordination of benzylamine to complex ruthenium(II)

3.2.1 ^1H NMR spectra of catalytic reaction after addition of amine to complex [RuHCl(CO)(PCy3)2]

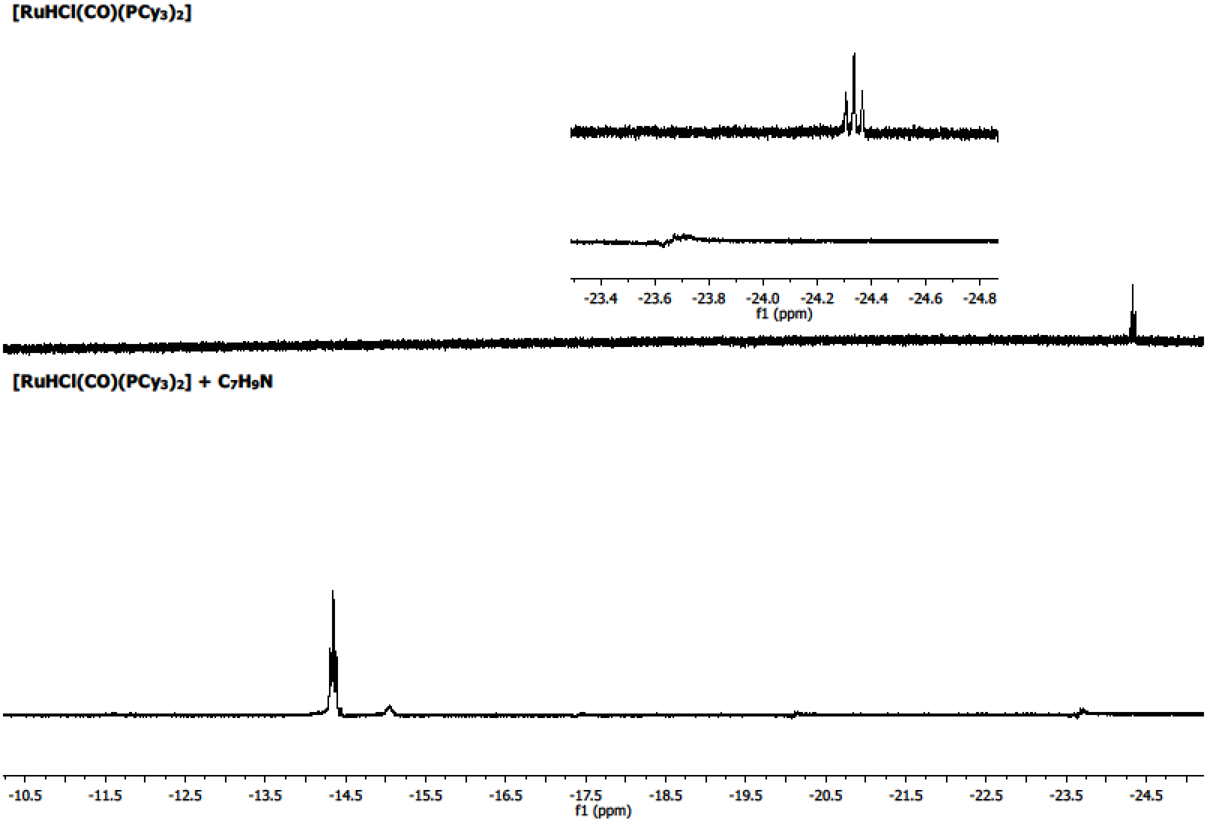


Fig. 2 ^1H NMR spectra of catalytic reaction after addition of benzylamine to complex ruthenium(II)

Formation of new ruthenium-hydride peak at -14.34 ppm (t, J_{PH} = 23.21 Hz) after addition of amine to complex [RuHCl(CO)(PCy3)2] at room temperature has been observed (Scheme 4, Fig. 2). The peak was assigned to ruthenium-hydride complex containing benzylamine as a ligand.

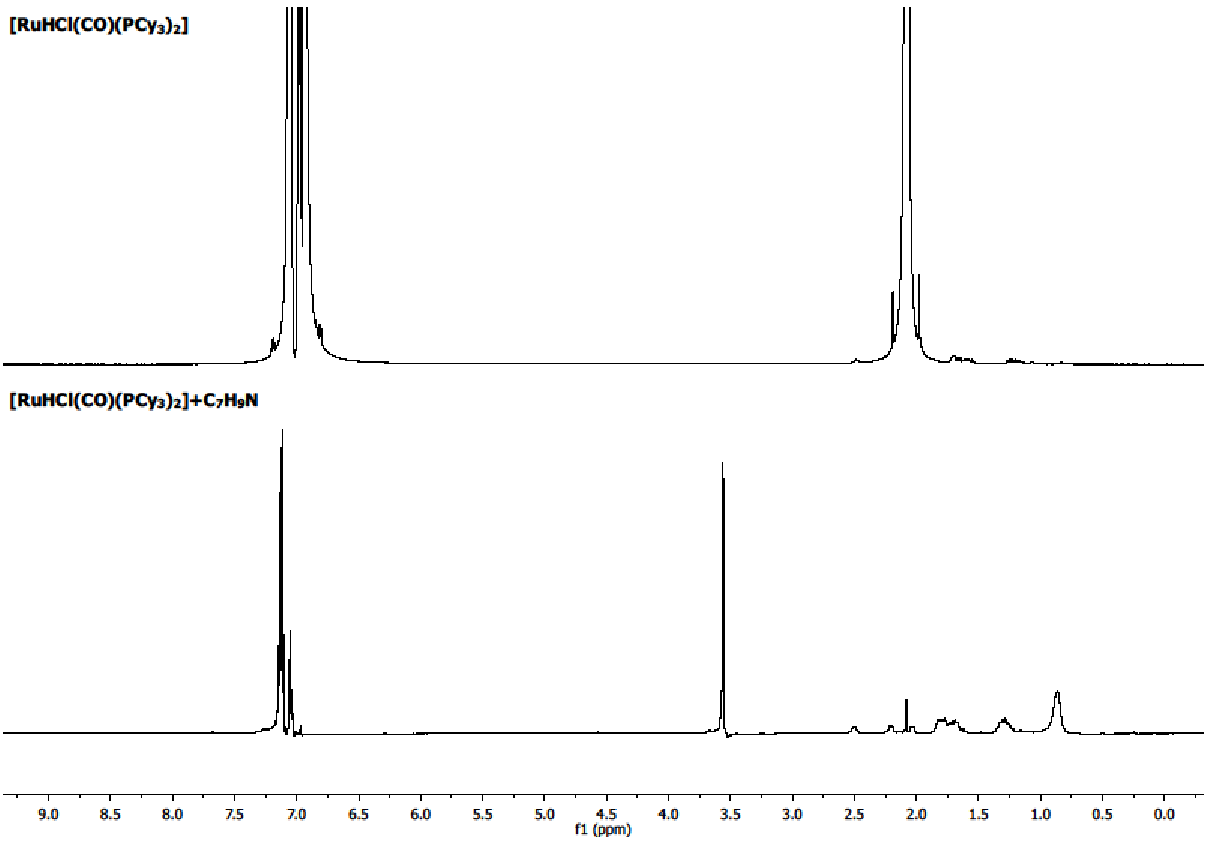


Fig. 3 ^1H NMR spectra of catalytic reaction after addition of benzylamine to ruthenium(II) complex

3.3 Reaction of benzylamine with ruthenium catalyst

3.3.1 ^{1}H NMR spectra of stoichiometric reaction between benzylamine and ruthenium catalyst

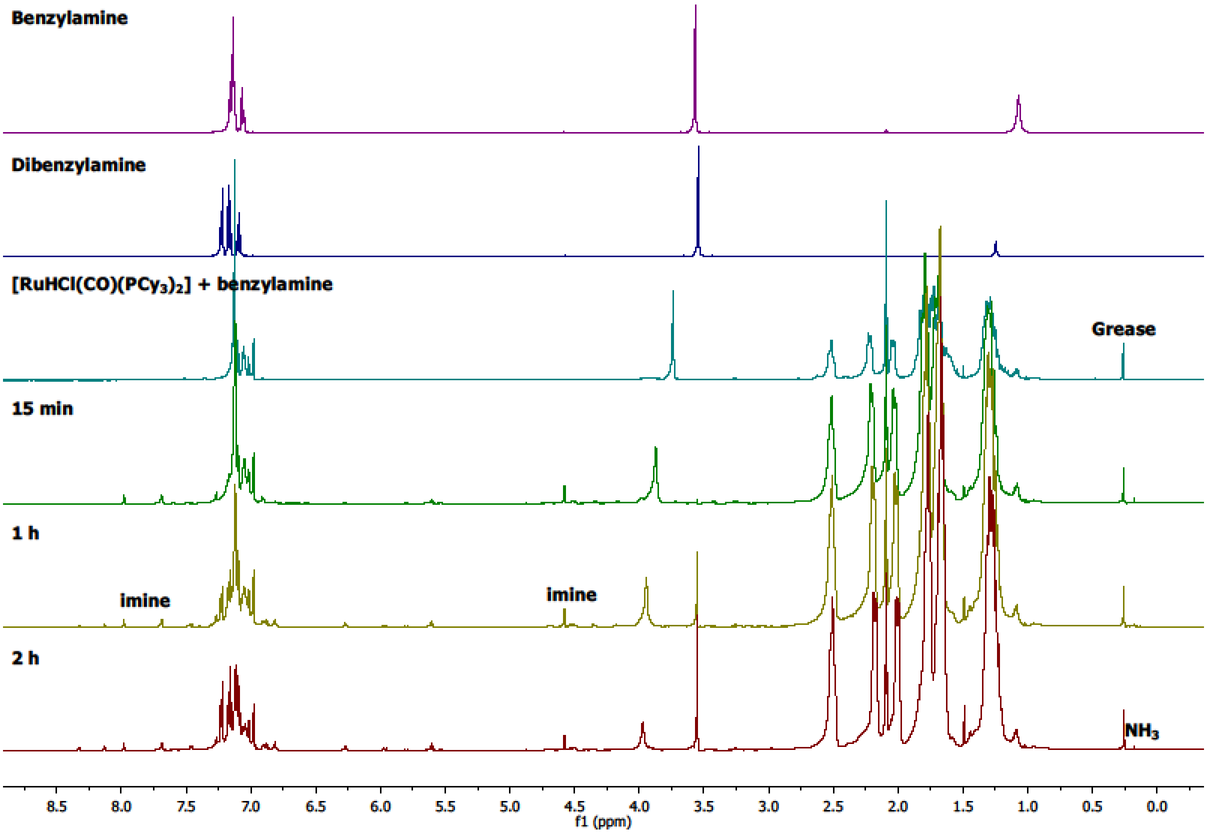


Fig. 4 ^{1}*H NMR spectra of stoichiometric reaction between benzylamine and* [RuHCl(CO)(PCy$_3$)$_2$]

Stoichiometric reaction between benzylamine and [RuHCl(CO)(PCy$_3$)$_2$] shows redistribution from benzylamine to dibenzylamine confirmed by ^{1}HNMR spectra (Fig. 4). The product is not bonded to ruthenium catalyst. In the reaction mixture formation of complexes containing imine intermediates were observed according to *Chem.Eur.J.* **2016**, *22*, 17758-17766.

3.3.2 2D NMR spectrum of stoichiometric reaction between benzylamine and ruthenium catalyst

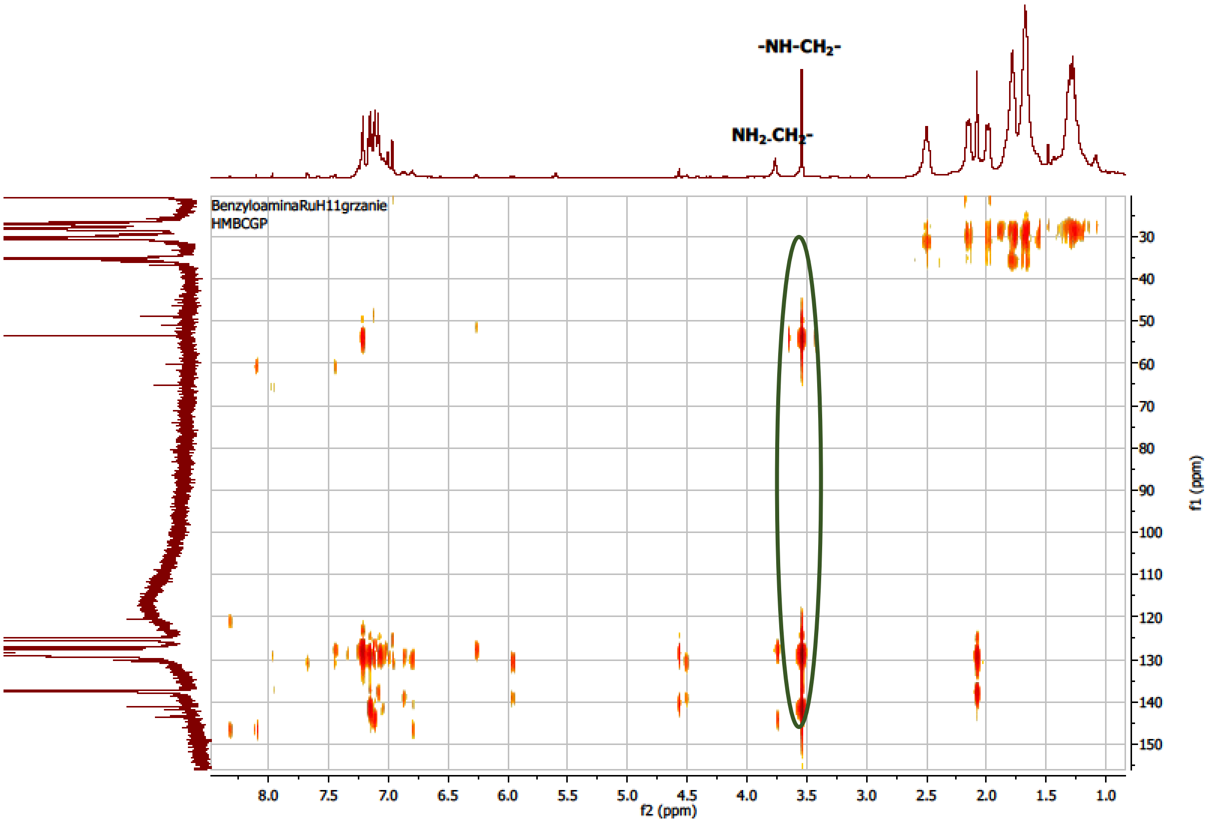


Fig. 5 2D NMR spectrum of stoichiometric reaction between benzylamine and $[RuHCl(CO)(PCy_3)_2]$

2D NMR analysis confirms the presence of product not bonded to ruthenium catalyst (Fig. 5). Signals from catalyst can be observed in the range of the 1 ppm to 2.5 ppm, while the signals from dibenzylamine are observed at 3.5 ppm.

3.3.3 ^{13}C NMR spectra of stoichiometric reaction between benzylamine and ruthenium catalyst

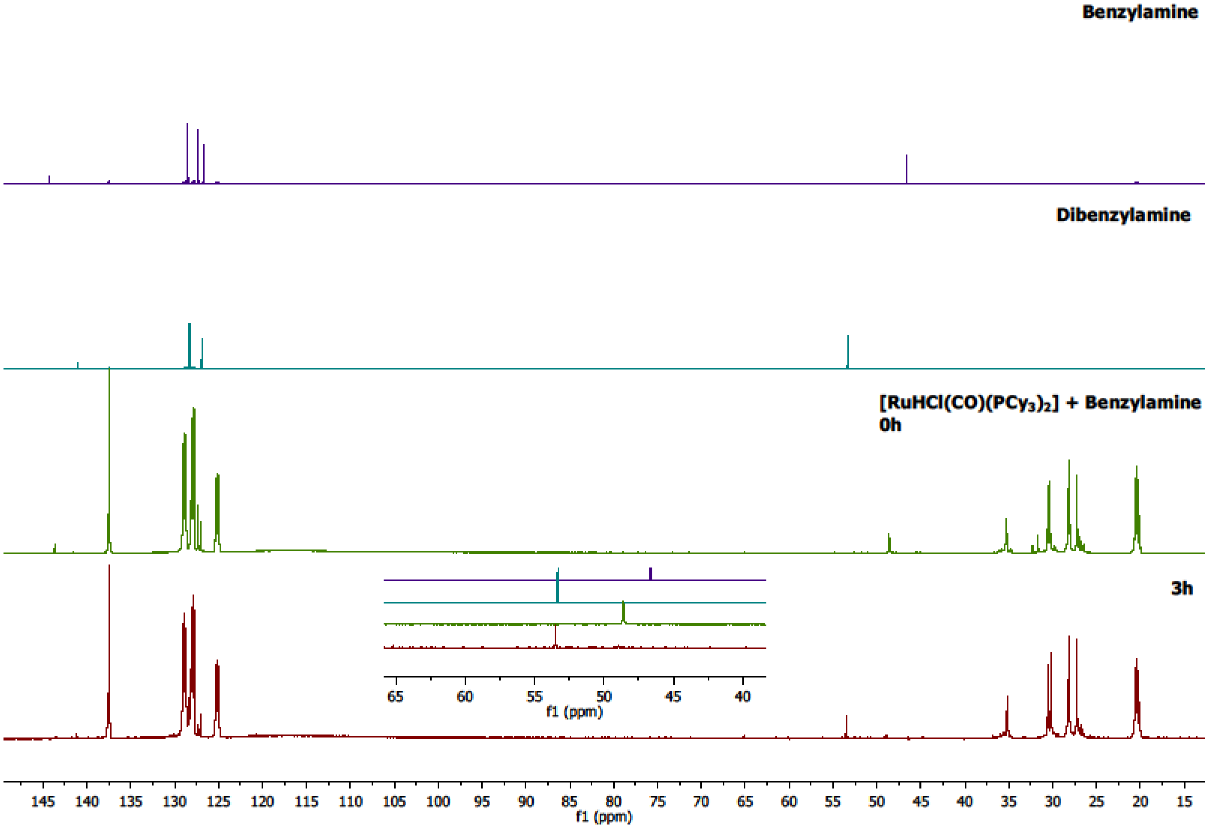


Fig. 6 ^{13}C NMR spectra of benzylamine and [RuHCl(CO)(PCy$_3$)$_2$] in molar ratio 1:1

^{13}C NMR spectra (Fig. 6) show decrease of benzylamine (s, CH_2-NH_2, 70.3 ppm) and formation of dibenzylamine (s, CH_2-NH-, 80.7 ppm). Reaction was carried out for 3 h at 120 °C.

3.3.4 ^{1}H NMR spectra confirming the presence of ammonia

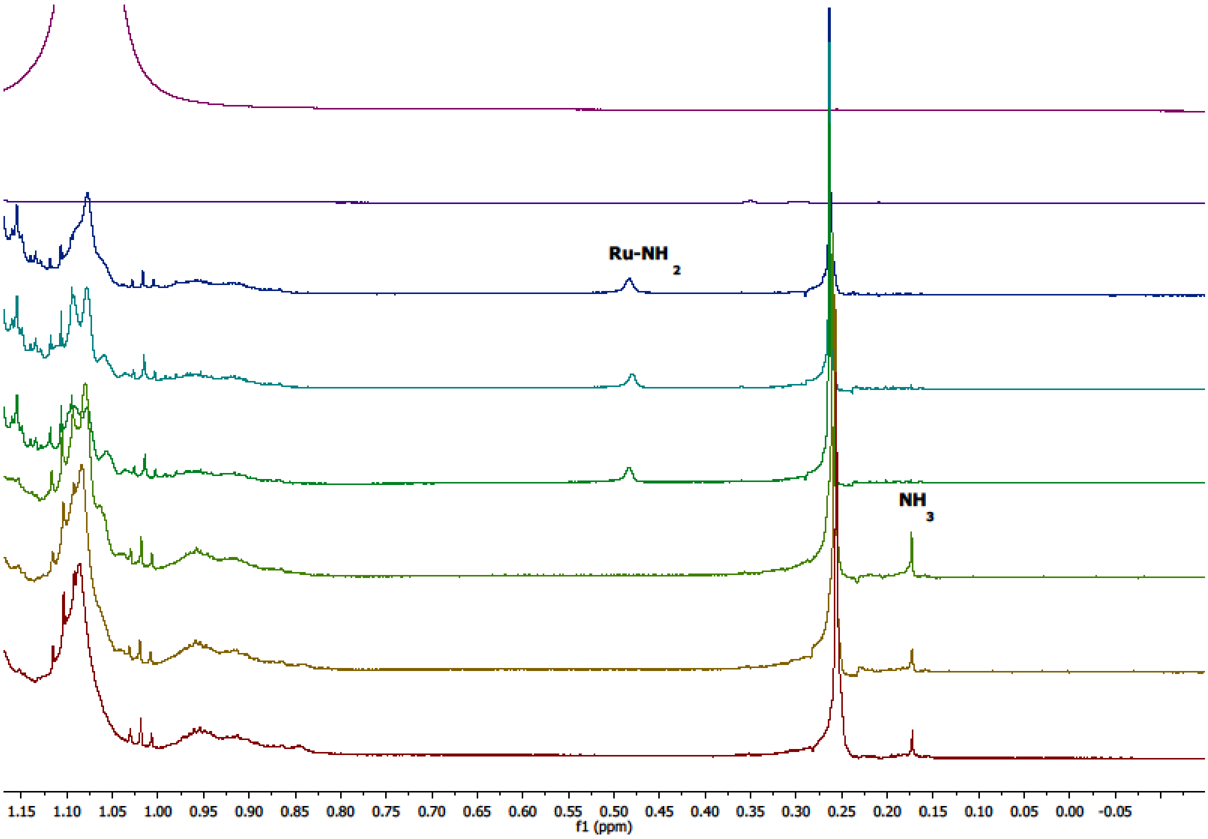


Fig. 7 *^{1}H NMR spectra of benzylamine and [RuHCl(CO)(PCy$_3$)$_2$] in molar ratio 1:1– confirming presence of ammonia*

The signal at 0.17 ppm belongs to the free ammonia. In the initial sample we observed signal at 0.48 ppm characteristic for bond Ru-NH (Fig. 7). While the reaction mixture was heated, the formation of new signal at 0.17 ppm was observed. (Aldridge *J. Am. Chem. Soc.*, **2016**, *138*, 4555−4564).

3.3.5 Catalytic reaction of benzylamine in the presence of ruthenium catalyst [RuHCl(CO)(PCy$_3$)$_2$]

The catalytic reaction of benzylamine in the presence of 5 mol% ruthenium(II) catalyst led to dibenzylamine. Progress of the reaction was monitored by ^{1}H NMR analyses (Fig. 8). The signal assigned to the initial ruthenium complex disappears and new signal appears at -14.36 ppm, J_{PH} = 22.83 Hz. This signal is a triplet, indicating the presence of two phosphine ligands in the complex. After two hours of heating at 120°C, the triplet shifted towards -14.23 ppm, J_{PH} = 22.63 Hz. After 20 hours, another form of the ruthenium - hydride complex (-13.91

ppm, J_{PH} = 22.07 Hz) was observed. This experiment demonstrated the presence of three different hydride-ruthenium complexes during the reaction.

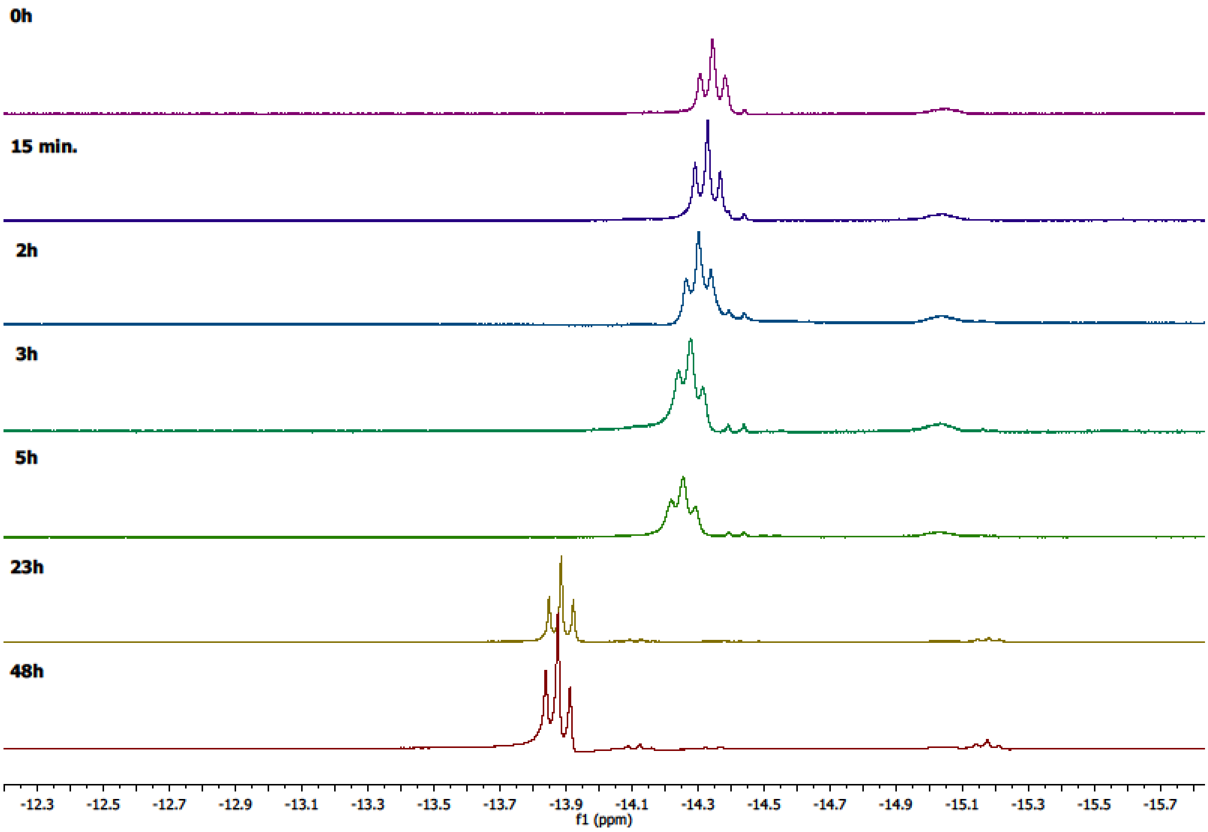
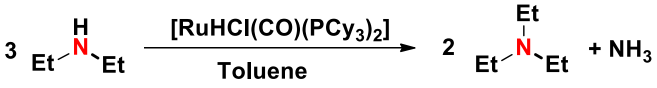


Fig . 8 Catalytic reaction of benzylamine and [RuHCl(CO)(PCy$_3$)$_2$] in molar ratio 1:0.05

3.4 Catalytic reaction of diethylamine in the presence of ruthenium catalyst [RuHCl(CO)(PCy$_3$)$_2$]



Scheme 5 Catalytic reaction of diethylamine and [RuHCl(CO)(PCy$_3$)$_2$] in molar ratio 1:0.05

3.4.1 ^{1}H NMR spectra of the catalytic reaction of diethylamine

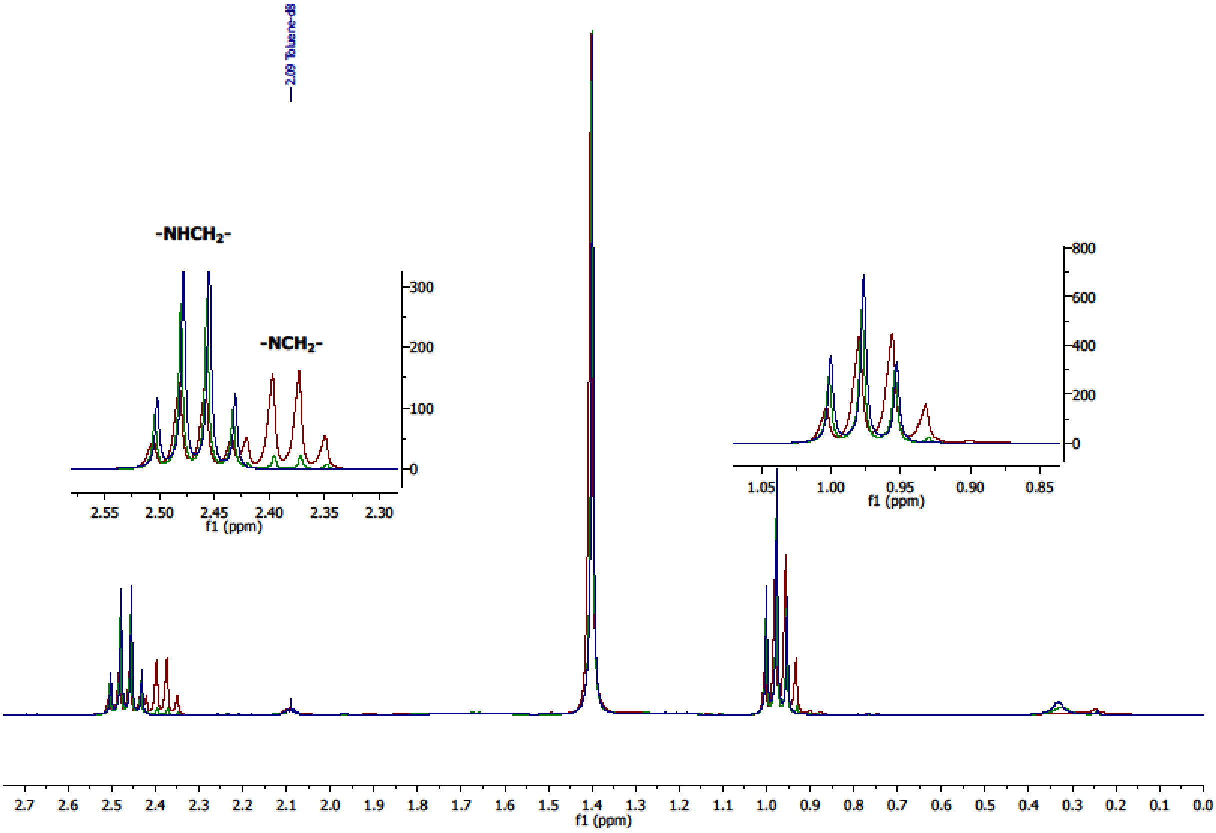


Fig. 9 ^{1}H NMR spectra of the catalytic redistribution of diethylamine

^{1}H NMR spectrum describes reaction of diethylamine in the presence of 5%mol ruthenium complex (Scheme 5, Fig. 9). This analysis clearly shows transformation of diethylamine to triethylamine (red color).

3.4.2 ^{13}C NMR spectra of the catalytic reaction of diethylamine

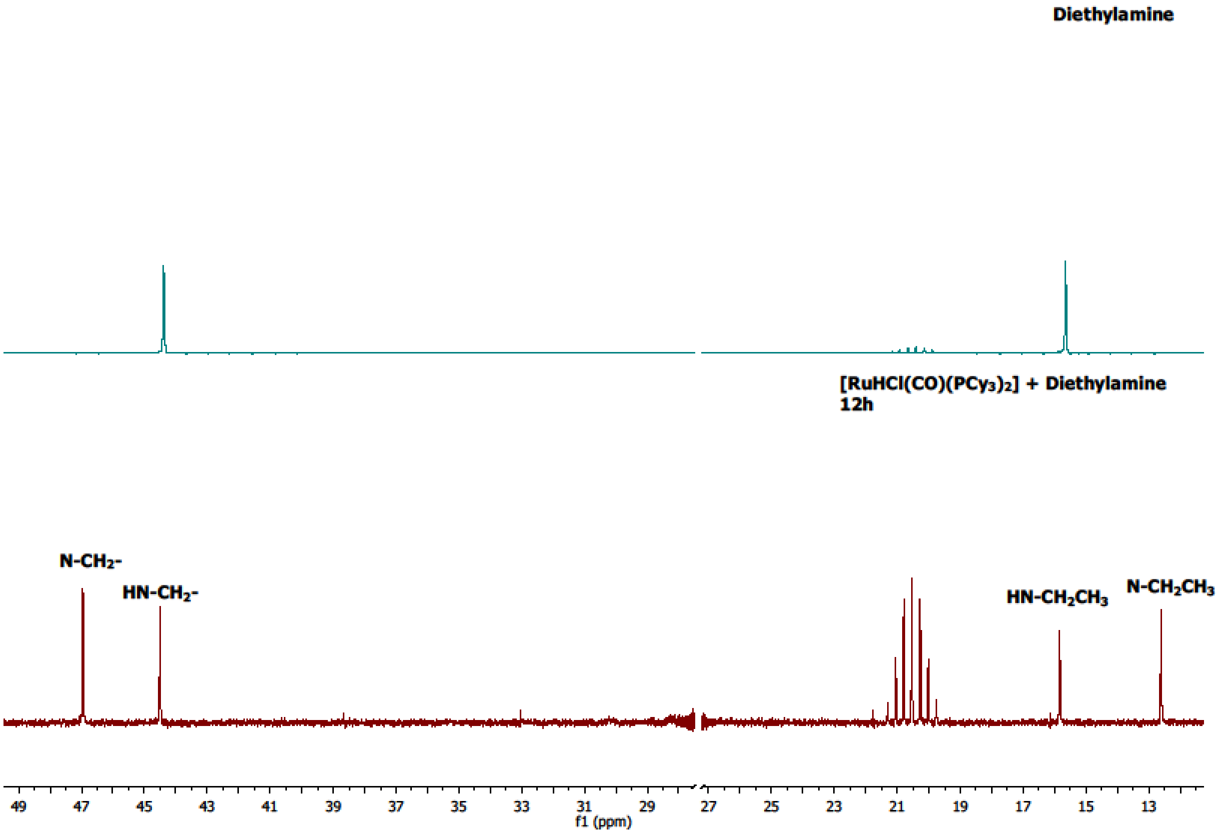
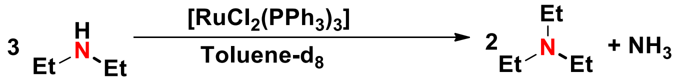


Fig. 10 ^{13}C NMR spectra of the catalytic redistribution of diethylamine

^{13}C NMR spectra show decrease of diethylamine and formation of triethylamine (Fig. 10). In the reaction mixture was not observed side products. Reaction was carried out for 3h at 120 °C.

3.5 The stoichiometric reaction between diethylamine and $RuCl_2(PPh_3)_3$



Scheme 6 The stoichiometric reaction between diethylamine and $RuCl_2(PPh_3)_3$

3.5.1 ^{1}H NMR spectra of stoichiometric reaction between diethylamine and $RuCl_2(PPh_3)_3$

Figure 11a presents $RuCl_2(PPh_3)_3$ dissolved in deuterated toluene and Figure 11b shows spectra after addition of amine to the system (Scheme 6) after 24 hours at room temperature. After heating new signal coming from ruthenium hydride complex was formed. As shown in spectra “c” and “d”, the signal at -17.6 ppm (J_{PH} = 25.7 Hz) becomes a distinct quartet, suggesting the presence of three phosphines in the system.

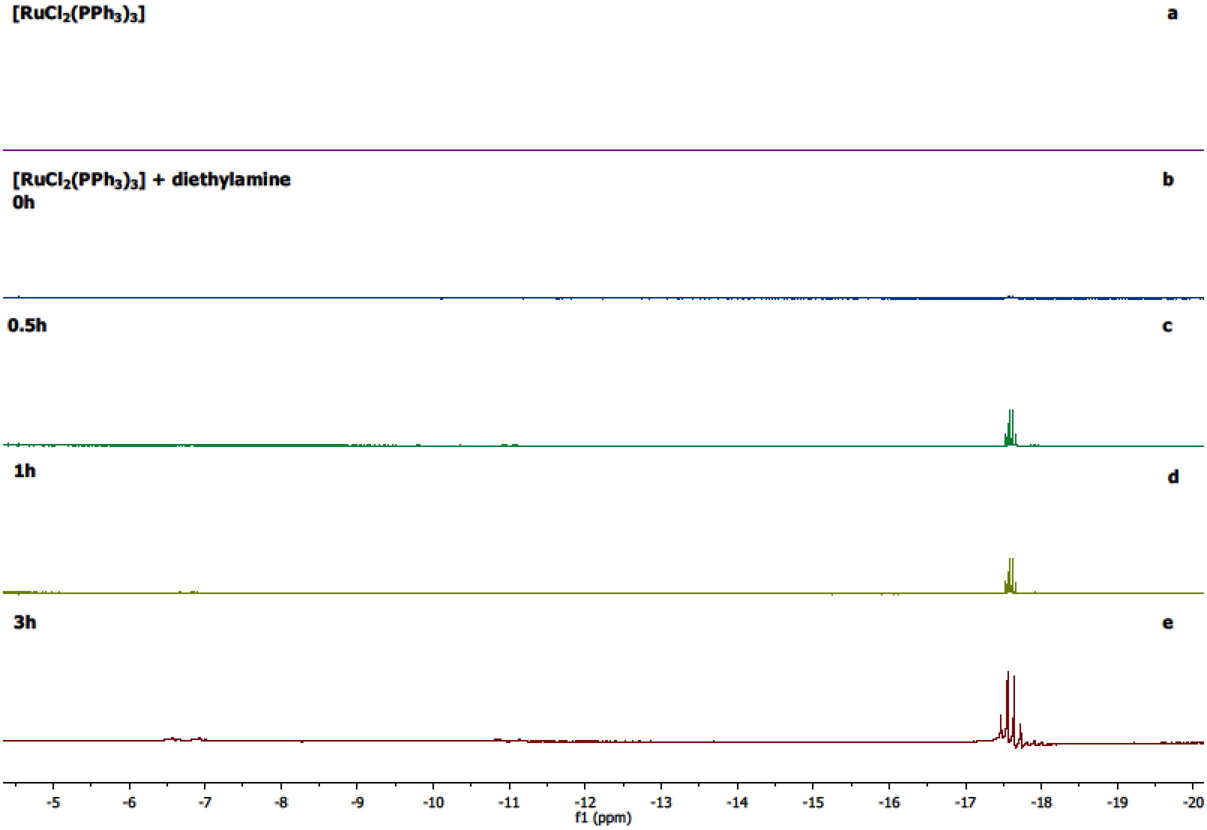


Fig. 11 ^{1}H NMR spectra of stoichiometric reaction between diethylamine and $RuCl_2(PPh_3)_3$

Chemical shift (0 ppm - 10 ppm) of ^{1}H NMR analysis confirms the gradual formation of triethylamine (Fig. 12). After 0.5 hours of reaction, the signals characteristic for the tertiary amine molecule were observed (2.43 ppm).

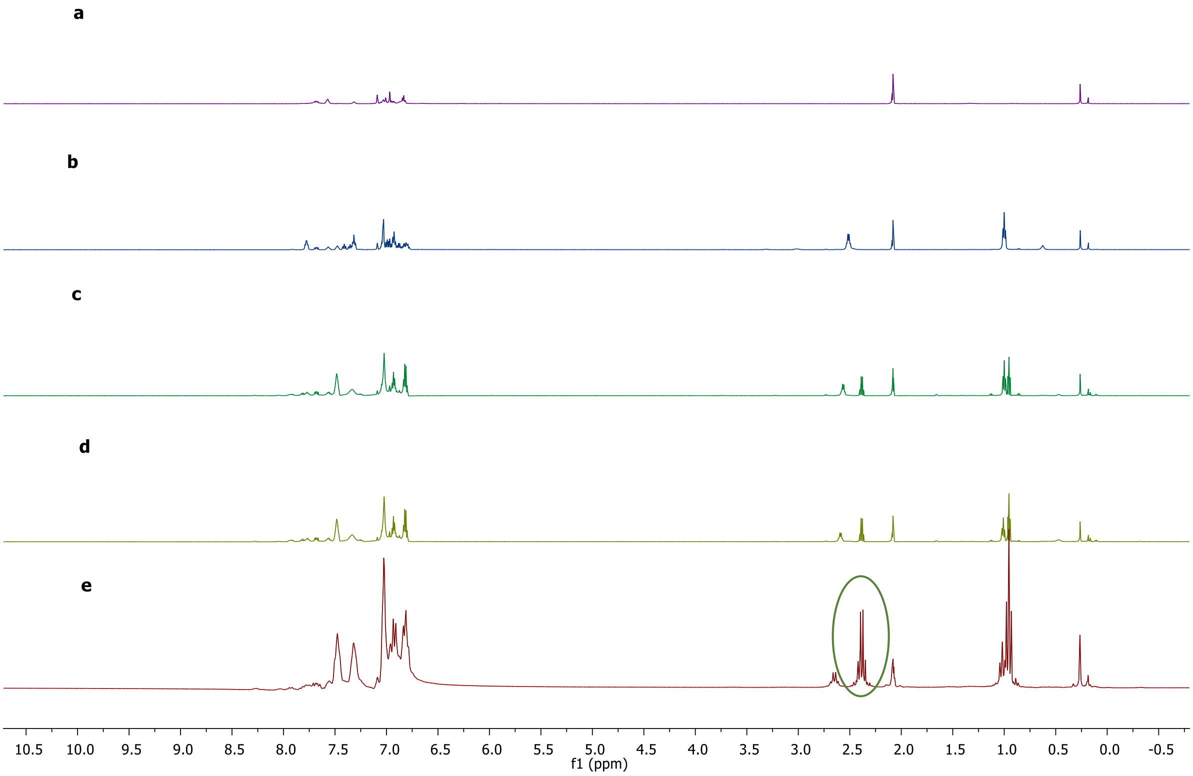
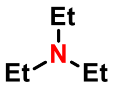
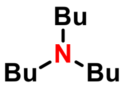
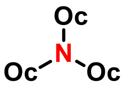
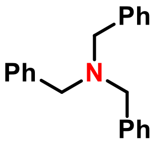


Fig. 12 ^{1}H NMR spectra of diethylamine and $RuCl_2(PPh_3)_3$ in molar ratio 1:1

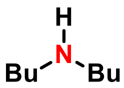
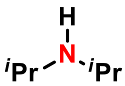
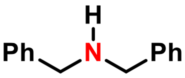
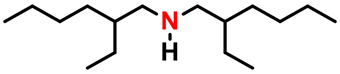
4 Characterization of the reaction products

4.1 Tertiary amines

	
N,N,N-triethylamine. ^{1}H NMR (300 Mz, $CDCl_3$) δ (ppm): 0.97 (9H,-CH_3), 2.43 (6H, -CH_2-). ^{13}C NMR (75 Mz, $CDCl_3$) δ (ppm): 11.48 (-CH_3), 46.46 (-CH_2-). **MS (EI): m/z (%)** = 101$^{\bullet+}$ (19), 86 (100), 58 (27), 44 (11), 30 (27), 29 (10.3);	*N,N,N*-tributylamine. ^{1}H NMR (300 Mz, $CDCl_3$) δ (ppm): 0.89 (9H, -CH_3), 1.35 (12H, -CH_2-), 2.42 (6H, -NCH_2-); ^{13}C NMR (75 Mz, $CDCl_3$) δ (ppm): 14.15 (9H, -CH_3), 20.89 (6H, -CH_2-), 29,51 (6H, -CH_2-), 54.12 (6H, -$N\underline{C}H_2$-). **MS (EI): m/z (%)** = 185$^{\bullet+}$ (6), 142 (100), 100 (30), 57 (10), 44 (12), 41 (13), 30 (10), 29 (18);
	
N,N,N-trioctylamine. ^{1}H NMR (300 Mz,	*N,N,N*-tribenzylamine. ^{1}H NMR (300 Mz,

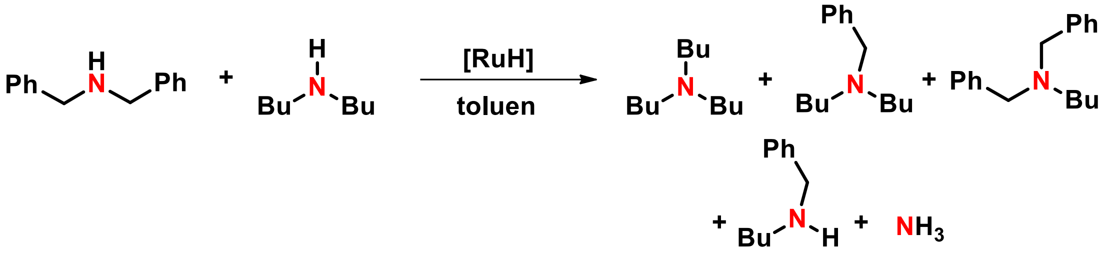
CDCl$_3$) δ (ppm): 0.88 (9H, -CH$_3$), 1.27 (36H, -CH$_2$CH$_3$), 1.45 (6H, -NCH$_2\underline{C}$H$_2$), 2.37 (6H, -NCH$_2$). **^{13}C NMR (75 Mz, CDCl$_3$)** δ (ppm): 14.11 (-CH$_3$), 22.75 (-CH$_2$-), 27.25 (-CH$_2$-), 27.77 (-CH$_2$-), 29.44 (-CH$_2$-), 29.71 (-CH$_2$-), 31.98 (-CH$_2$-), 54.41 (-NCH$_2$-). **MS (EI): m/z (%) =** 353$^{\bullet+}$ (3), 255 (19), 254 (100), 142 (11), 44 (10), 43 (11);	**CDCl$_3$)** δ (ppm): 3.65(6H, -NCH$_2$-), 7.33-7.28 (3H, C$_6$H$_5$), 7.42-7.38 (6H, C$_6$H$_5$), 7.59-7.42 (6H, C$_6$H$_5$); **^{13}C NMR (75 Mz, CDCl$_3$)** δ (ppm): 58.1 (-NCH$_2$-), 127.0 (C$_6$H$_5$), 128.4 (C$_6$H$_5$), 128.9 (C$_6$H$_5$), 139.8 (C$_6$H$_5$). **MS (EI): m/z (%) =** 287$^{\bullet+}$ (16), 210 (20), 196 (15), 92 (14), 91 (100), 65 (12);

4.2 Secondary amines

	
N,N-di(*n*-butyl)amine. **^{1}H NMR (300 Mz, CDCl$_3$)** δ (ppm): 0.92 (6H, -CH$_3$), 1.21-1.55 (8H, -CH$_2$), 2.56 (4H, CH$_2$N-). **^{13}C NMR (75 Mz, CDCl$_3$)** δ (ppm): 14.03 (-CH$_3$), 20.64 (-CH$_2$), 32.51 (-$\underline{C}$H$_2$-CH$_2$NH-), 49.85 (-NCH$_2$-). **MS (EI): m/z (%) =** 129$^{\bullet+}$ (), 86 (100), 57 (18), 44 (72), 41 (18), 30 (62), 29 (21);	*N,N*-di(*i*-propyl)amine. **^{1}H NMR (300 Mz, CDCl$_3$)** δ (ppm): 1.04 (12H,-CH$_3$), 2.91 (2H, -NCH-). **^{13}C NMR (75 Mz, CDCl$_3$)** δ (ppm): 23.49 (-CH$_3$), 45.32 (-NCH-). **MS (EI): m/z (%) =** 101$^{\bullet+}$ (4), 86 (42), 44 (100);
	
N,N-dibenzylamine. **^{1}H NMR (300 Mz, CDCl$_3$)** δ (ppm): 1.61 (-NH), 3.78 (4H, -NCH$_2$), 7.29 (10H, C$_6$H$_5$). **^{13}C NMR (75 Mz, CDCl$_3$)** δ (ppm): 53.11, (-NCH$_2$), 126.85 (*p*-C$_6$H$_5$), 128.08 (*m*-C$_6$H$_5$), 128.31 (*o*-C$_6$H$_5$), 140.33 (C$_6$H$_5$). **MS (EI): m/z (%) =** 197$^{\bullet+}$ (6), 106 (53), 92 (25), 91 (100), 65 (15);	*N,N*-bis(2-ethylhexyl)amine. **^{1}H NMR (300 Mz, CDCl$_3$)** δ (ppm): 0.87 (6H, -CH$_3$), 0.9 (6H, -CH$_3$), 1.27 (8H, -CH$_2$), 1.29-1.37 (8H, -CH$_2$), 1.43 (2H, -CH), 2.47 (4H, -NCH$_2$). **^{13}C NMR (75 Mz, CDCl$_3$)** δ (ppm): 10.95 (-CH$_3$), 14.16 (-CH$_3$), 23.24 (-CH$_2$), 29.13 (-CH$_2$), 31.56 (-CH$_2$), 39.47 (CH), 53.68 (-NCH$_2$). **MS (EI): m/z (%) =** 143 (11), 142 (100), 71 (15), 57 (24), 44 (98), 43 (25), 41 (18), 30 (37), 29 (12);

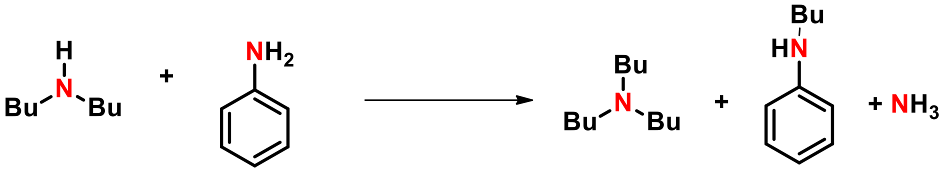
4.3 Products of the reaction between two different amines

4.3.1 Reaction between dibutylamine and dibenzylamine



N,N,N-tributylamine. MS (EI): m/z (%) = $185^{\bullet +}$ (6), 142 (100), 100 (30), 57 (10), 44 (12), 41 (13), 30 (10), 29 (18); *N*-benzyl-*N,N*-dibutylamine. MS (EI): m/z (%) = 176 (48), 91 (100); *N*-dibenzylobutyloamina. MS (EI): m/z (%) = 210 (37), 91 (100), 65 (13); *N*-benzyl-*N*-butylamine. MS (EI): m/z (%) = 120 (36), 91 (100), 65 (12);

4.3.2 Reaction between dibutylamine and aniline



N,N,N-tributylamine. MS (EI): m/z (%) = $185^{\bullet +}$ (6), 142 (100), 100 (30), 57 (10), 44 (12), 41 (13), 30 (10), 29 (18); *N*-butylaniline. MS (EI): m/z (%) = $149^{\bullet +}$ (17), 106 (100), 79 (10), 77 (14);

5 References:

[1] C. S. Yi, Z. He, I. A. Guzei, *Organometallics* 2001, *20* (17), pp 3641 – 3643

[2] C. J. Yue, Y. Liu, R. He, *Journal of Molecular Catalysis A: Chemical* 2006, *259*, 17 – 23