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# THE INTERACTION BETWEEN [Pt($\mu$-Cl)(Safrole-1H)]$_2$ AND 1,3-DIISOPROPYLBENZIMIDAZOLIUM BROMIDE IN THE PRESENCE OF Ag$_2$O

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**Abstract.** The interaction of the dimeric complex [Pt($\mu$-Cl)(Saf)]$_2$ (**1**) (Saf: deprotonated safrole) with the salt $^i$Pr$_2$-bimy·HBr in the presence of Ag$_2$O in acetone solvent was studied. The obtained products were mixed complexes of [PtCl(Saf)($^i$Pr$_2$-bimy)] (**2**) and [PtBr(Saf)($^i$Pr$_2$-bimy)] (**3**) with different molar ratio (**P$_{I}$, P$_{II}$, P$_{III}$**) depending on the used molar ratio of **1**:salt:Ag$_2$O. In the case of this ratio being 1 : 2 : 1.1, **P$_I$** was obtained, which is a mixture of **2** and **3** at a 3 : 1 ratio. When concentration of the salt was increased, the product was **P$_{II}$** with the molar ratio of **2** : **3** at 1 : 3. However, when both the salt and Ag$_2$O were increased at the same time, **2** and **3** in **P$_{III}$** had equal molar proportions. The two main factors affecting the **2** : **3** molar ratio are the different solubility of AgCl and AgBr, and the different coordinating ability of Cl$^-$ and Br$^-$ ligands. The structures of **2** and **3** were determined by $^1$H NMR spectra and single crystal X-ray diffraction. The results reveal that Saf in **2** and **3** coordinates with Pt(II) *via* C=C$_{\text{olefinic}}$ and C5 of benzene ring. Meanwhile, $^i$Pr$_2$-bimy coordinates with Pt(II) through C$_{\text{NCN}}$ and is in *trans*-position compared to the C=C$_{\text{olefinic}}$ bond.

*Keywords:* Pt(II) complex, safrole, 1,3-diisopropylbenzimidazol-2-ylidene, $\eta^2$-olefin.

*Classification numbers:* 2.6.1, 2.10.2, 2.10.3.

## 1. INTRODUCTION

Studies on transition metal complexes containing *N*-heterocyclic carbene (NHC) have been attracting scientists over the past two decades because of their large applications in many fields such as medicine, material chemistry and especially in catalysis for organic synthesis [1 - 4]. For example, Pt(II) complexes bearing NHC derived from imidazole and benzimidazole have shown the high catalytic potential for atomic optimization reactions such as hydroamination and hydrosilylation [3].

Karstedt's catalyst, a $\eta^2$-olefin platinum(0) complex, is known as one of the two common catalysts for the hydrosilylation reaction in the industry [5]. Despite the good activity, the catalyst has low selectivity and it is unstable. Thus, NHC-Pt(0)-olefin complexes have been developed and demonstrated higher catalytic activities [5 - 8]. However, their preparation is difficult due to the need of dry, air-free conditions in dry solvent with strong base as NaH/$^t$BuOK and a long reaction time. In contrast, NHC-Pt(II)-olefin complexes are stable, easy to be synthesized but they have been surprisingly rare [9 - 11].

Among synthetic methods of Pt(II)-NHC complexes, the procedure starting from simple Pt(II) compounds and azolium salts in the presence~~t~~ of common bases such as $Ag_2O$ is the most used [4, 9]. Recently, our study on the reaction of [Pt($\mu$-Cl)($^i$PrEug)]$_2$ ($^i$PrEug: deprotonated isopropyleugenoxyacetate) with 1,3-diisopropylbenzimidazolium bromide ($^i$Pr$_2$-bimy·HBr) with the molar ratio of 1 : 2 using different bases including $Ag_2O$, $Na_2CO_3$, $CH_3COONa$, $NaHCO_3$, NaOH afforded the mixture of [PtCl($^i$PrEug)($^i$Pr$_2$-bimy)] and [PtBr($^i$PrEug)($^i$Pr$_2$-bimy)] [10]. In continuation of this research direction, we herein describe in detail the reaction of [Pt($\mu$-Cl)(Saf)]$_2$ (**1**, Saf: deprotonated safrole**)** and $^i$Pr$_2$-bimy·HBr in the presence of $Ag_2O$ to investigate factors controlling the reaction direction, which could orient for synthesis of complexes of type [PtX(arylolefin)(NHC)].

## 2. MATERIALS AND METHOD

### 2.1. Equipment

Thin layer chromatography (TLC) was performed on commercial glass plates coated with 0.25 mm silica gel (Merck, Kieselgel 60 F254) and visualized under UV-light at 254 nm. The $^1$H NMR spectra were recorded on Bruker AVANCE 500 MHz with TMS as internal standard in acetone-$d_6$ at the Vietnam Academy of Science and Technology. Single crystal X-ray diffraction was carried out on a Bruker AXS SMART APEX diffractometer using graphite-monochromatic MoKα radiation (λ = 0.71073 Å) at Department of Chemistry, National University of Singapore.

### 2.2. The interaction between complex 1 and $^i$Pr$_2$-bimy·HBr

Complex [Pt($\mu$-Cl)(Saf)]$_2$ (**1**) [12] and $^i$Pr$_2$-bimy·HBr [10] were synthesized as previously reported.

### *2.2.1. The interaction without base*

A mixture of **1** (80 mg, 0.1 mmol) and $^i$Pr$_2$-bimy·HBr (57 mg, 0.2 mmol) in 10 mL acetone was stirred at ambient temperature (AT). The starting suspension converted quickly to a transparent clear yellow solution after stirring for 5 mins. After 4 hours, the solvent was removed under vacuum to obtain yellowish solids. The solids were washed with warm water (3 × 3 mL) and the precursor complex (**1**) was obtained.

### *2.2.2. The interaction using base*

A mixture of **1** (80 mg, 0.1 mmol), $^i$Pr$_2$-bimy·HBr and $Ag_2O$ with different amounts was suspended in 10 mL acetone and stirred in the dark at AT for different intervals (N$^0$. 2-12, Table 1). At the end of each reaction, the reaction mixture was filtered through Celite and the residue was washed thoroughly by acetone until the colorless filtrate. The solvent of the filtration was removed in vacuo to give solids, which were then washed with warm water (3 × 3 mL) and cold ethanol (2 × 3 mL). Drying under vacuum at 50 $^o$C for 5 hours gave the product as a yellow powder.

Yellow crystals suitable for single crystal X-ray diffraction (XRD) were obtained after slow evaporation in $CHCl_3$/ethanol solution of the product of the experiment N°. 5 (Table 1) at ambient conditions within 10 h. The resulting analyses of TLC, $^1$H NMR spectra and XRD indicated that all products in the experiment N°. 2-12 were a mixture of two complexes [PtCl(Saf)($^i$Pr$_2$-bimy)] (**2**) and [PtBr(Saf)($^i$Pr$_2$-bimy)] (**3**) with different molar ratios.

## 3. RESULTS AND DISSCUSION

The interaction between [Pt($\mu$-Cl)(Saf)]$_2$ (**1**) and $^i$Pr$_2$-bimy·HBr was conducted in acetone solvent at ambient temperature with different time and molar ratio of **1** : salt : $Ag_2O$. The results listed in Table 1 indicate that in the absence of base (N°. 1), the reaction did not give the expected complex containing $^i$Pr$_2$-bimy but the precursor **1** despite the unambiguous reaction phenomenon.

*Table 1*. Experiments for the reaction of **1** with $^i$Pr$_2$-bimy·HBr (* determined by $^1$H NMR spectrum).

| N°. | Molar ratio of **1** : salt : $Ag_2O$ | Time (hours) | Characteristic of product | Product | Molar ratio of **2** : **3** | Yield |
|---|---|---|---|---|---|---|
| 1 | 1 : 2 : 0 | 4 | Yellowish powder | **1** | - | - |
| 2 | 1 : 2 : 1.1 | 4 | Yellow powder | $\mathbf{P_{I}}$ | 3 : 1* | 45 % |
| 3 | 1 : 2 : 1.1 | 5 | Yellow powder | $\mathbf{P_{I}}$ | 3 : 1 | 60 % |
| 4 | 1 : 2 : 1.1 | 6 | Yellow powder | $\mathbf{P_{I}}$ | 3 : 1 | 75 % |
| 5 | 1 : 2 : 1.1 | 7 | Yellow powder | $\mathbf{P_{I}}$ | 3 : 1 | 87 % |
| 6 | 1 : 2 : 1.1 | 9 | Yellow powder | $\mathbf{P_{I}}$ | 3 : 1 | 87 % |
| 7 | 1 : 4 : 1.1 | 7 | Yellow powder | $\mathbf{P_{II}}$ | 1 : 3* | 90 % |
| 8 | 1 : 8 : 1.1 | 7 | Yellow powder | $\mathbf{P_{II}}$ | 1 : 3 | 91 % |
| 9 | 1 : 10 : 1.1 | 7 | Yellow powder | $\mathbf{P_{II}}$ | 1 : 3 | 91 % |
| 10 | 1 : 4 : 2.2 | 7 | Yellow powder | $\mathbf{P_{III}}$ | 1 : 1* | 90 % |
| 11 | 1 : 8 : 4.4 | 7 | Yellow powder | $\mathbf{P_{III}}$ | 1 : 1 | 92 % |
| 12 | 1 : 10 : 5.5 | 7 | Yellow powder | $\mathbf{P_{III}}$ | 1 : 1 | 92 % |

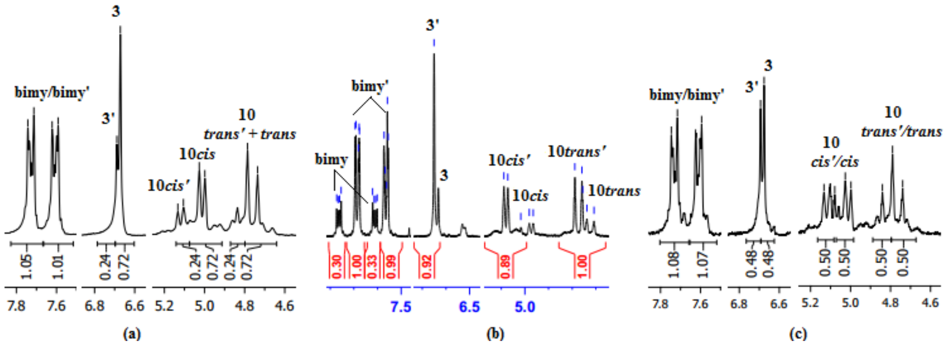
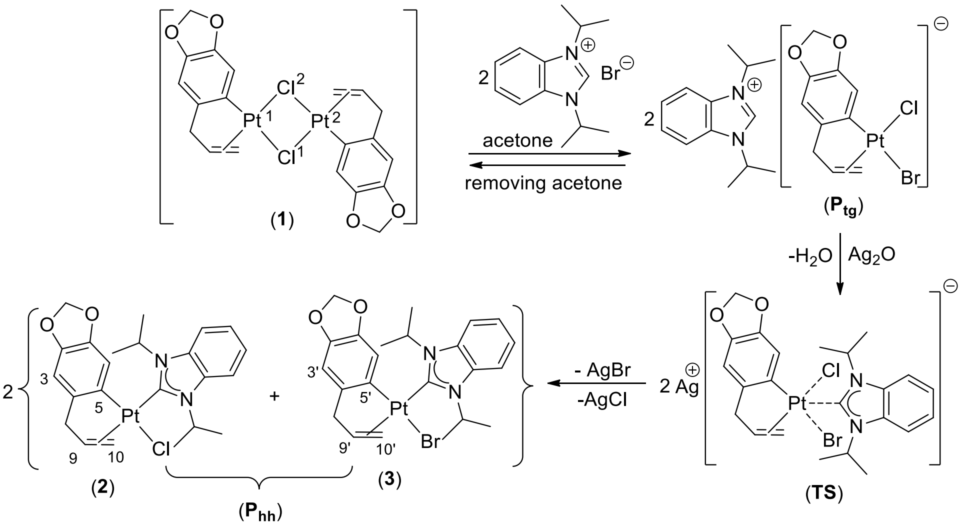


*Figure 1*. Assigned partial $^1$H NMR spectra of $\mathbf{P_{I}}$ (a), $\mathbf{P_{II}}$ (b) and $\mathbf{P_{III}}$ (c).

The similar result has been observed in the case of using [Pt($\mu$-Cl)($^i$PrEug)]$_2$ instead of complex **1** [10]. These findings affirm that complexes of general formula [PtX(arylolefin)(NHC)] could not be synthesized by the reaction between dimer complex [Pt($\mu$-Cl)(arylolefin)]$_2$ and NHC·HX without base. Therefore, in the experiments N$^o$. 2-12, $Ag_2O$ was added with different molar ratio of **1** : salt : $Ag_2O$. The TLC and $^1H$ NMR spectra results (Fig. 1) show that the obtained products in all these experiments are a mixture of two complexes [PtCl(Saf)($^i$Pr$_2$-bimy)] (**2**) and [PtBr(Saf)($^i$Pr$_2$-bimy)] (**3**) with different molar ratio depending on the reaction conditions. In which, the mixtures with the **2** : **3** ratio of 3:1, 1:3 and 1:1 are denoted **P$_I$**, **P$_{II}$** and **P$_{III}$**, respectively (Table 1). Thus, a possible pathway for formation of **2** and **3** is suggested in Scheme 1.

As shown in Scheme 1, firstly, the $Pt^1$-$Cl^1$ or $Pt^2$-$Cl^2$ bond weaker than $Pt^1$-$Cl^2$ or $Pt^2$-$Cl^1$ bond in complex **1** [10] are easily cleaved by $^i$Pr$_2$-bimy·HBr to form ionic complex ($^i$Pr$_2$-bimy·H)$^+$[Pt(Br)(Cl)(Saf)]$^-$ (**P$_{tg}$**), which is stable in the solution but converts to the starting complex **1** when the solvent is evaporated. Then the cation ($^i$Pr$_2$-bimy·H)$^+$ is deprotonated to give NHC, *i.e.* $^i$Pr$_2$-bimy, in the presence of $Ag_2O$ and the transition state (**TS**) as five-coordinate Pt(II) complex is formed. Finally, the elimination of $Ag^+$ and $X^-$ ($Cl^-$/$Br^-$) from **TS** by generating AgX precipitate to form four-coordinate Pt(II) complexes (**P$_{hh}$**) more stable. This step is influenced by 2 competitions: (i) the better solubility of AgCl than AgBr; (ii) the lower coordinating ability with $Pt^{2+}$ of $Cl^-$ than $Br^-$ as the soft cation $Pt^{2+}$ prefers to coordinate with the softer anion $Br^-$.



*Scheme 1*. Possible pathway for formation of **2** and **3** from **1** and $^i$Pr$_2$-bimy·HBr in the present of $Ag_2O$.

This pathway helps us to understand more about the experimental results in Table 1. Specifically, in the experiments N$^o$. 2-6, complex **1** and $^i$Pr$_2$-bimy·HBr were used as their reaction stoichiometry, $Ag_2O$ was used slightly excess due to the long reaction time. The reaction time prolongation leaded to the increase of the reaction yield of forming the product **P$_I$**. And the optimized reaction time was 7 h with the yield of 87 % (N$^o$. 5). The explanation for this result is that the less solubility of AgBr dominates over resulting in the prevalence of complex bearing $Cl^-$ (**2**). It is worthy to note that, when replacing complex **1** in the experiments N$^o$. 5 by its analogue, [Pt($\mu$-Cl)($^i$PrEug)]$_2$, it also gave the similar result [10]. In the experiments N$^o$. 7-9 the reactions were carried out under the same conditions as in the N$^o$. 5 except for the larger amount of ${}^{i}Pr_2$-bimy·HBr. Remarkably, the obtained product was $\mathbf{P_{II}}$, but the yield only increased slightly compared to that in the N$^{o}$. 5 and unchanged (≈ 91 %) when the molar ratio of salt: **1** increased from 4 (N$^{o}$. 7) *via* 8 (N$^{o}$. 8) to 10 (N$^{o}$. 9). In this case, the excess $Br^-$ in the solution makes it difficult to separate the $Br^-$ in the inner coordination sphere of **TM** according to the principle of equilibrium shift. As a consequence, the complex bearing $Br^-$ (**3**) bec~~a~~omes dominant. To clarify this, in the experiments N$^{o}$. 10-12, the amount of $Ag_2O$ and ${}^{i}Pr_2$-bimy·HBr were simultaneously increased to 2, 4, 5 times more than in the N$^{o}$. 5. This means that $Ag^+$, $Br^-$ and ${}^{i}Pr_2$-bimy are all excessive after the second step. Therefore, the precipitation of AgBr is easy but competitive with the formation of (${}^{i}Pr_2$-bimy)-Ag-Br complex [4]. Because of this, the resulting product in these three experiments is $\mathbf{P_{III}}$ with the similar yield (around 92 %).

*Table 2*. $^{1}H$ NMR signals of free safrole (SafH) and Saf in **1**–**3**, δ (ppm), *J* (Hz).

| Comp. | H8a | H8b | H9 | H10*cis* | H10*trans* | H3 | H5 | H6 | H7 |
|---|---|---|---|---|---|---|---|---|---|
| SafH[12] | 3.26 | | 5.90 | 5.02 | 5.04 | 6.65 | 6.60 | 6.70 | 5.85 |
| **1**[12] | 2.76 | 3.66 | 5.08 | 4.30 | 3.94 | 6.66 | - | 6.76 | 5.83/5.84 |
| **2** | 3.18 d $^{3}J_{PtH}$ 90 | 3.85 dd $^{2}J$ 16.5 $^{3}J$ 6.5 | 5.94 m | 5.02 d $^{3}J$ 8.5 $^{2}J_{PtH}$ 50 | 4.76 d $^{3}J$ 14.5 $^{2}J_{PtH}$ 60 | 6.67 s | - | 5.95 s $^{3}J_{PtH}$ 60 | 5.75 s |
| **3** | 3.16 d $^{3}J_{PtH}$ 90 | 3.79 dd $^{2}J$ 16.5 $^{3}J$ 6.5 | 5.92 m | 5.08 d $^{3}J$ 8.5 $^{2}J_{PtH}$ 50 | 4.77 d $^{3}J$ 14.5 $^{2}J_{PtH}$ 60 | 6.65 s | - | 5.92 s $^{3}J_{PtH}$ 60 | 5.70 s |

In order to determine the structures of complexes **2** and **3**, their $^{1}H$ NMR spectra are assigned and listed in Table 2 and Table 3. The distinctive sets of signals of **2** and **3** in the $^{1}H$ NMR spectra of $\mathbf{P_{hh}}$ are based on their intensity ratio in the spectra and the XRD result of $\mathbf{P_{I}}$.

*Table 3*. $^{1}H$ NMR signals of ${}^{i}Pr_2$-bimy·HBr and ${}^{i}Pr_2$-bimy in **2** and **3**, δ (ppm), *J* (Hz).

| | NHCN | $H_{bimy}$ | CH | $CH_3$ |
|---|---|---|---|---|
| ${}^{i}Pr_2$-bimy·HBr[13] | 11.37 s | 7.76 dd $^{3}J$ 6.5, $^{4}J$ 3.0<br>7.61 dd; $^{3}J$ 6.5, $^{4}J$ 3.0 | 5.18 m | 1.80 d; $^{3}J$ 7.0 |
| **2** | - | 7.73/7.61 dd $^{3}J$ 7.0, $^{4}J$ 1.5; 7.34-7.32 m | 6.16/5.75 m | 1.88/1.85/1.67/1.32 d $^{3}J$ 6.0 |
| **3** | - | 7.68/7.56 dd $^{3}J$ 7.0, $^{4}J$ 1.5; 7.31-7.29 m | 6.10/5.71 m | 1.82/1.80/1.63/1.25 d $^{3}J$ 6.0 |

Table 2 shows the proton signals characteristic for the coordination of Saf with Pt(II) in **2** and **3.** Specifically, i) two protons H8 in SafH give rise to one doublet signal, but in the spectra of **2** and **3** there are two separate signals for H8a and H8b; ii) The absence of H5 signal and the $^{195}Pt$ satellites from H10, H8 and H6 are clear with the distance between them, $^{2}J_{PtH}$ for H10, 50–60 Hz; $^{3}J_{PtH}$ for H8 and H6, 90 and 60 Hz, respectively; iii) The ethylenic resonances of **2** and **3** shift remarkably downfield compared to the parent complex **1** and resemble to those of SafH (Table 2). These findings show that in **2** and **3**, deprotonated safrole has coordinated with Pt(II) *via* the C5 of benzene ring and the $C{=}C_{ethylenic}$ of the allyl group, which is in *trans* position with respect to the strong donor ligand [9, 10], in this case being ${}^{i}Pr_2$-bimy. The evidences for the coordination of ${}^{i}Pr_2$-bimy with Pt(II) through the carbon atom of NCN group in the $^{1}H$ NMR spectra of **2** and **3** are: i) the absence of the NCHN proton signal in precursor ${}^{i}Pr_2$-bimy·HBr; ii) the inequivalence of two CH groups and four $CH_3$ groups of the iso-propyl branch, which are equivalent in ${}^{i}Pr_2$-bimy·HBr (Table 3).

Evaporation from concentrated solution of $\mathbf{P_I}$ in $CHCl_3$/ethanol afforded single crystals suitable for X-ray study. Some selected results are listed in Table 4. Interestingly, the result shows that complexes **2** and **3** in $\mathbf{P_I}$ crystalize isomorphously in the monoclinic with space group of P2(1)/c. The molecular formula of $\mathbf{P_I}$ clearly proves that the molar ratio of **2** : **3** is 3 : 1 with their molecular structures depicted in Fig. 2.

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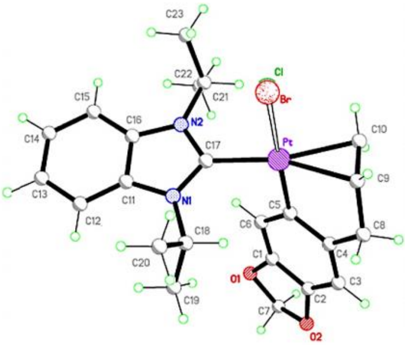

*Figure 2.* Molecular structure of $\mathbf{P_I}$.

*Table 4.* Selected crystallographic data and bond parameters of $\mathbf{P_I}$.

| | | | |
|---|---|---|---|
| Empirical formula | $C_{23}H_{27}N_2O_2Br_{0.25}Cl_{0.75}Pt$ ($P_I$) | Pt–Br | 2.486(8) |
| Formula weight | 605.12 | Pt–Cl | 2.397(6) |
| Crystal system | Monoclinic | Pt–C5 | 2.000(4) |
| Space group | P2(1)/c | Pt–C9 | 2.219(3) |
| Unit cell dimensions | | Pt–C10 | 2.212(4) |
| a = 10.1421(16) Å | $\alpha = 90.00^o$ | C9–C10 | 1.371(5) |
| b = 26.034(4) Å | $\beta = 115.641(3)^o$ | Pt–$C_{NCN}$ | 2.005(3) |
| c = 8.9055(14) Å | $\gamma = 90.00^o$ | C5–Pt–Br | 173.6(4) |
| Volume (Å$^3$) | 2119.9(6) | C5–Pt–Cl | 177.6(4) |
| Z | 4 | C5–Pt–$C_{NCN}$ | 90.1(6) |
| Density (calculated) | 1.896 mg/m$^3$ | Br–Pt–$C_{NCN}$ | 89.3(5) |
| Absorption coefficient | 7.207 mm$^{-1}$ | Cl–Pt–$C_{NCN}$ | 92.37(14) |
| Crystal size | 0.34 × 0.28 × 0.7 mm$^3$ | Cl–Pt–Br | 4.4(7) |
| Reflections collected | 14821 | | |
| Final R indices [I>2sigma(I)] | R1 = 0.0254, wR2 = 0.0609 | | |
| R indices (all data) | R1 = 0.0292, wR2 = 0.0622 | | |

The Pt(II) center is surrounded by the 75 % Cl and 25 % Br atoms and three different types of carbon donors, *i.e.* anionic aryl, neutral $^iPr_2$-bimy, $\eta^2$-olefin in a distorted square-planar geometry. The Cl and Br atoms are arranged in different positions in space, in which they make an angle of 4.4 (7)° with the Pt(II) atom (Cl–Pt–Br). The allyl group is indeed *trans*-position with the $^iPr_2$-bimy ligand. Comparing the four Pt–C bond distances in the coordination sphere shows that the Pt–$C_{NHC}$ and Pt–C5 bonds are very similar and smaller than Pt–C9 equal to Pt–C10 bond. These four bond distances are consistent with the distances observed in the complexes [PtX($^i$PrEug)(benzimidazolin-2-ylidenes)] [9, 10] analogous to **2** and **3**. Additionally, the C=C bond length in **2**, **3** and theirs analogues are much longer than that in the allylaryl fragments searched in Cambridge Structural Database [13]. This indicates the notably weaker olefinic character in these complexes than in the free arylolefins.

## 4. CONCLUSION

The interaction of the dimeric complex [Pt($\mu$-Cl)(Saf)]$_2$ (**1**) with the salt $^i$Pr$_2$-bimy·HBr in the presence of Ag$_2$O in acetone solvent was studied. The results of TLC, $^1$H NMR spectra and XRD showed that the obtained products were mixed complexes of [PtCl(Saf)($^i$Pr$_2$-bimy)] (**2**) and [PtBr(Saf)($^i$Pr$_2$-bimy)] (**3**) with different molar ratio depending on the used molar ratio of **1**:salt: Ag$_2$O. In the case of this ratio being 1 : 2 : 1.1, the mixture **P$_I$** with 75 % for the complex **2** was obtained. When the ratio was 1 : 4 : 1.1, 1 : 8 : 1.1 or 1 : 10 : 1.1, the obtained product was **P$_{II}$** with the molar ratio of **2** : **3** being 1 : 3. However, when both the salt and Ag$_2$O were increased to 1 : 4 : 2.2, 1 : 8 : 4.4 or 1 : 10 : 5.5, complexes **2** and **3** in **P$_{III}$** had equal molar proportions. The two main factors affecting the **2** : **3** molar ratio are the different solubility of AgCl and AgBr, and the different coordinating ability of Cl$^-$ and Br$^-$ ligands. The single crystal X-ray diffraction of **P$_I$** indicated that in **2** and **3** Saf coordinates with Pt(II) *via* C=C$_{olefinic}$ and C5 of the benzene ring. Meanwhile, $^i$Pr$_2$-bimy coordinates with Pt(II) through the C$_{NCN}$ and is in *trans*-position compared with the C=C$_{olefinic}$ bond.

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