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Formation of a Stable Cationic Hydridodimethylplatinum(IV) Complex by the Reaction of Bis(μ-dimethyl sulfido)tetramethyldiplatinum(II) with Protonated 1,4,7-Triisopropyl-1,4,7-triazacyclononane. A First Example of Oxidative N–H Addition to a Platinum(II) Complex

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Abstract — The complex bis(μ-dimethyl sulfido)tetramethyldiplatinum(II) reacts with 1,4,7-triisopropyl-1,4,7-triazacyclopropanone (L) over the course of 24 h at room temperature in THF-d_8 to give $[PtMe_2(L)]$ and $PtMe_2(SMe_2)_2$. Subsequent addition of 1 mol of trifluoromethanesulfonic acid results in immediate formation of a previously unknown stable cationic complex $[PtMe_2H(L)]^+SO_3CF_3^-$. This product can also be prepared by oxidative addition of the N–H bond of $LH^+SO_3CF_3^-$ to the starting complex, which points to a higher basicity of the platinum atom in $[PtMe_2(L)]$ compared with the nitrogen atom in the ligand L. The pK_a of the cationic hydride of platinum(IV) was estimated.

Alkylhydridoplatinum(IV) complexes discovered and characterized about five years ago [1–4] present interest as model compounds or potential intermediates in stoichiometric and catalytic activation of hydrocarbons with platinum(II) compounds [5]; they are formed by the oxidative addition scheme. The discovery that alkylplatinum(II) complexes are protonated under the action of Brønsted acids has been an important event in the history of alkylhydridoplatinum(IV) complexes. Such complexes are extremely unstable even at low temperatures if they contain monodentate N-, P-, or O-donor ligands [3], but their stability is much enhanced by *cis*-chelating bidentate [2] and, especially, *fac*-chelating tridentate N-donor ligands [1]. We previously theoretically interpreted the ability of the latter ligands to thermodynamic and kinetic stabilization of d^6-alkylhydridopalladiums [6]. Some our predictions as to the existence of yet unknown compounds of this class proved to be true [7]. The coordination of a *fac*-chelating ligand with a d^8 metal brings the frontier orbitals closer to each other, thus enhancing the nucleophilicity and basicity of the complex.

To obtain further experimental evidence for our theoretical predictions and aiming at designing lypophilic metal complexes capable of activating hydrocarbon C–H bonds in hydrocarbon media, we synthesized a new stable highly lypophilic cationic hydridomethyl platinum(IV) complex $[PtMe_2H(tacn\text{-}i\text{-}Pr_3)]^+SO_3CF_3^-$ (tacn-*i*-Pr_3 is 1,4,7-triisopropyl-1,4,7-triazacyclononane, L) (compound **I**). For the source of the dimethylplatinum fragment we chose the complex $[PtMe_2(SMe_2)]_2$ (compound **II**) [8]. A distinguishing feature of the developed synthetic procedure is that for the proton-donor reagent we used a salt derived from the ligand L, $LH^+SO_3CF_3^-$, which is much more convenient in operation [scheme (1)].

$$[PtMe_2(L')]_2 + LH^+SO_3CF_3^- \longrightarrow [PtMe_2H(L)]^+SO_3CF_3^-\ PtMe_2(L')_2. \quad (1)$$

Here L' is the dimethyl sulfide ligand exchanged for the ligand L in the course of the reaction.

Since in the course of the reaction platinum coordinates both with nitrogen and with hydrogen, this reaction can be considered as a first example of oxidative addition of ammonium N–H bond to pla tinum(II).

We found that the exchange of the dimethyl sulfide ligand in compound **II** for L is a slow reaction whose equilibrium is characterized by ca. 50% conversion of

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^{1}H NMR spectra (THF-d_8) of the compounds present in the reaction mixtures

Compound	δ, ppm (*J*, Hz)
$[PtMe_2H(tacn\text{-}i\text{-}Pr_3)]^+CF_3SO_3^-$	–21.6 s (1H, PtH, $^1J_{PtH}$ 1390), 0.80 s (6H, $PtCH_3$, $^2J_{PtH}$ 67), 1.26 d (6H, CCH_3, $^3J_{HH}$ 6.4), 1.29 d (6H, CCH_3, $^3J_{HH}$ 6.4), 1.43 d (6H, CCH_3, $^3J_{HH}$ 6.6), 3.10–3.55 m (CH and CH_2)
$[PtMe_2(SMe_2)]_2$	0.51 s (12H, $PtCH_3$, $^2J_{PtH}$ 88), 2.74 s (12H, SCH_3, $^3J_{PtH}$ 21)
$PtMe_2(SMe_2)_2$	0.51 s (6H, $PtCH_3$, $^2J_{PtH}$ 84), 2.36 s (12H, SCH_3, $^3J_{PtH}$ 24)
tacn-*i*-Pr_3	1.00 d (18H, CCH_3, $^3J_{HH}$ 6.2), 2.64 s (12H, NCH_2CH_2), 2.86 heptet (3H, NCH, $^3J_{HH}$ 6.2)
$[tacn\text{-}i\text{-}Pr_3H]^+CF_3SO_3^-$	1.22 d (18H, CCH_3, $^3J_{HH}$ 6.7), 2.80–2.95 m (6H, NCH[a]), 2.96–3.10 m (6H, NCH[b]), 3.28 heptet (3H, NCH, $^3J_{HH}$ 6.7), 9.9 s (1H, NH)
SMe_2	2.09 s
CH_4	0.23 s

compound **II** and is established within 20–30 h [scheme (2)].

$$[PtMe_2(L')]_2 + L \longrightarrow [PtMe_2(L)] + PtMe_2(L')_2. \quad (2)$$

Therefore, we first tried to synthesize compound **I** in two stages: to exchange L' for L in the absence of protonating agent and to introduce a powerful protonating agent, trifuoromethanesulfonic acid, only after the exchange equilibrium has been established.

On treatment with a small excess of trifluromethanesulfonic acid the complex dimethyl(1,4,7-triisopropyl-1,4,7-triazacyclononane)platinum(II) formed directly in the reaction mixture by scheme (2) immediately and completely converts into compound **I** [scheme (3)].

$$[PtMe_2(L)] + HSO_3CF_3 \longrightarrow [PtMe_2H(L)]^+SO_3CF_3^-. \quad (3)$$

The structure of the cation $[PtMe_2H(tacn\text{-}i\text{-}Pr_3)]^+$ was proved by ^{1}H NMR. The spectrum in tetrahydrofuran-d_8 contains signals, δ, ppm, of the hydrogen atom on platinum, –21.6 s (1H, PtH, $^1J_{PtH}$ 1390 Hz), two methyl groups, 0.51 s (6H, $PtSH_3$, $^2J_{PtH}$ 67 Hz), CH_3 protons of three nonequivalent isopropyl groups, 1.26 d (6H, CCH_3, $^3J_{HH}$ 6.4 Hz), 1.29 d (6H, CCH_3, $^3J_{HH}$ 6.4 Hz), 1.43 d (6H, CCH_3, $^3J_{HH}$ 6.6 Hz), ethylene fragments (ill-resolved multiplets), and isopropyl methine protons, 3.10–3.55 m (CH and CH_2). The multiplets at δ 3.10–3.20 and 3.21–3.32 ppm form a pattern characteristic of the triazacyclononane ring bound with a metal atom, and the multiplets at δ 3.32–3.42 and 3.42–3.55 ppm (*J* 6.4–6.6 Hz) are assigned to methine protons.

Further evidence for the proposed signal assignment and structure of the hydride comes from the similarity of the ^{1}H NMR spectra of solutions of $[PtMe_2H(tacn\text{-}i\text{-}Pr_3)]^+CF_3SO_3^-$, obtained by us, and the spectra of solutions of $[PtHMe_2(tacn)]^+CF_3SO_3^-$ in acetone-d_6, reported in [7], δ, ppm: –19.5 s (1H, PtH, $^1J_{PtH}$ 1339 Hz), 0.63 s (6H, $PtCH_3$, $^2J_{PtH}$ 70 Hz), 3.17 m (6H, CH_2), 3.35 m (6H, CH_2), 5.28 br (1H, NH), 5.75 br (2H, NH).

The nonequivalence of the isopropyl groups follows from their nonequivalent positions: One of them is attached to the nitrogen atom located *trans* to the hydride ligand in, apparently, scewed conformation, and, as follows from the quantum-chemical models of these systems, rendering nonequivalent the other two isopropyl groups. The isopropyl group that gives the most downfield signal is likely to be located oppositely to the hydride ligand.

According to ^{1}H NMR data, 40 min after addition of trifluoromethanesulfonic acid, the reaction mixture prepared from 1 mol of compound **I**, 1 mol of the ligand L, and 1 mol of the acid contained three platinum complexes, $[PtMe_2(SMe_2)]_2$, $PtMe_2(SMe_2)_2$ (**III**), and $[PtMe_2H(tacn\text{-}i\text{-}Pr_3)]^+CF_3SO_3^-$ (**I**) in a 12 : 46 : 42 molar ratio. One day after, the starting binuclear complex **I** was consumed completely, and the molar ratio between the two remaining complexes **III** : **I** became equal to 45 : 55. Moreover, excess free ligand L and its salt with excess trifluoromethanesulfonic acid, a little free dimethyl sulfide, and traces of methane were present. The chemical shifts and coupling constants of the mentioned compounds are listed in the table.

In a sealed ampule in tetrahydrofuran purified over potassium anthracene mirror, complex **I** is stable for at least 1 month. On contact with air it slowly decomposes with liberation of metallic platinum, methane, and 1,4,7-triisopropyl-1,4,7-triazacyclononane complexes. Ethane was not found among the

decomposition products. After 10-h heating at 80°C, the ^{1}H NMR spectrum no longer shows signals in the $PtMe_2$ resonance region.

In the ^{1}H NMR spectrum measured short after addition of the acid we found two closely located resonance signals, δ, ppm: –21.6 s (PtH, $^1J_{PtH}$ 1390 Hz) and –21.8 s (PtH, $^1J_{PtH}$ 1434 Hz), with close coupling constants. We suggest that they belong to two conformers of compound **I**. Actually, rotation of the isopropyl radical about the C–N bonds gives rise to three different staggered conformers of each of the isopropyl groups: +*gauche*, –*gauche*, and *trans*. Moreover, rotation of the CH_2 group of the ligand about the C–C bond changes the configuration of the spirochiral fragment Pt(tacn), which increases the number of possible rotational diastereomers. If the barrier to transfer of at least one of them into the most stable conformer is sufficiently high (> 92 kJ/mol), then the appearance of two upfield signals with close characteristics can be explained in terms of rotational isomerism. The integral intensity ratio of primarily observed signals is 83 : 17. After one day, only one upfield signal remains (δ –21.6 ppm), which, in the context of the concept of retarded conformational transitions, may suggest isomerization into the most thermodynamically stable conformer.

Assuming that coordination of L with platinum(II) increases the basicity of the latter so that it becomes more basic than the ligand nitrogen atom, we attempted to make use of this property of the platinum(II) derivatives studied for preparing complex **I** by a simplified scheme not involving consecutive ligand exchange [scheme (2)] and protonation [scheme (3)]. For the source of both ligand L and protonating agent we took a 1 : 1 ammonium salt of 1,4,7-triisopropyl-1,4,7-triazacyclononane with trifluoromethanesulfonic acid.

The reaction of complex **II** with a mixture containing equimolar (per platinum) quantity of 1,4,7-triisopropyl-1,4,7-triazacyclononane and half the quantity of trifluoromethanesulfonic acid, gave rise within 25–30 h at room temperature, to complete consumption of the ammonium salt and formation of the target compound **I**. The resulting ^{1}H NMR spectrum contained only one upfield signal (δ –21.6 ppm) belonging to the hydride ligand.

Within 5 days at room temperature, the concentration of the amine salt remained almost unchanged, and the starting platinum complex **II** disappeared almost completely, forming only little $PtMe_2(SMe_2)_2$ and methane. It is important to note that on mixing of $[PtMe_2(SMe_2)]_2$ with the salt (1 mol protonated ligand per 1 mol platinum) in the absence of free tacn-*i*-Pr_3 no hydride formation is observed. A possible reason here is that the ligand exchange involving the positively charged ion of the monoprotonated amine occurs very slowly.

The above result provides unambiguous evidence showing that the platinum(II) atom in the triazacyclononane complex possesses a higher basicity than the nitrogen atom in the free ligand. This result is also consistent with data of Prokopchuk *et al.* [7] who observed reversible protonation of $PtMe_2$(tacn) with methanol when the latter was used as a solvent. Reaction (1) can be considered as a first example of oxidative addition of the N–H bond of a tertiary ammonium ion to platinum(II).

To gain further information about the basicity of the central metal atom in compound **I**, we, like the above referees [7], could effect protonation of the corresponding dimethylplatinum(II) complex with such a weak acid as methyl alcohol. To this end, the ligand-exchange reaction (2) was perfomed directly in methanol-d_4, which allowed us to follow the reaction progress by ^{1}H NMR. It was found that the dimethylplatinum complex with the ligand L is not detected, like in the case of the tetrahydrofuran solution, but converts into the cationic species $[PtMe_2D(tacn\text{-}i\text{-}Pr_3)]^+$ whose counterion is the trideuteromethylate anion present in the solution [scheme (4)].

$$[PtMe_2(L')]_2 + L + CD_3OD \longrightarrow [PtMe_2D(L)]^+CD_3O^- + PtMe_2(L')_2. \quad (4)$$

Evidence for this conclusion comes the observation in the ^{1}H NMR spectrum (δ, ppm) of signals assignable to the methyl groups on platinum in the cationic hydride [0.74 s ($PtCH_3$, $^2J_{PtH}$ 69 Hz)] and the isopropyl methyl groups in the cation $[PtMe_2D(tacn\text{-}i\text{-}Pr_3)]^+$ [1.25 d (CCH_3, $^3J_{HH}$ 6.5 Hz), 1.43 d (CCH_3, $^3J_{HH}$ 6.6 Hz)], analogous to those we found for complex **I** in tetrahydrofuran-d_8, as well as those observed for $[PtMe_2D(tacn)]^+CD_3O^-$ in acetone-d_6 [7]: δ 0.60 ppm, s ($PtCH_3$, $^2J_{PtH}$ 69 Hz). The solution of $[PtMe_2D(L)]^+CD_3O^-$ in deuteromethanol is unstable and decomposes completely within 1 h.

The experiments performed earlier and described in the present work allow estimation of the basicity of platinum in the complexes $[PtMe_2(tacn)]$ and $[PtMe_2(tacn\text{-}i\text{-}Pr_3)]$. According to [7], $[PtHMe_2(tacn)]^+ \cdot CF_3SO_3^-$ does not change on treatment with excess pyridine, and, consequently, its $pK_{a(PtH^+)}$ is at least higher than 5.3 (pK_B of pyridine [9]). From our present data on the proton transfer from LH^+ onto the platinum atom in $[PtMe_2(L)]$ follows the estimate $pK_a(\mathbf{I}) > pK_a(LH^+) + 4$, provided that the detection

limit of the nonprotonated dimethylplatinum(II) complex and the LH^+ salt is less than 1% of the total quantity of the reaction products. Furthermore, from the deuteromethanol protonation experiment on the assumption [**I**] = [MeO^-] = [Pt] (total concentration of platinum 0.01 M), [**I**]/([Pt] – [**I**]) > 100 (corresponds to the detection limit of the starting complex) for [MeOH] 24.6 M, and, taking pK_a(MeOH) 15.5 [9], the basicity of platinum in $PtMe_2$(tacn) corresponds to $pK_a[PtMe_2H(tacn)^+] > 15.0$.

Thus, we obtained a new stable cationic hydridodimethylplatinum(V) complex and showed that the basicity of the metal atom in the complex of dimethylplatinum(II) with 1,4,7-triisopropyl-1,4,7-triazacyclononane in tetrahydrofuran is higher than that of the nitrogen atom in the free ligand. This finding allowed us to develop a new method of synthesis of the target compound via oxidative addition of the N–H bond in the corresponding ammonium salt to platinum(II).

EXPERIMENTAL

The ^{1}H NMR spectra were measured on Varian Unity-300 (300 MHz) and Bruker DRX-500 (500.1 MHz) instruments.

Bis(μ-dimethyl sulfido)tetramethyldiplatinum(II) (**II**) was synthesized by the procedure in [10].

1,4,7-Triisopropyl-1,4,7-triazacyclononane(tacn-*i*-Pr$_3$) was prepared by a modified procedure [11] without isolation of the ligand as a salt. A solution of 1.51 g of 1,4,7-triazacyclononane and 4.84 g isopropyl bromide in 7.5 ml of toluene was stirred at 80°C for 4 h. A colorless precipitate formed. Potassium hydroxide, 2.5 g, was stirred to the mixture, and it was allowed to stir for an additional 4 h at 80°C. The precipitate of potassium bromide of filtered off and washed with several portions of toluene. The combined toluene solutions were evaporated, dried at reduced pressure, and distilled at 100°C (0.05 mm) to obtain 2.12 g (71%) of the reaction product as a colorless oil. ^{1}H NMR spectrum ($CDCl_3$, 300 MHz), δ, ppm: 0.96 d (18H, CCH_3, $^3J_{HH}$ 6.5 Hz), 2.62 s (12H, NCH_2CH_2), 2.87 heptet (3H, NCH, $^3J_{HH}$ 6.5 Hz). ^{1}H NMR spectrum (C_6D_6, 500 MHz), δ, ppm: 0.97 d (18H, CCH_3, $^3J_{HH}$ 6.6 Hz), 2.63 s (12H, NCH_2CH_2), 2.79 heptet (3H, NCH, $^3J_{HH}$ 6.6 Hz). ^{1}H NMR spectrum [$(CD_3)_2CO$, 300 MHz)], δ, ppm: 0.95 d (18H, CCH_3, $^3J_{HH}$ 6.6 Hz), 2.60 s (12H, NCH_2CH_2), 2.80 heptet (3H, NCH, $^3J_{HH}$ 6.6 Hz).

Complex [PtMe$_2$H(tacn-*i*-Pr$_3$)]$^+$SO$_3$CF$_3^-$ (I). All experiments were performed in a glass all-sealed evacuated cell comprising two conjoint vertical cylindrical sections (knees) and equipped with an NMR ampule. Tetrahydrofuran-d_8 of analytical grade was purified over a potassium anthracene mirror immediately before use and distilled in a vacuum, collecting the distillate directly into the cell (the residual pressure over the solvent frozen with liquid nitrogen was ~10^{-3}–10^{-2} mm). The cell was sealed off from the vacuum line, and the reaction was performed. After the reaction had been complete, the NMR ampule was sealed off from the cell, and NMR spectra were recorded at regular intervals.

Protonation of a mixture of [PtMe$_2$(SMe$_2$)]$_2$ (II) and tacn-*i*-Pr$_3$ with trifluoromethanesulfonic acid. Compound **I**, 13.4 mg, and 13.0 mg of tacn-*i*-Pr_3 were placed into the different knees of the cell, and 1 ml of tetrahydrofuran-d_8 into the knees. The resulting transparent colorless solutions were mixed, and the reaction mixture was left to stand for 24 h at 20°C. Trifluoromethanesulfonic acid (2 μl) was introduced by breaking its containing evacuated glass ampule. By NMR we found in the solution $[PtMe_2(SMe_2)]_2$, $PtMe_2(SMe_2)_2$, and $[PtMe_2H(tacn\text{-}i\text{-}Pr_3)]^+CF_3SO_3^-$ in a 12 : 46 : 42 molar ratio. A day after, the starting binuclear complex **I** was consumed completely, and the **III** : **I** ratio became 45 : 55. The solution contained excess free ligand L, its salt with excess trifluoromethanesulfonic acid, and traces of dimethyl sulfide and methane. The NMR parameters of these compounds are listed in the table.

Reaction of complex II with a mixture of [tacn-*i*-Pr$_3$H$^+$]SO$_3$CF$_3^-$ and tacn-*i*-Pr$_3$. Complex **I**, 13.2 mg, and a mixture of tacn-*i*-Pr_3 with $[tacn\text{-}i\text{-}Pr_3H]^+CF_3SO_3^-$ were placed into different knees of the cell. The mixture was prepared by adding 2 μl of trifluoromethanesulfonic acid to a solution of 13.0 mg of tacn-*i*-Pr_3 in 1.5 ml of dry acetone, followed by vacuum evaporation to dryness. The solid compounds were dissolved in 1 ml of tetrahydrofuran-d_8, and the solutions were mixed. According to ^{1}H NMR data, after 25–30 h at room temperature the ammonium salt disappeared completely, and compound **I** formed. After 5 days at room temperature, the concentration of the amine salt did not change, while the starting platinum complex **II** disappeared almost completely, forming a little $PtMe_2(SMe_2)_2$ and methane (see table).

Formation of platinum(IV) hydrides in methanolic [PtMe$_2$(SMe$_2$)]$_2$ and tacn-*i*-Pr$_3$. Methanol-d_4 (0.8 ml) degassed and dried over molecular sieves was added under argon to a mixture of 6.2 mg of compound **II** and 6.5 mg of tacn-*i*-Pr_3, placed in an NMR ampule. The ampule was exposed to ultrasound for a short time for faster dissolution of compound **II**. After that, the ^{1}H NMR spectrum of the reaction mixture was recorded. The spectrum contained signals of the

Pt–Me groups of the cationic hydride [δ, 0.74 ppm, s (Pt–CH_3, $^2J_{PtH}$ 69 Hz)] and of the isopropyl methyl groups of the cation $[PtMe_2D(tacn\text{-}i\text{-}Pr_3)]^+$ [δ 1.25 ppm, d (C–CH_3, $^3J_{HH}$ 6.5 Hz), 1.43 ppm, d (C–CH_3, $^3J_{HH}$ 6.6 Hz)]. The solution of the complex $[PtMe_2D \cdot (L)]^+CD_3O^-$ completely decomposes within 1 h.

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