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Synthesis and X-ray crystal structure of a new μ-hydroxo dinuclear cobalt complex containing one *cis*-β folded and one planar salen moiety

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Abstract

The reduction of $[Co(III)(tmsalen)(py)_2]^+$ with $NaBH_4/PdCl_2$ and the successive oxidative addition of CH_2ClI, carried out in neutral methanolic solution and followed by the addition of NaOH, afford a new dinuclear complex. The molecular structure reveals that it is formed by an octahedral [Co(tmsalen)(py)(OH)] unit connected to a β-folded $[Co(tmsalenCH_2)]^+$ fragment, in such a way that the latter metal completes the coordination sphere with the hydroxo group and a tmsalen oxygen from the former unit.

Keywords: Organocobalt; Salen; Dinuclear complex; X-ray structure

1. Introduction

There is currently a renewed interest toward metal complexes in which the equatorial ligand is a tetradentate Schiff base derived from salicylaldehyde and diamines (salen-type ligands), in connection with a number of possible applications, which range from asymmetric catalysis [1] to material sciences [2]. These complexes generally adopt a *trans* planar geometry but, in some instances, the quadridentate ligand may assume a *cis*-β configuration [3].

We have reported the synthesis and the characterization of several organometallic RCo(tmsalen) derivatives, where tmsalen = 4,4′,7,7′ tetramethylsalen [4,5]. The organometallic complexes were prepared by the reduction of $[Co(III)(tmsalen)(py)_2]ClO_4$ (**1**) (Chart 1) with $NaBH_4/PdCl_2$ in alkaline methanolic solution, followed by the oxidative addition of the appropriate alkyl iodide. Available structural data show that the RCo(tmsalen) complexes, either dimers or pentacoordinated [4], adopt a planar *trans* geometry except for $R = CH_2Cl$. In this case the reaction gave, beside the expected *trans* organometallic species **2** (Chart 1), the *cis*-β organometallic derivative $[Co(tmsalenCH_2)(py)(H_2O)]^+$, **3** (Chart 1), formed by intramolecular reaction of the axial chloromethyl group with the equatorial chelate [5]. Previous examples of intramolecular reaction of CH_2X axial groups (X = Cl, Br, I) with imino-oxime or amino-oxime equatorial ligands have been described [6]. In both cases the generation of an equatorial negatively charged nitrogen is required and therefore the reaction occurs only in strongly basic medium. In the present case, the negative charge on the oxygen atom makes it prone to a nucleophilic attack on the CH_2 group, so that the cyclization occurs also in neutral medium. Quite unexpectedly, when in the course of a synthesis of **3**, the reduction–alkylation sequence was carried out before the addition of sodium hydroxide, a new dinuclear species was obtained. We report here the synthesis and the structural characterization of this new complex, suggesting a plausible mechanism for its formation.

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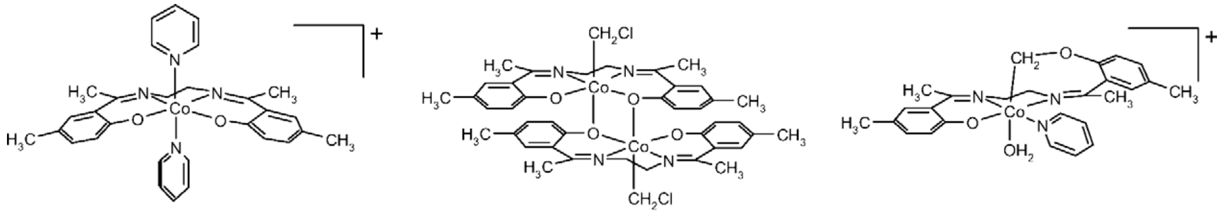

1 2 3

Chart 1.

2. Results and discussion

2.1. Synthesis

The standard sequence for the synthesis of the RCo(tmsalen) derivatives involves the reduction of [Co(III)(tmsalen)$(py)_2]^+$ with $NaBH_4/PdCl_2$ in alkaline methanolic solution, followed by the oxidative addition of the appropriate alkyl iodide. When the alkylating agent is CH_2ClI, this sequence allows to isolate the *trans* derivative **2** (Chart 1), if the precipitate is recovered from the reaction mixture within one or two hours [5]. If it is left for longer time in contact with the reaction mixture, a further process occurs, which leads to the formation of [Co(tmsalenCH_2)(py)$(H_2O)]^+$, **3** (Chart 1) [5]. In **3**, the tetradentate ligand assumes a *cis*-β configuration, with the formation of a seven membered ring, the other two positions being occupied by one py and one water molecule. The product is obtained as a racemic mixture of Δ and Λ enantiomers.

In the course of a synthesis of **3**, the addition of NaOH was accidentally delayed after the reduction and the alkylation, so that these processes occurred in almost neutral medium (about pH 8). In these experimental conditions, a novel dinuclear species **4** was recovered (Fig. 1). As the only difference between the syntheses of **3** [5] and **4** is the timing of the addition of NaOH, the formation of **4** under these experimental conditions may be rationalized by evaluating the effect of pH on the rate of the three consecutive reaction steps (reduction, alkylation, and cyclization).

It is well known that the reduction of the Co(III)salen complexes with $NaBH_4$ does not occur in neutral or mildly alkaline solutions, owing to the high Co(II)/Co(I) reduction potential, which causes the instability of the Co(I) nucleophile [7]. The reduction is possible in alkaline solution (pH >13) or in presence of Pd^{2+} [7]. Furthermore, in less basic solution, the Co(I) species may pick up a proton

$$\mathrm{Co(I)} + \mathrm{H}^+ \rightleftarrows \mathrm{Co(III)} - \mathrm{H}$$

to form what is formally a cobalt(III)-hydrido species, which is much less nucleophilic than the Co(I) species [8]. These evidences suggest that both the reduction and the alkylation may be considerably slower in almost neutral solution.

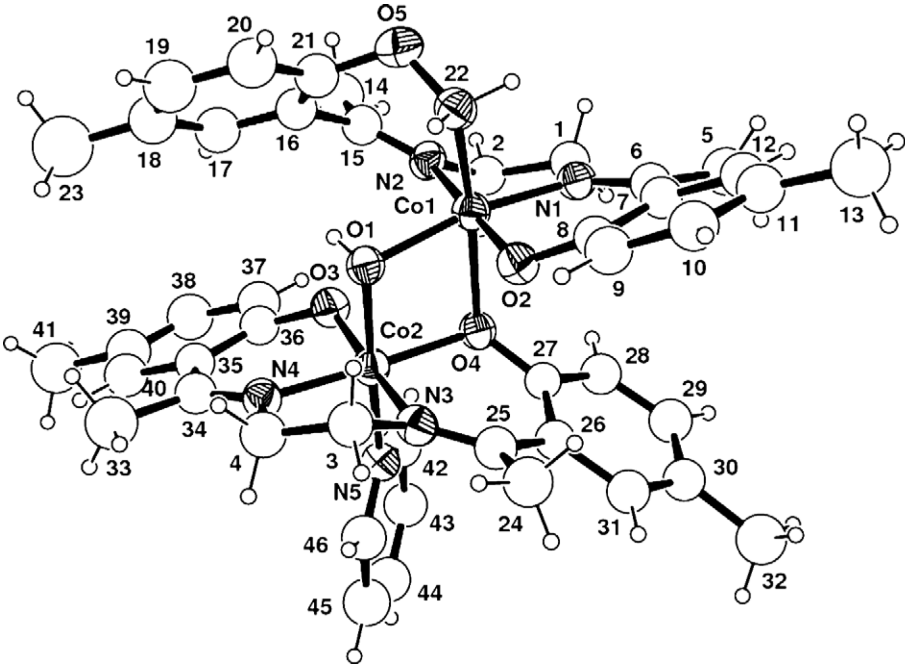

Fig. 1. ORTEP drawing of the complex cation of **4** (ellipsoids at 30% probability level, only number of C atoms indicated for sake of clarity).

We have studied the effect of pH on the cyclization rate of **2** in the range 5.6–13.0. Kinetic data (Table 1) show that the cyclization rate is independent on pH in the range 5.6–11, increases at pH >11, and is almost double at pH 13. Therefore, when the reaction is carried out in almost neutral solution, a non negligible amount of **1** may be still present in solution, while the already alkylated complex is undergoing cyclization. The final addition of NaOH promotes the formation of the μ-hydroxo dinuclear complex.

To test this hypothesis, we reacted equimolar amounts of **1** and **3** with NaOH in aqueous methanol. The dinuclear complex **4** was effectively recovered from the solution by addition of water and was identified by ^{1}H and ^{13}C NMR spectroscopy.

2.2. NMR characterization

The ^{1}H NMR spectrum of **4** is quite complex, as neither the two halves of the planar tmsalen ligand or those of the non planar tmsalenCH$_2$ are chemically equivalent. The protons belonging to the same ligand can be easily identified through ^{1}H^{1}H COSY and ROESY experiments, but assigning the signals to one of the two non equivalent halves of each ligand is not straightforward. For tmsalenCH$_2$, the assignment has been carried out by analogy with the spectrum of **3** [5]. In the ^{13}C NMR spectrum of **3**, the *C*H$_3$C=N carbon of the planar half resonates at 18.53 ppm and that of the folded one at 24.26 ppm. Consequently, in the ^{13}C NMR spectrum of **4**, the signal at 17.91 ppm was assigned to C5 and that at 23.82 to C14. A HSQC experiment allowed the identification of the proton bonded to C14 and, consequently, the full assignment of the proton signals of the Co(tmsalenCH$_2$) fragment. For (tmsalen), the assignment has been made on the basis of a cross-peak in the ROESY spectrum between *H*-C9 and *H*-C24, which allowed to assign the latter signal to the half of tmsalen ligand which is O-bonded to the cobalt of the other unit. The HSQC experiment allowed also a partial complete assignment of the ^{13}C spectrum.

The most striking feature in the ^{1}H NMR spectrum of **4** is the singlet at −5.60 ppm, due to to the proton of the μ-OH group, which disappears very fast by addition of CD$_3$OD. Furthermore, it is noteworthy the strong deshielding of *H*-C2, which resonates at 6.11 ppm, as a consequence of the magnetic anisotropy of the three aromatic rings in its neighbourhood.

Table 1
Rate constants for the cyclization of **2** at various pH in 50% CH$_3$OH – 50% H$_2$O (w/w) at 25 °C ($I = 0.1$, NaClO$_4$)

$k_{\text{obs}} \times 10^{-4}$ (s^{-1})	pH
(2.00 ± 0.01)	5.6
(2.02 ± 0.01)	7.1
(1.91 ± 0.01)	7.6
(1.91 ± 0.01)	8.2
(1.88 ± 0.01)	9.2
(1.90 ± 0.01)	9.7
(1.82 ± 0.01)	11.0
(2.46 ± 0.01)	12.0
(4.93 ± 0.14)	13.0

2.3. X-ray structure

Crystals of **4** are built up by dinuclear complex cations, perchlorate anions and disordered lattice water molecules. The ORTEP drawing of the complex cation with the atom numbering scheme is depicted in Fig. 1. The coordination distances and angles are given in Table 2.

The dinuclear species can be described as formed by an octahedral [Co(tmsalen)(py)(OH)] unit connected to a β-folded Co(tmsalenCH$_2$) fragment, in such a way that the latter metal completes the coordination sphere with the hydroxo group and a tmsalen oxygen from the former unit (Fig. 1). The distance measured between the metals is 3.000(1) Å. The tetradentate tmsalen coordinates Co(2) in the four equatorial positions, while an hydroxo and a pyridine ligand are located at the axial positions. The overall conformation of the equatorial tmsalen moiety can be described as an asymmetric umbrella shape with α and β angles of 20.0(2)° and 29.7(2)°, where α and β are the dihedral angles between the N$_2$O$_2$ equatorial donor plane and the two salycilaldiminate residues, respectively [9]. The equatorial N$_2$O$_2$ plane presents nearly coplanar atoms, with the metal slightly displaced by 0.060(3) Å from the mean plane towards the pyridine nitrogen.

On the other hand, the folded tmsalenCH$_2$ ligand coordinates Co(1) in a *cis*-β fashion, forming a seven-membered metallocycle with a boat-like conformation, the other two positions about the cobalt being occupied by a bridging OH group and by the O(4) atom from the Co(2) coordination sphere. The axial distances Co(1)–C(22) and Co(1)–

Table 2
Selected bond lengths (Å) and bond angles (°) for compound **4**

Co(1)–O(1)	1.938(5)	Co(2)–O(1)	1.914(5)
Co(1)–O(2)	1.867(5)	Co(2)–O(3)	1.876(5)
Co(1)–O(4)	2.210(5)	Co(2)–O(4)	1.899(4)
Co(1)–N(1)	1.890(6)	Co(2)–N(3)	1.915(6)
Co(1)–N(2)	1.923(6)	Co(2)–N(4)	1.903(6)
Co(1)–C(22)	1.931(7)	Co(2)–N(5)	1.964(6)
O(1)–Co(1)–O(2)	85.3(2)	O(1)–Co(2)–O(3)	89.2(2)
O(1)–Co(1)–O(4)	77.70(18)	O(1)–Co(2)–O(4)	86.4(2)
O(1)–Co(1)–N(1)	168.1(2)	O(1)–Co(2)–N(3)	87.8(2)
O(1)–Co(1)–N(2)	95.6(2)	O(1)–Co(2)–N(4)	89.6(2)
O(1)–Co(1)–C(22)	95.6(3)	O(1)–Co(2)–N(5)	176.4(3)
O(2)–Co(1)–O(4)	89.94(19)	O(3)–Co(2)–O(4)	87.8(2)
O(2)–Co(1)–N(1)	93.4(2)	O(3)–Co(2)–N(3)	176.9(2)
O(2)–Co(1)–N(2)	179.1(2)	O(3)–Co(2)–N(4)	93.5(2)
O(2)–Co(1)–C(22)	86.3(3)	O(3)–Co(2)–N(5)	88.7(2)
O(4)–Co(1)–N(1)	90.4(2)	O(4)–Co(2)–N(3)	91.3(2)
O(4)–Co(1)–N(2)	90.0(2)	O(4)–Co(2)–N(4)	175.9(2)
O(4)–Co(1)–C(22)	172.6(3)	O(4)–Co(2)–N(5)	90.5(2)
N(1)–Co(1)–N(2)	85.7(2)	N(3)–Co(2)–N(4)	87.2(3)
N(1)–Co(1)–C(22)	96.2(3)	N(3)–Co(2)–N(5)	94.3(3)
N(2)–Co(1)–C(22)	93.9(3)	N(4)–Co(2)–N(5)	93.4(3)
Co(2)–O(1)–Co(1)	102.3(2)	Co(2)–O(4)–Co(1)	93.45(19)

O(4) have values of 1.931(7) and 2.210(5) Å, respectively, with the oxygen that undergoes the strong trans effect of the methylene group. The coordination sphere around Co(1) deviate considerable from the ideal geometry, and in this respect it is worth of note the narrow angle O(1)–Co(1)–O(4) of 77.70(18)°. The equatorial coordination plane of Co(1) shows a slight tetrahedral distortion (deviations of $\pm 0.097(3)$ Å), with the cobalt displaced by 0.100(3) Å towards the axial carbon atom. It should be noted that the Co(1)–N(1) distance of 1.890(6) Å is significantly shorter than the Co(1)–N(2) one (1.923(6) Å), a feature already found in **3** and in agreement with theoretical DFT calculations [5]. The Co(1)–OH–Co(2) bridge is characterized by Co(1)–O and Co(2)–O distances of 1.938(5) and 1.914(5) Å, respectively and by a Co–O–Co angle of 102.3(2)°. A comparison of the O–Co–C axial fragment in the present complex **4** with data of Co(CH$_2$tmsalen)(py)(H$_2$O) (**3**) [5] indicates close comparable long Co-O distances, of 2.210(5) and 2.213(3) Å in **4** and **3**, respectively, while the present Co–C distance (1.931(7) Å) appears slightly shorter than that of 1.965(4) Å measured in **3**.

In summary, the geometry of the *cis*-β folded ligand is very similar to that found in **3** and the geometry of the Co(tmsalen)(py) unit is close to that observed in the [Co(tmsalen)(L)$_2$]$^+$ cations (L = py (**1**) and N-MeIm) [4].

3. Conclusion

We have shown that the reduction of [Co(III)(tmsalen)(py)$_2$]ClO$_4$ with NaBH$_4$/PdCl$_2$ in methanolic solution, followed by the oxidative addition of CH$_2$ClI may afford the planar dimeric species **2** [4], the folded complex **3** [5], or the dinuclear species **4** with a relatively little variation of the experimental conditions (time of reaction and pH). In particular, the dinuclear species **3** has been obtained by carrying both the reduction and the alkylation in neutral medium, with a final addition of NaOH. Further investigations are aimed to investigate the effect of the substitution of the neutral ligand.

4. Experimental

4.1. General information

All the manipulations were performed in the dark. [Co(III)(tmsalen)(py)$_2$]ClO$_4$ was synthesized as previously described [4]. All other reagents were analytical grade and used without further purification. NMR spectra were recorded on a Jeol EX-400 (^{1}H at 400 MHz and ^{13}C at 100.4 MHz). Electrospray mass spectra were recorded in positive mode by using an API 1 mass spectrometer (Perkin–Elmer). Reaction kinetics were recorded using a Uvikon 941 plus (Kontron Instruments). The pH of the solutions was measured using a Radiometer PHM 220 pH meter.

Caution: Perchlorate salts of metal complexes with organic ligands are potentially explosive. Only small amounts of material should be prepared, and these should be handled with great care.

4.2. Synthesis

Method A: [Co(III)(tmsalen)(py)$_2$]ClO$_4$ (0.25 g, 0.4 mmol) was suspended in MeOH (100 ml) and the suspension was deaerated several times with continuous stirring. NaBH$_4$ (0.035 g, 1 mmol) in H$_2$O (2 ml) was added to the suspension under nitrogen, followed by the addition of few drops of an aqueous 10% PdCl$_2$ solution. A very fast reduction occurred, evidenced by the instantaneous change of the suspension color from light brown to red. CH$_2$ClI (1.3 ml) was added, and the stirring was continued. One pellet of NaOH dissolved in 1 ml of water was added after 1 h. A brown solution was obtained and was left aside for 2 h. After filtration, the solution was treated with 100 ml of cold water, added drop by drop. The precipitate was collected by filtration, washed with water, and dried in air. X-ray-quality crystals were obtained by recrystallization from a methanol/water solution. Yield: 30 mg (15%). Calc. for C$_{46}$H$_{52}$ClCo$_2$N$_5$O$_9$: C, 56.82; H, 5.39; N, 7.20. Found: C, 56.60; H, 5.40; N, 6.81%. ESI-MS (90 V, CH$_3$OH, m/z^+) calcd for C$_{46}$H$_{52}$ClCo$_2$N$_5$O$_9$, 972.25; found 872.8 (**4** ClO$_4^-$, 80%).

^{1}H NMR (CDCl$_3$, TMS, see Fig. 1 for numbering scheme): δ (ppm) = −5.60 (s, 1H, O–H), 1.88 (s, 3H, H–C32), 1.89 (s, 3H, H–C23), 2.08 (s, 3H, H–C13), 2.12 (s, 3H, H–C41), 2.13 (s, 3H, H–C5), 2.51 (s, 3H, H–C33), 2.55 (s, 3H, H–C24), 2.56 (s, 3H, H–C14), 3.32 (m, 1H, H–C1), 3.65 (m, 1H, H–C4), 3.93 (m, 1H, H–C1), 4.20 (m, 2H, H–C2 and H–C3), 4.41 (m, 2H, H–C3 and H–C4), 6.11 (m, 1H, H–C2), 6.39 (s, 1H, H–C17), 6.45 (d, 1H, J = 8.6 Hz, H–C9), 6.69 (m, 2H, H–C29 and H–C31), 6.74 (m, 2H, H–C10 and H–C22), 6.86 (s, 1H, H–C12), 6.90–6. 98 (m, 4H, H–C22, H–C38, H–C42 and H–C45), 7.03–7.09 (m, 5H, H–C19, H–C20, H–C28, H–C37 and H–C40), 7.18 (m, 1H, H–C44), 8.29 (d, 2H, J = 5.9 Hz, H–C41 and H–C46); ^{13}C NMR (CDCl$_3$, TMS, see Fig. 1 for numbering scheme): δ (ppm) = 17.91 (C5), 19.03 (C33), 20.10 (C24), 20.30 (C32), 20.34 (C41), 20.41 (C13), 20.50 (C23), 23.82 (C14), 53.94 (C3), 54.14 (C4), 55.03 (C2), 55.82 (C1), 81.94 (C22), 120.88, 120.96, 121.91, 122.00, 122.76 (C9), 122.91, 123.22, 123.91, 125.70, 128.52, 128.63 (C12), 129.47 (C17), 129.89, 131.37, 132.35, 132.79, 133.39, 134.39, 137.84 (C44), 153.61 (C46, C41), 153.81, 160.61, 163.67, 163.96, 169.76, 172.86, 174.28, 175.51.

Method B: [Co(III)(tmsalen)(py)$_2$]ClO$_4$ (44.2 mg, 70×10^{-3} mmol) and **3** (40.6 mg, 70×10^{-3} mmol) were mixed in methanol (50 ml) and 1 pellet of NaOH, previously dissolved in a little amount of water, was added. The beaker was covered and left in refrigerator overnight. The solution was then treated with 100 ml of cold water, added drop by drop. The precipitate was collected by filtration, washed with water, and dried in air. Yield: 10 mg (15%).

4.3. Kinetics

The cyclization kinetics of **2** were followed spectrophotometrically in the mixed solvent 50% CH_3OH – 50% H_2O (w/w) in the pH range 5.6–13.0 at 25 °C ($I = 0.1$ M using $NaClO_4$). The buffer solutions (pH 5.6–9.7) were prepared according to Ref. [10]. In the pH range 11.0–13.0, an excess of NaOH was used to keep constant the pH value. The reaction progress was monitored by observing absorbance changes at 325 nm. The k_{obs} rate constants were obtained from the linear plots of $\ln(A - A_\infty)$ versus time.

4.4. X-ray structure determination

The data collection was carried out at 293(3) K using Mo Kα radiation ($\lambda = 0.71073$ Å) on a Nonius DIP-1030 H system. Cell refinement, indexing and scaling of the data set were performed using the DENZO and SCALEPACK suite of programs [11]. The structure was solved by Patterson and subsequent Fourier analyses and refined by full-matrix least-squares on F^2 with all observed reflections [12]. Due to the low number of reflections with $I > 2\sigma(I)$, all carbon atoms (except C(22) bound to cobalt) were isotropically refined. A residual in the ΔF map was interpreted as a water oxygen (factor occupancy = 0.5), at 2.733 Å from a perchlorate oxygen. The contribution of H atoms at calculated positions, except the OH hydrogen located on the ΔF map, was included in the final cycles of refinement; H atoms of the lattice water were not included. All the calculations were performed using the WINGX System, Ver 1.70.01[13].

Crystallographic data for $\mathbf{4} \cdot 0.5(H_2O)$: $C_{46}H_{53}ClCo_2N_5O_{9.5}$, $M = 981.24$, monoclinic, space group $P2_1/c$, $a = 10.887(3)$, $b = 18.167(4)$, $c = 24.507(4)$ Å, $\beta = 99.09(3)°$, $V = 4786.3(19)$ Å^3, $Z = 4$, $D_c = 1.362$ g/cm^3, μ(Mo Kα) = 0.808 mm^{-1}, $F(000) = 2044$, θ range = 2.02–23.29°. Final $R_1 = 0.0672$, $wR_2 = 0.1726$, $S = 0.979$ for 355 parameters and 25964 reflections, 6099 unique [$R_{int} = 0.0747$], of which 2926 with $I > 2\sigma(I)$, max positive and negative peaks in ΔF map 0.496, −0.392 e Å^{-3}.

5. Supplementary material

Crystallographic data for compound **4** have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication CCDC 298822. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK [fax: (+44) 1223 336033, e-mail: deposit@ccdc.cam.ac.uk].

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