

New organobimetallic compounds containing catecholate and *o*-semiquinolate ligands

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Abstract

New organobimetallic compounds $\text{CatSn}[\text{CpM}(\text{CO})_n]_2$ (**2–4**) were obtained by the insertion of $\text{CatSn}(\text{II})$ (**1**) into the metal–metal bond of $[\text{CpM}(\text{CO})_n]_2$ (Cat – 3,6-di-*tert*-butylcatecholate dianion; M = Fe (**2**), $n = 2$; M = Mo (**3**), W (**4**), $n = 3$). The structure of $\text{CatSn}[\text{CpMo}(\text{CO})_3]_2$ was determined by X-ray analysis. The oxidation of compounds **2–4** with silver(I) triflate was found to produce stable paramagnetic *o*-semiquinolate derivatives which keep both Sn–M bonds. New paramagnetic tin(IV) complexes were investigated by EPR spectroscopy.

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1. Introduction

The one of the promising fields in modern organometallic and coordination chemistry is the metal complexes with “redox-active” ligands including various reduced forms of *o*-quinones, *o*-iminoquinones, *o*-diimines and their derivatives. The interest in this type of ligands is caused by ability to the reversible electron addition and to preservation of coordination bonds with metal at the same time. The most used “redox-active” ligands are *o*-quinones and their reduced forms. The overwhelming majority of complexes containing this type of ligand were synthesized for transition metals. Some of these complexes have unique magnetic and electronic properties [1]. Earlier, *o*-semiquinolate radical-anions were successfully used as spin-labelled ligands [2]. EPR spectra of such complexes can provide rich information on their structures and mechanisms of transformation in solution [3]. At the same time these data are very pure for compounds containing metal–metal bonds. Previ-

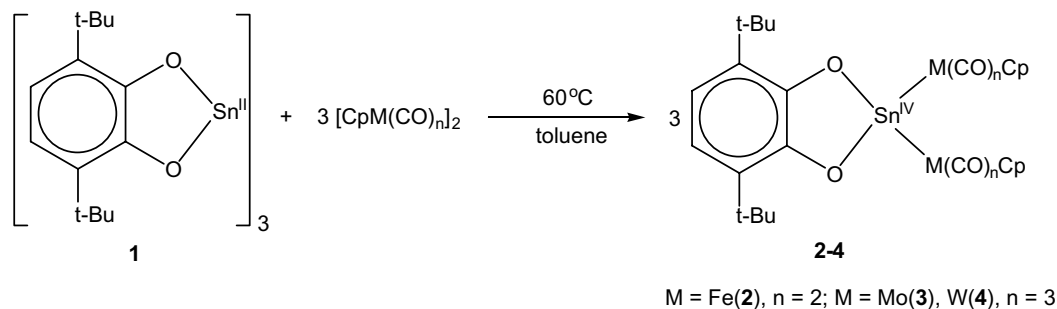
ous studies only revealed the formation of intermediates but no any compounds were isolated in individual state [4]. In the present study, we have synthesized and characterized organobimetallic derivatives involving 3,6-di-*tert*-butylcatecholate and 3,6-di-*tert*-butyl-*o*-semiquinolate ligands and Sn–M (M = Fe, Mo, W) bonds.

2. Results and discussion

The insertion of tin(II) derivatives into metal–metal bond is known to be a versatile method for preparing tin(IV) compounds containing Sn–M bonds [5]. We have found that the interaction of trimeric tin(II) 3,6-di-*tert*-butylcatecholate (**1**) [6] with cyclopentadienyl metal carbonyls $[\text{CpM}(\text{CO})_n]_2$ (M = Fe, $n = 2$; M = Mo, W, $n = 3$) allows to obtain new complexes (**2–4**) with good yields according to Scheme 1. The reaction was carried out in toluene under heating and stirring conditions. The initial deposits of **1** and $[\text{CpM}(\text{CO})_n]_2$ dissolved during reaction to give red (M = Fe) or red-orange (M = Mo, W) solutions. Compounds **2–4** are stable on air in crystalline state for several days but slowly decompose in solutions.

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Scheme 1.

It is necessary to note that the analogous reaction between these organometallic dimers and tin(II) complex containing chelated diazabutadiene dianion leads to the formation of unstable three-coordinated paramagnetic tin(II) derivatives [7].

Compounds **2–4** were characterized by elemental, IR- and ^1H , ^{13}C and ^{119}Sn NMR-analysis. The structure of complex **3** was determined by the X-ray diffraction.

The formation of catecholate complexes **2–4** is evident from a number of typical bands in their IR spectra in the range of $700\text{--}2100\text{ cm}^{-1}$. The $\nu(\text{C--O})$ vibrations of catecholate C–O bonds appear at about $1200\text{--}1300\text{ cm}^{-1}$. Furthermore, IR exhibits a number of distinct vibration bands of carbonyls in the range of $1900\text{--}2100\text{ cm}^{-1}$ as it was observed for related complexes based on cyclopentadienylmetal carbonyl fragments (e.g. $[\text{CpM}(\text{CO})_n]_2\text{SnCl}_2$ [8]). ^1H and ^{13}C NMR spectra of **2–4** at room temperature display one single resonance corresponding to cyclopentadienyl rings. The ^{13}C NMR spectrum shows only one resonance in the diagnostic region of carbonyl carbon atoms for **2** and two ones (1:2) for **3**, **4**. It indicates the equivalency of transition metal fragments in coordination sphere of tin atom in solutions of complexes **2–4**.

The molecular structure of **3** is illustrated in Fig. 1. Selected bond lengths and angles are presented in Table 1. The solid-state structure exhibits a bridging “CatSn” moiety bonded to two $[\text{CpMo}(\text{CO})_3]$ units via Sn–Mo bonds. The coordination geometry of tin atom deviates from an ideal tetrahedral geometry. The angle in catecholate chelate O–Sn–O is $81.95(16)^\circ$. This value is typical for tin(IV) 3,6-di-*tert*-butylcatecholate derivatives ($81\text{--}85^\circ$) [6,9] but noticeably more than analogous angle in initial complex **1** ($65.19\text{--}71.12^\circ$) [6]. The sufficiently large Mo–Sn–Mo angle ($126.74(2)^\circ$) is caused by the sterical repulsion between large transition metal fragments. Cp-ligands of transition metal fragments are situated in *trans*-position to each other that minimizes non valent interaction between them. However, it is common for compounds containing $\text{Cp}(\text{CO})_3\text{Mo--Sn--Mo}(\text{CO})_3\text{Cp}$ fragment [10]. Mo–Sn bond distances ($2.7869(7)$, $2.8170(7)$ Å) are very close to those observed for $[\text{Cp}(\text{CO})_3\text{Mo}]_2\text{SnBr}_2$ (2.794 , 2.809 Å [10c]). Distances C–O of catecholate ligand ($1.374(7)$, $1.366(7)$ Å) lie accurately in the range of single C–O bond lengths in metal catecholates (from 1.33 Å to

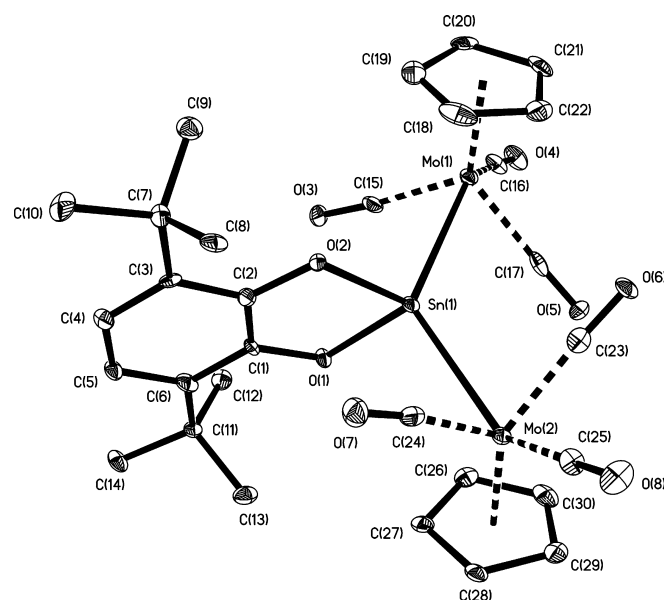


Fig. 1. An ORTEP view of molecule **3** with 30% thermal probability ellipsoids. H atoms are omitted for clarity.

Table 1
Selected bond distances and angles of complex **3**

Bond	<i>r</i> (Å)	Angle	ω ($^\circ$)
Sn(1)–O(1)	2.021(4)	O(1)–Sn(1)–O(2)	81.95(16)
Sn(1)–O(2)	2.027(4)	O(1)–Sn(1)–Mo(1)	114.54(12)
Sn(1)–Mo(1)	2.7869(7)	O(2)–Sn(1)–Mo(1)	106.19(11)
Sn(1)–Mo(2)	2.8170(7)	O(1)–Sn(1)–Mo(2)	103.81(12)
O(1)–C(1)	1.374(7)	O(2)–Sn(1)–Mo(2)	114.82(11)
O(2)–C(2)	1.366(7)	Mo(1)–Sn(1)–Mo(2)	126.74(2)
C(1)–C(2)	1.397(9)	C(1)–O(1)–Sn(1)	111.6(4)
C(1)–C(6)	1.399(9)	C(2)–O(2)–Sn(1)	111.5(4)
C(2)–C(3)	1.412(8)		
C(3)–C(4)	1.384(9)		
C(4)–C(5)	1.395(9)		
C(5)–C(6)	1.415(9)		

1.39 Å [11]). Geometrical parameters of this ligand are typical for *O,O*-coordinated catecholate dianions.

It was found that complexes **2–4** can be oxidized in toluene solution with silver triflate to produce new paramagnetic tin(IV) complexes. The reaction is accompanied by the silver metal precipitation. The resulting violet reaction mixture exhibits strong signal in EPR which indicates the

oxidation of the catechololate ligand of initial complexes to *o*-semiquinolone one (Scheme 2).

Complexes **5–7** are stable in toluene solutions for a long time under light-protection conditions. Compound **6** was isolated in a pure state as thin violet needles. The IR spectrum of **6** shows $\nu(\text{C}\cdots\text{O})$ vibrations at 1435, 1411 cm^{-1} which are characteristic for *o*-semiquinolone metal derivatives [11a]. At the same time spectrum remains vibrations of carbonyls in the range of 1900–2100 cm^{-1} . It confirms the presence of cyclopentadienylmetal carbonyl fragments in the resulting compound.

EPR-spectra of complexes **5–7** are well resolved at room temperature (Figs. 2 and 3). The hyperfine structure arises from hyperfine coupling (HFC) of unpaired electron with magnetic nuclei [12] ^1H (99.98%, $I = 1/2$, $\mu_N = 2.7928$), ^{117}Sn (7.68%, $I = 1/2$, $\mu_N = 1.000$), ^{119}Sn (8.58%, $I = 1/2$, $\mu_N = 1.046$) and ^{57}Fe (2.1%, $I = 1/2$, $\mu_N = 0.09044$) for **5**, ^{95}Mo (15.92%, $I = 5/2$, $\mu_N = 0.9133$), ^{97}Mo (9.55%, $I = 5/2$, $\mu_N = 0.9335$) for **6** and ^{183}W (14.28%, $I = 1/2$, $\mu_N = 0.111778$) for **7**. EPR parameters are summarized in Table 2.

HFC constants due to magnetic isotopes of tin $A_i(^{117}\text{Sn})$ and $A_i(^{119}\text{Sn})$ in EPR-spectra of **5–7** exceed values peculiar to tin(IV) complexes (5–20 G) containing *o*-semiquinolone ligand [3b,9,13]. But they are very close to those obtained for tin(IV) *o*-iminosemiquinolone derivative [14]. HFC constants increasing are caused by decreasing of electron withdrawing properties of organometallic substituents in comparison with alkyl, aryl or carboxyl groups. The analogous changes were obtained in EPR-spectra of unstable paramagnetic tin(II) complexes with radical-anion of diazabutadiene [7].

EPR-spectra of complexes **5–7** show well-resolved satellite lines caused by isotopes of transition metals. Complexes **5–7** contain two Fe, Mo or W atoms correspondingly. Therefore it is possible to obtain signals in EPR-spectra of different isotope forms of these compounds (one or two magnetic atom) in addition to main signal which do not include magnetic isotopes of transition metals. The hyperfine structures of these EPR-spectra for complexes **6** and **7** (Figs. 2 and 3) as well as for **5** correspond completely to theoretical ones calculated in accordance with natural abundance of magnetic isotopes ^{57}Fe (2.15%, $I = 1/2$), ^{95}Mo (15.9%, $I = 5/2$), ^{97}Mo (9.6%, $I = 5/2$), ^{183}W (14.3%, $I = 1/2$) [12].

We were unable to obtain crystals of complex **6** suitable for X-ray analysis, but it is possible to presume the structure of this product on base of EPR-spectrum. The later demonstrate the non-equivalence of *o*-semiquinolone protons in 4th and 5th positions and equivalence of transition metal substituents. Such situation can be realized in five-coordinated trigonal bipyramidal structure where $\text{CpM}(\text{CO})_3$ fragments lie in equatorial plane of coordination polyhedron, while triflate ligand occupies an axial position (Scheme 3). In this case the ax/eq-arrangement of the chelating *o*-semiquinolone ligand will initiate *trans*-influence in one linear axial TfO-Sn-O fragment. The *trans*-position for O1 is occupied by triflate ligand, while that one for O2 remains vacant. This type of *trans* effect appearing as non-equivalence of back bonded protons in EPR spectrum has been already described for *o*-semiquinolone complexes of platinum, palladium and nickel [3c,15].

3. Conclusions

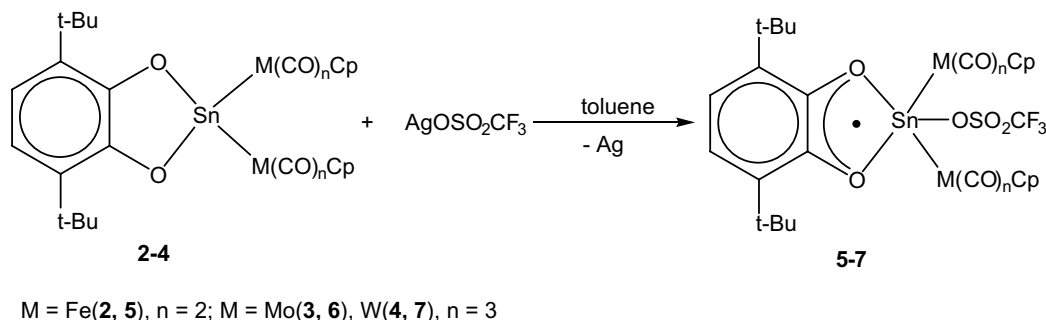
New complexes containing bonds Sn–M (M = Fe, Mo, W) and 3,6-di-*tert*-butylcatechololate ligand were synthesized by the direct insertion of the tin(II) catechololate into metal–metal bond of organometallic dimers. The oxidation of these new organobimetallic compounds by silver triflate give stable paramagnetic *o*-semiquinolone tin(IV) derivatives which keep metal–metal bonds.

4. Experimental

Infrared spectra of complexes in the 4000–400 cm^{-1} range were recorded on a Specord M-80 spectrophotometer in nujol. NMR spectrum of **1** was recorded in CDCl_3 solution using Bruker DPX-200 instrument with Me_4Si as internal standard.

EPR spectra were recorded on Bruker ER 200 D-SRC (working frequency ~ 9.5 GHz) spectrometer with ER041 MR microwave bridge, ER 4105 DR double resonator and ER 4111 VT variable temperature unite. The g_i values were determined using DFPH as the reference ($g_i = 2.0037$). HFC constants were obtained by simulation with the WinEPR SimFonia Software (Bruker).

All reagents were grade. Solvents were purified following standard methods [16]. All manipulations on complexes



Scheme 2.

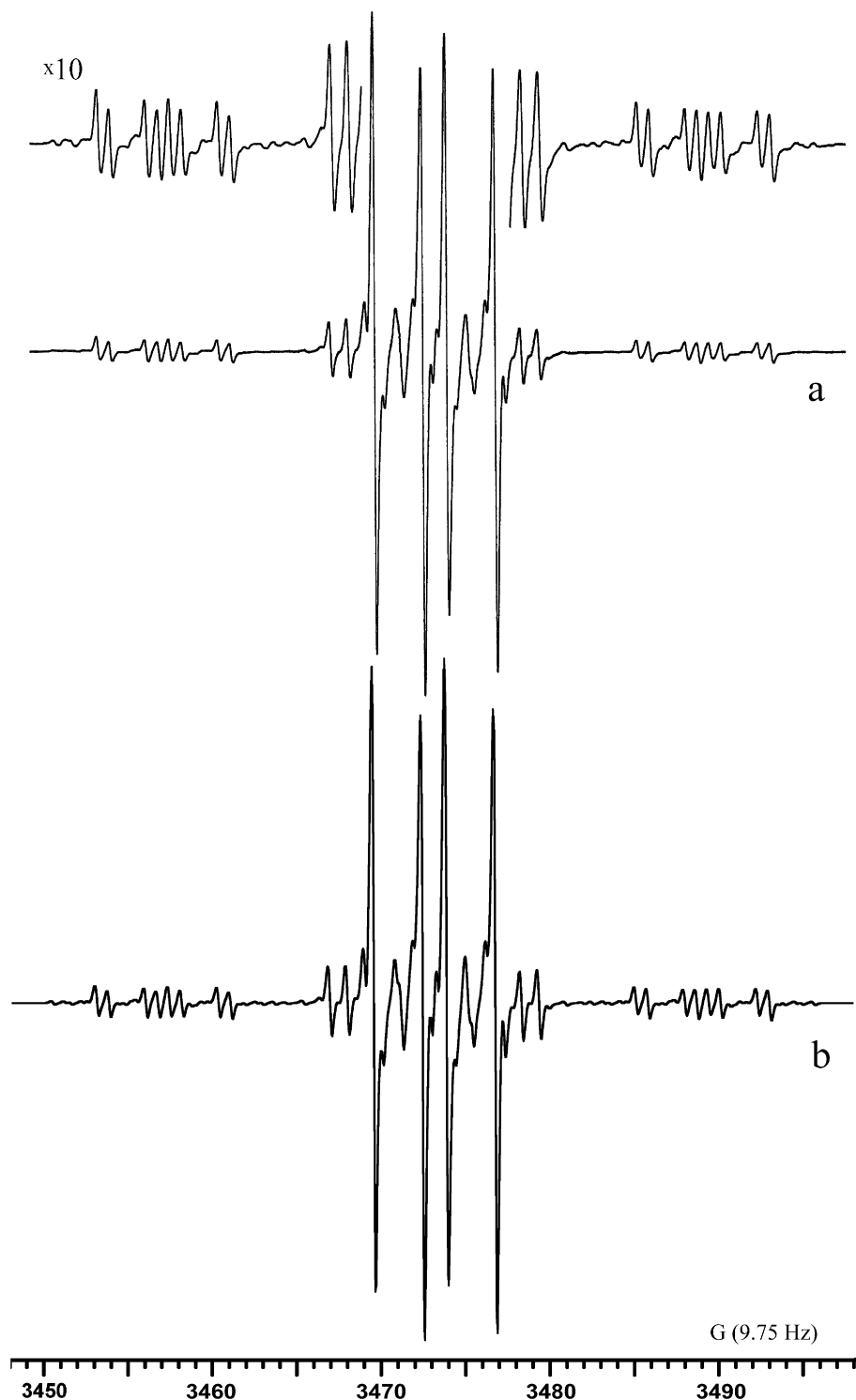


Fig. 2. Experimental X-band EPR spectrum of **6** in toluene at 290 K (a) and its simulation (b) (line width 0.25 G).

were carried out under conditions excluding air oxygen and moisture.

4.1. Synthesis of tris[3,6-di-*tert*-butylcatecholotin(II)] (**1**)

Complex **1** was synthesized as follows: the suspension of thallium catecholate in THF [17] (1.57 g, 2.5 mmol),

was added to THF solution containing $\text{SnCl}_2 \cdot \text{diox}$ [18] complex (0.695 g, 2.5 mmol). The solution was separated from thallium chloride precipitate by filtration. After THF evaporation, **1** was recrystallized from a hot toluene. Product obtained was identified by NMR, IR and elemental analysis as the known trimer of tin(II) 3,6-di-*tert*-butylcatecholate [6] (Yield: 0.71 g, 2.1 mmol).

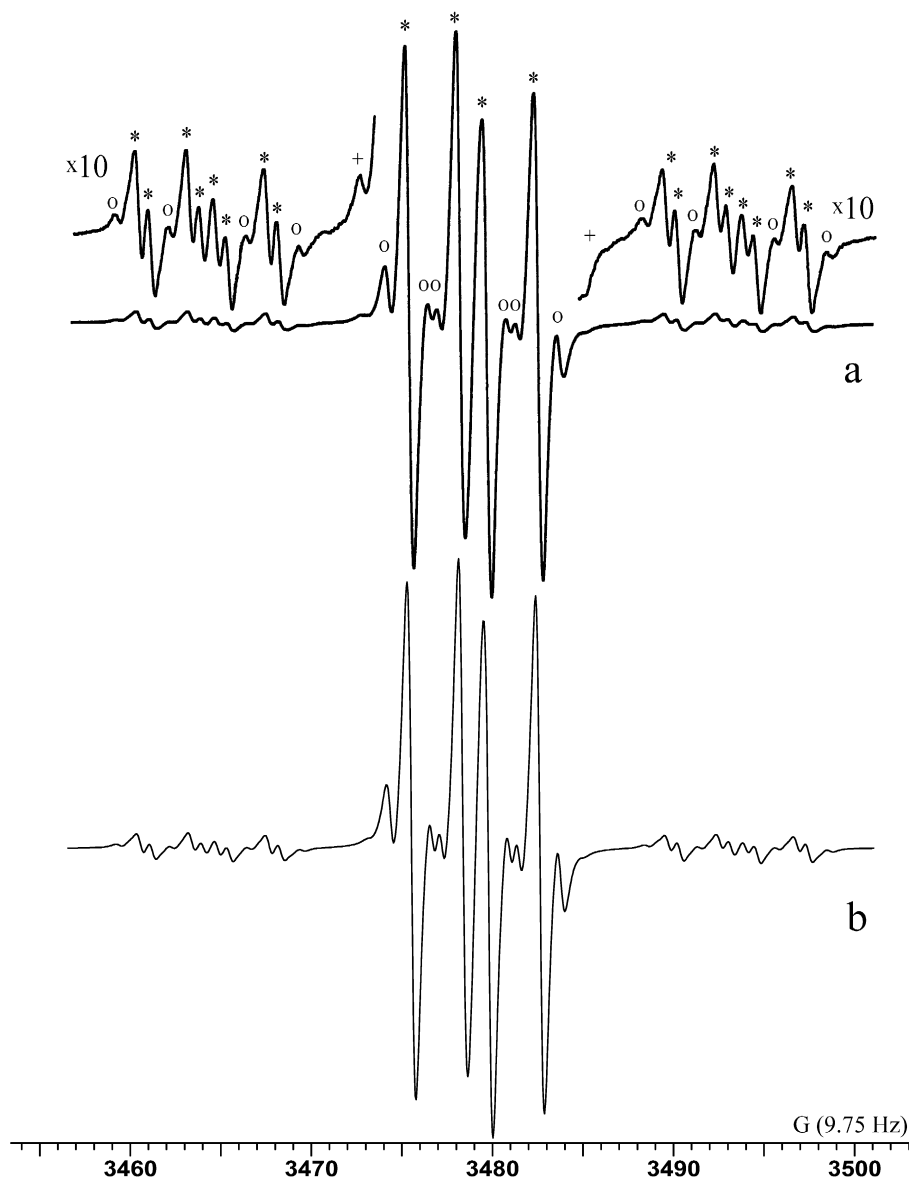


Fig. 3. Experimental X-band EPR spectrum of **7** in toluene at 290 K (a) and its simulation (b) (line width 0.5 G). * lines related to complex without magnetic isotopes of tungsten; o lines related to complex with one ^{183}W atom; + lines related to complex with two ^{183}W atoms.

Table 2
EPR parameters (A_i in G) for complexes **5–7**

Compound	$A_i(\text{H})$	$A_i(\text{H})$	$A_i(^{117}\text{Sn})$	$A_i(^{119}\text{Sn})$	$A_i(\text{M})$	g_i
5	2.85	4.15	37.15	38.85	2.70 (^{57}Fe)	2.0030
6	2.85	4.25	31.20	35.65	1.03 (^{95}Mo) 1.05 (^{97}Mo)	2.0021
7	2.85	4.25	28.50	29.80	2.30 (^{183}W)	1.9987

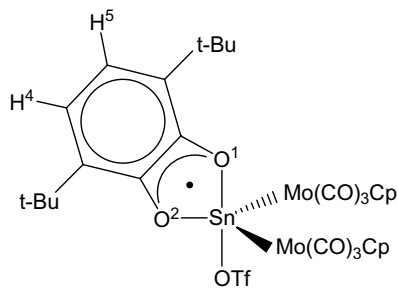
4.2. The reaction of tris[3,6-di-*tert*-butylcatecholato]tin(II) with organometallic dimers $[\text{CpM}(\text{CO})_n]_2$ ($M = \text{Fe}$, $n = 2$; $M = \text{Mo}$, W , $n = 3$)

The tin(II) catecholate (**1**) (0.847 g, 2.5 mmol) and 2.5 mmol of $[\text{CpM}(\text{CO})_n]_2$ (0.88 g, $M = \text{Fe}$; 1.23 g, $M = \text{Mo}$; 1.66 g, $M = \text{W}$) were stirred in 40 ml of toluene at 60 °C (4 h, $M = \text{Fe}$; 8 h, $M = \text{Mo}$; 24 h $M = \text{W}$). Result-

ing solution was evaporated and residue was recrystallized from a hot hexane ($M = \text{Fe}$) or CH_2Cl_2 /hexane mixture ($M = \text{Mo}$, W). Compounds **2–4** are stable on air in crystalline state for several days.

4.2.1. 3,6-Di-*tert*-butylcatecholato-bis(η^5 -cyclopentadienyl)-dicarbonyl-iron)-tin(IV) (**2**)

Cherry-red crystals which is soluble in the most of organic solvents (Yield: 1.61 g, 2.3 mmol, 93.1%). IR, ν : 2029, 2017, 1986, 1961, 1278, 1253, 1239, 1147, 971, 937, 846, 793, 722, 693, 653, 633, 577, 515. ^1H NMR (20 °C, CDCl_3): δ 1.47 (s, 18H, Bu^t); 4.90 (s, 10H, 2 Cp-groups); 6.57 (s, 2H, H_{arom}). ^{13}C NMR (20 °C, CDCl_3): δ 29.5 ($\text{C}(\text{CH}_3)_3$); 34.5 ($\text{C}(\text{CH}_3)_3$); 82.5 ($\eta^5\text{-C}_5\text{H}_5$); 113.2 (C_{arom} , $\text{C}'\text{Bu}$); 133.9 (C_{arom} , C-H); 153.5 (C_{arom} , C-O); 211.8 (CO). ^{119}Sn NMR (20 °C, CDCl_3): δ 579.9. Anal. Calc.



Scheme 3.

for $C_{28}H_{30}Fe_2O_6Sn$ (692.93): C, 48.53; H, 4.36; Fe, 16.12; Sn, 17.13. Found: C, 48.74; H, 4.39; Fe, 16.05; Sn, 16.97%.

4.2.2. 3,6-Di-tert-butylcatecholato-bis(η^5 -cyclopentadienyl)-tricarbonyl-molybdenum)-tin(IV) (**3**)

Red crystals which is soluble in the most of organic solvents except hexane (Yield: 1.81 g, 2.2 mmol, 87.4%). IR, ν : 2020, 1996, 1957, 1942, 1926, 1394, 1257, 1240, 1140, 971, 829, 805, 694, 651, 581, 569, 558, 503. 1H NMR (20 °C, $CDCl_3$): δ 1.46 (s, 18H, Bu^t); 5.43 (s, 10H, 2 Cp-groups); 6.58 (s, 2H, H_{arom}). ^{13}C NMR (20 °C, $CDCl_3$): 29.5 (C(CH₃)₃); 34.5 (C(CH₃)₃); 90.3 (η^5 -C₅H₅); 113.7 (C_{arom}, C^tBu); 134.1 (C_{arom}, C–H); 153.0 (C_{arom}, C–O); 223.6 (CO); 228.3 (CO). ^{119}Sn NMR (20 °C, $CDCl_3$): δ 616.1. Anal. Calc. for $C_{30}H_{30}O_8SnMo_2$ (829.11): C, 43.45; H, 3.62; Sn, 14.32; Mo, 23.14. Found: C, 43.50; H, 3.68; Sn, 14.30; Mo, 23.20%.

4.2.3. 3,6-Di-tert-butylcatecholato-bis(η^5 -cyclopentadienyl)-tricarbonyl-tungsten)-tin(IV) (**4**)

Orange-red crystals which is soluble in the most of organic solvents except hexane (Yield: 2.31 g, 2.3 mmol, 92.0%). IR, ν 2019, 1998, 1945, 1932, 1910, 1420, 1395, 1256, 1237, 1148, 971, 933, 917, 839, 801, 693, 651, 573, 550, 481, 454. 1H NMR (20 °C, $CDCl_3$): δ 1.45 (s, 18H, Bu^t); 5.51 (s, 10H, 2 Cp-groups); 6.57 (s, 2H, H_{arom}). ^{13}C NMR (20 °C, $CDCl_3$): δ 29.5 (C(CH₃)₃); 34.5 (C(CH₃)₃); 88.9 (η^5 -C₅H₅); 113.7 (C_{arom}, C^tBu); 134.1 (C_{arom}, C–H); 152.8 (C_{arom}, C–O); 212.9 (CO); 215.9 (CO). ^{119}Sn NMR (20 °C, $CDCl_3$): δ 331.5. Anal. Calc. for $C_{30}H_{30}O_8SnW_2$ (1004.39): C, 35.84; H, 2.99; Sn, 11.82; W, 36.61. Found: C, 35.90; H, 3.00; Sn, 11.75; W, 36.65%.

4.3. The synthesis of complex **6**

The toluene solution of silver(I) triflate (0.13 g, 0.5 mmol) was added droply to the solution of complex **3** (0.41 g, 0.5 mmol) in the same solvent. The solution turned dark violet immediately and the black deposit of silver metal was precipitated. Reaction mixture was filtered off on glass filter and solvent was evaporated. The residue was recrystallized from CH_2Cl_2 /hexane mixture. The complex **6** was obtained as thin violet needles (Yield: 0.42 g, 0.4 mmol, 86.3%). IR, ν 2042, 2032, 2013, 1948, 1927, 1915, 1435, 1411, 1366, 1308, 1295, 1233, 1211, 1173,

1065, 1018, 970, 951, 840, 676, 653, 635, 578, 552, 495. Anal. Calc. for $C_{31}H_{30}O_{11}SnSF_3Mo_2$ (977.76): C 38.05; H 3.07; Sn 12.14; S, 3.27; Mo, 19.63. Found: C 38.15; H 3.10; Sn 12.10; S, 3.25; Mo, 19.60%.

Complexes **5**, **7** were investigated by EPR spectroscopy in solutions and were not isolated in individual state.

4.4. X-ray crystallographic study of **3**

Crystals of **3** suitable for X-ray structure determination were obtained from CH_2Cl_2 /hexane solution. Crystal data: $C_{30}H_{30}Mo_2O_8Sn$, $M = 829.11$, monoclinic, $a = 10.1731(15)$, $b = 12.0016(18)$, $c = 23.936(4)$ Å, $\beta = 93.807(4)^\circ$, $V = 2916.0(8)$ Å³, space group $P2(1)/n$, $Z = 4$, $\mu = 1.747$ mm⁻¹, 15483 reflections measured, 5043 unique ($R_{int} = 0.0906$) which were used in all calculations. $R^1 [I > 2\sigma(I)] = 0.0545$, R_1 (all data) = 0.0915.

Intensity data for **3** was collected at 100 K on a Smart Apex diffractometer with graphite monochromated Mo $K\alpha$ radiation ($\lambda = 0.71073$ Å) in the φ - ω scan mode ($\omega = 0.3^\circ$, 10 s on each frame). The intensity data were integrated by SAINT program [19]. SADABS [20] was used to perform area-detector scaling and absorption corrections. The structures were solved by direct method and were refined on F^2 using all reflections with SHELXTL package [21]. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were placed in calculated positions and refined in the “riding-model” ($U_{iso}(H) = 1.5U_{eq}(C)$ in CH_3 -groups and $U_{iso}(H) = 1.2U_{eq}(C)$ in other ligands). Selected bond distances and angles for **3** are given in Table 1.

5. Supplementary material

CCDC 649213 contains the supplementary crystallographic data for **3**. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Acknowledgements

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