

Monoand trinuclear Sn(IV), Sn(II), Co(II), Ni(II), Cu(II) complexes with thioglycolate sodium salt or bis (sodium glycolate) disulfide

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ABSTRACT

The reaction between the tetrachloride with mercapto acetic acid sodium salt in NaOH or of the metal with bis (sodium glycolate) disulfide yield the following compounds of the formula $[\text{Sn}(\text{OOCCH}_2\text{SNa})_4]$, and $[\text{Sn}(\text{SCH}_2\text{COONa})_4]$ in high yield. Direct reactions of the above compounds with $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ or SnCl_4 produce a new trinuclear compounds of the formula $[\text{Sn}^{(\text{IV})}\text{Sn}_2^{(\text{II})}(\mu^2\text{-OOCCH}_2\text{S})_4]$, $[\text{Sn}^{(\text{IV})}\text{Sn}_2^{(\text{II})}(\mu^2\text{-SCH}_2\text{COO})_4]$, $[\text{Sn}_3(\mu^2\text{-OOCCH}_2\text{S})_4\text{Cl}_4]$ and $[\text{Sn}_3(\mu^2\text{-SCH}_2\text{COO})_4\text{Cl}_4]$, where as their reactions with $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$, $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ or $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ gave new trinuclear complex of general formula $[\text{Sn}(\mu^2\text{-OOCCH}_2\text{S})_4(\text{MCl}_2)_2]$ or $[\text{Sn}(\mu^2\text{-SCH}_2\text{COO})_4(\text{MCl}_2)_2]$ respectively. The prepared compound and complexes were characterized by micro elemental analysis (CHNS), metal content, IR, UV/Vis/ ^1H -NMR spectroscopy, conductivity and magnetic measurements. Electronic and magnetic measurements indicates that all tri compounds have tetrahedral geometry, while for Co(II), Ni(II) and Cu(II) complexes some of them contain a square planer and the other have tetrahedral geometry.

Keywords: tin complexes, thioglycolate sodium, disulfide complexes.

INTRODUCTION

The chemistry of difunctional ligands with transition or nontransition metals has been extensively studied⁽¹⁻²⁾ mainly due to versatile bonding features of the difunctional ligands. The reactions of diol or dithiol with group 4 metallocene derivatives gave complexes of twisted dimeric structure, or the opencyclic dimeric structure⁽³⁻⁴⁾.

Metal complexes of organosilicon (IV) and organotin (IV) halides with nitrogen, oxygen and sulfur donor ligands have received much more attention during the last few years⁽⁵⁻⁶⁾. The study of these complexes has been a fascinating area of current research interest to inorganic chemist all over the world. Silicon and tin complexes have been a variety of industrial⁽⁷⁾ and environmental⁽⁸⁾ applications.

The solid-state structures of trialkyltin(IV) carboxylates can be either monomeric⁽⁹⁾, oligo and polymeric⁽¹⁰⁾ or cyclo oligomeric⁽¹¹⁾, where by the oligo and polymeric structures are formed through intermolecular $\text{Sn-O-C=O} \rightarrow \text{Sn}$ bonds. Since these bonds are relatively weak, a discrete molecular structure can be expected in solution.

The novel dialkyl tin 2,3-bis(diphenyl phosphino) malic acid adducts $\{\text{R}_2\text{Sn}(\text{O-O'-dpmaa})\}$, $[\text{R} = \text{Me}, \text{Bu}, \text{dpmaa} = \text{bis}(\text{diphenyl phosphino}) \text{ maleic acid}]$ where synthesized from dpmaa and R_2SnCl_2 or Bu_2SnO . They were fully characterized by elemental analysis, IR- and multinuclear NMR- spectroscopies as well as x-ray crystallography⁽¹²⁾.

Reaction of Hg(II) salts with thioether carboxylic acids $o\text{-C}_6\text{H}_4\{\text{CH}(\text{SCH}_2\text{COOH})_2\}_2$ and $\text{phCH}(\text{SCH}_2\text{COOH})_2$ in water were found to lead to the decomposition of these ligands with the formation of mercury (II) mercaptoacetate $\text{Hg}(\text{SCH}_2\text{COOH})_2$ and the aldehydes $o\text{-C}_6\text{H}_4(\text{CHO})_2$ and phCHO respectively. The x-ray structure of $\text{Hg}(\text{SCH}_2\text{COOH})_2$ shows a linear of S-Hg-S bonds⁽¹³⁾.

A series of new five-coordinated ionic organotin (IV) complexes with general formula $[\text{Q}][\text{Me}_2\text{Sn}(\mu^2\text{-SCH}_2\text{COO})\text{Cl}]$, $\text{Q} = \text{diethylammonium, triethyl ammonium, tripropyl ammonium, tri-n-butylammonium, pyrimidinium, 3-picolinium, and dimethyl phenyl ammonium}$, where synthesized by the reaction of mercaptoacetic acid and dimethyltin dichloride in the presence of an organic base. These complexes have been characterized by elemental analyses, IR and ^1H -MMR spectroscopies. The crystal structure of $[(n\text{-Pr})_3\text{NH}][\text{Me}_2\text{Sn}(\mu^2\text{-SCH}_2\text{COO})\text{Cl}]$ was determined by x-ray crystallography. The structure consists of an anion part, and a tri-n-propyl ammonium cation part as a counter ion⁽¹⁴⁾.

The complexes of general formal $[\text{Sn}(\text{tch})_2\{\text{MCL}_2\}_2]$ were prepared from the precursor $\text{Sn}(\text{tch})_2$ and MCL_2 , where tch= thiocarbohydrazide, M= Mn(II), Fe(II), Co(II), Ni(II) and Cu(II). It was allowed to react with diethyl dithiocarbonate which yielded the trinuclear complexes of the type $[\text{Sn}(\text{tch})_2\{\text{M}_2(\text{dtc})_4\}]$. They were characterized on the basis of micro analytical, spectral (IR, UV-Vis, $^1\text{H-NMR}$) studies, conductivity measurement and magnetic moment data ⁽¹⁵⁾.

In view of these results and in continuation of our comprehensive studies on the Sn, Bi and in metal complexes with sulfur containing ligand ⁽¹⁶⁻¹⁹⁾ we have prepared some new tin(IV) compounds and their trinuclear complexes with Sn(II), Co(II), Ni(II) and Cu(II) complexes.

EXPERIMENTAL

All the chemical used were of high parity according to supplied (Fluka). Analysis of ligand and complexes were carried out using a CHNS elemental analyzer model 2400 Perkin Elmer. The metal content was estimated spectrophotometric using a shimadzu AA670 spectrophotometer. Melting point or decomposition temperature were determine on a melting point apparatus and were uncorrected, infrared spectra were recorded on a FIIR Bruckner Tensor 27C° spectrophotometer in the 250-4000 cm^{-1} range using CsI and KBr disc. Electronic spectra were obtained with a shimadzu UV/Vis. recording UV160 spectrophotometer at room temperature. The measurements were recorded using a concentration of 10^{-3}M of the complexes in DMSO. The $^1\text{H-NMR}$ spectra were obtained with Perkin Elmer (300 MHz) spectrometer, The ^1H chemical shifts were measured in DMSO- d_6 relative to tetramethyl silane as the internal reference. The magnetic moment measurement were carried out at 25°C on the solid state by Faraday's method using Bruker BM6 instrument. Conductivities were measured using conductivity meter model (PCM3-Jenway). These measurement were carried out using DMSO over the 10^{-3}M at 25°C.

Preparation of bis (sodium glycolate) disulfide (SCH_2COONa)₂:

A stirred solution of mercaptoacetic acid sodium salt (2.28g, 0.02mol) in (10 cm^3) distil water was treated drop wise with ethanolic solution of sodium hydroxide (0.80g, 0.02mol). Then ethanolic solution of iodine was added drop wise to the mixture with stirring for 1hr. After refluxing this mixture for 2hr, it was cooled to room temperature for 12hr to obtain a yellowish green solution, the product was precipitated by adding (40 cm^3) of methanol. It was filtered and washed several times with ethanol, chilled in diethylether and dried in vacuum for several hours.

Preparation of tin (IV) compound:

Two different methods were used, the classical method that include the substitution reaction and the other method is oxidative addition method:

1- preparation of $[\text{Sn}(\text{OOCH}_2\text{SNa})_4]$ (1) :

Treatment of disodium thioglycolate (prepared from the reaction of an equivalent amount of NaOH (1.60g, 0.04mol) and (4.56g, 0.04 mol) mercaptoacetic acid sodium salt in ethanol (30 cm^3) with tintetrachloride (2.60 g, 0.01 mol) in (10 cm^3) water. The mixture was refluxed for 4hr then the mixture was cooled for 24hr at room temperature to give a white solid, which was filtered off, washed with ethanol and diethylether then dried, under vacuum.

2- preparation of $[\text{Sn}(\text{SCH}_2\text{COONa})_4]$ (2):

Tin metal and bis(sodium glycolate) disulfide (SCH_2COONa)₂(4.52g, 0.02mol) were refluxed in toluene (30 cm^3) for 10hr, by which time the metal was completely dissolved and the colour of tin is charged. A dark yellow solid was precipitated, refluxing was stopped at this stage and the stirred mixture was cooled to ambient temperature, petroleum ether (60-80°C) (20 cm^3) was then added. The solid product was collected by filtering, washed twice with petroleum ether and dried in vacuum.

3-preparation of $[\text{Sn}^{(\text{IV})}\text{Sn}_2^{(\text{II})}(\mu^2\text{-OOCCH}_2\text{S})_4]$ (3):

This compound were prepared by reacting of methanolic solution of $[\text{Sn}(\text{OOCH}_2\text{SNa})_4]$ (1) (5.70g, 0.01mol) in (10 cm^3) and ethanolic solution (10 cm^3) of $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ (2.25g, 0.02mol). The reaction mixture was stirred under reflux for 3hr, after slow evaporation of the solvent the solid complex formed was filtered off and washed with ethanol followed by diethyl ether then dried under vacuum for several hours.

4-preparation of $[\text{Sn}^{(\text{IV})}\text{Sn}^{(\text{II})}(\mu^2\text{-SCH}_2\text{COO})_4]$ (4):

This compound was prepared by using previous procedure but, by using complex (2) instead of complex(1).

5-preparation of $[\text{Sn}_3(\mu^2\text{-OOCCH}_2\text{S})_4\text{Cl}_4]$ (5):

A clear solution of $[\text{Sn}(\text{OOCCH}_2\text{SNa})_4]$ (1) (0.57g, 0.001mol) in methanol (10cm^3) was added to a solution of SnCl_4 (0.26g, 0.002mol) in methanol (10cm^3). The reaction mixture was stirred under reflux for 3hr, the found precipitate was filtered off washed with methanol and diethylether then dried in vacuum.

6-preparation of $[\text{Sn}_3(\mu^2\text{-SCH}_2\text{COO})_4\text{Cl}_4]$ (6):

This compound was prepared by using the above procedure but, by using complex (2) instead of complex (1).

7- preparation of $[\text{SnM}_2(\mu^2\text{-OOCCH}_2\text{S})_4]$ (7-9), (M=Co,Ni,Cu):

A clear solution of $[\text{Sn}(\text{OOCCH}_2\text{SNa})_4]$ (1) (0.57g, 0.001mol) in methanol (10cm^3) was added to a solution of $\text{MCl}_2 \cdot 6\text{H}_2\text{O}$, M= Co, Ni, or $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ (0.002 mol) in distil water (10cm^3). The reaction mixture was stirred under reflux for 3hr, the formed colour precipitate was filtered off, washed with methanol and diethyl ether and dried in vacuum.

8-Preparation of $[\text{SnM}_2(\mu^2\text{-SCH}_2\text{COO})_4]$ (10-12), (M=Co,Ni,Cu):

These complexes were prepared using the above procedure but, by using complex (2) instead of complex (1).

RESULTS AND DISCUSSION

In the synthesis of the tin compounds (1) and (2), two general synthetic method have been used. The first method involves a direct reaction of tin tetrachloride with bis (sodium thioglycolate) ligand (prepared from equivalent amount of 2-merceptoacetic acid sodium salt with NaOH), this reaction is similar to those used for the preparation of $[\text{M}(\text{Sph})_n]$ compound of main elements involving the reaction of MCl_n with NaSph or LiSph⁽²⁰⁾, and the second method involves the oxidative addition reaction of tin metal with bis (sodium glycolate) disulfide ligand. The mechanism of this reaction involves the initial cleavage of the -S-S- bond of the ligands to form the thiolate ions and the oxidation of tin metal from Sn^0 to $\text{Sn}^{+4(16)}$.

Treatment of the tin compounds in alcoholic solution with aqueous solution of SnCl_2 or SnCl_4 or metal chloride in (1:2) molar ratio gave the trinuclear complexes of the types $[\text{SnSn}_2(\text{L})_4]$, $[\text{Sn}_3(\text{L})_4]$ and $[\text{Sn}(\text{L})_4(\text{MCl}_2)_2]$ when $\text{L} = (\text{OOCCH}_2\text{S})_4$ or $(\text{SCH}_2\text{COO})_4$ ligand. The coordination of oxygen or sulfur atom in tin compounds and in the prepared complexes, the carboxyl groups and the thiolate ions are also responsible for the formation of new complexes. The physical properties of the compounds and the complexes are listed in Table (1).

The compound and complexes are quite stable in dry air and melt or decompose over 200°C . The compounds are soluble in alcoholic solvent while the complexes are insoluble in most organic solvent but soluble in dimethyl formamide (DMF) or dimethyl sulfoxide (DMSO).

The most important IR assignment of tin compounds and their complexes are listed in Table (2). The most significant in formations on the geometry of these complexes were came from the analysis of carboxylate and thiolate absorption region. Stretching frequencies of these functional groups are closely related of the way in which they are coordinated to the metal atom⁽²¹⁾. The IR spectra of the tin compounds and complexes showed broad and intense bands ranging between ($1583\text{-}1630\text{ cm}^{-1}$) and ($1362\text{-}1430\text{ cm}^{-1}$) assigned for asym. $\nu(\text{COO})$ and for sym. $\nu(\text{COO})$ respectively (Table2), the magnitude of $\Delta\nu$ ($\Delta\nu = \nu_{\text{asym,COO}} - \nu_{\text{sym,COO}}$) were in the range ($150\text{-}180\text{cm}^{-1}$) suggested monodentate bounding of carboxylic group to metal ion⁽²²⁾ and a bidentate attachment when $\Delta\nu$ was between ($190\text{-}295\text{cm}^{-1}$)⁽²³⁾. In some cases, when $\Delta\nu > 200\text{cm}^{-1}$ it means that the only way in which all carboxylate groups are bound to the metal ion is though bridging two metal ions⁽²⁴⁾.

Further support of this argument came from the IR of complexes which showed new bands at ($462\text{-}490\text{ cm}^{-1}$) attributable to $\nu(\text{M-O})$. They also showed a band in the region ($290\text{-}320\text{cm}^{-1}$) which may be due to $\nu(\text{M-Cl})$ vibration frequency⁽²⁵⁾.

The $\nu(\text{C-S})$ band of the ligand was observed at (956cm^{-1}) upon coordination with metal ion, it was shifted to lower frequency values (Table2). Further support for this coordination has been provided by the appearance of new bands in the ($340\text{-}380\text{cm}^{-1}$) ranges which are tentatively attributed to $\nu(\text{M-S})$ ⁽²⁶⁾.

The electronic spectra of the compounds and their complexes were recorded as (10^{-3}M) solution in DMSO and the results were presented in Table (2).

The bands observed at 39700cm^{-1} and 33222cm^{-1} (Table 2) due to disulfide ligand which may be assigned as $n\text{-}\pi^*$ or $\pi\text{-}\pi^*$ transition respectively. The UV spectral bands of the ligand were observed at higher region upon formation of the

tin compounds which observed at (28751-3450cm⁻¹). This can be attributed to charge transfer from filled ligand orbitals to the vacant tin orbitals⁽²⁷⁾.

The magnetic moment values of Co(II) complexes (**7,10**) are (2.30 and 4.41 B.M) these values correspond to low spin square planar geometry for complex (**7**) and tetrahedral environment for complex (**10**)⁽²⁸⁾. The electronic spectra of Co(II) complex (**7**) show a band at (15790cm⁻¹) which may be assigned to ²A_{1g}→²E_g transition in square planar geometry and a band at (29520cm⁻¹) which may be assigned as charge transfer. The Co(II) complex (**10**) show the presence of two bands in the region 12658cm⁻¹ and 18182cm⁻¹ corresponding to ⁴A₂(F)→⁴T₂F and ⁴A₂(F)→⁴T₁F transition characteristic of tetrahedral Co(II) ion⁽²⁹⁾.

The magnetic moments of Ni(II) complexes (**8,11**) are (0.0 and 3.38 B.M) which suggest a square planar and tetrahedral geometry of these complexes respectively.

The electric spectra of Ni(II) complex(**8**) showed two bands at (12500cm⁻¹) and (25000cm⁻¹), these bands were assigned to ¹A_{1g}→¹B_{2g} and ¹A_{1g}→¹E_g transition. These results suggested square planar geometry around the nickel ion⁽³⁰⁾. The Ni(II) complex(**11**) show the presence of one band at (15460cm⁻¹) correspond to ³T₁(F)→³T₁(P) transition, this show that the geometry of the complex is tetrahedral⁽³¹⁾.

The magnetic moment of Cu(II) complexes (**9,12**) has been found (1.70 and 1.98 B.M) which indicate the presence of one unpaired electron. The electronic spectra of the complexes showed a band at (13300cm⁻¹) and (12150cm⁻¹) which are assigned to ²T₂→²E transition in tetrahedral environment⁽³²⁾.

The ¹H-NMR specter of the compound and complexes (Table 2) are mainly composed of one part the methylene protons in SCH₂ moiety. The chemical shifts of the protons in SCH₂ exhibit signals at (3.41-3.82ppm) as triplet which is cancel by long distance Sn-H coupling⁽³³⁾.

The molar conductivities of (10⁻³M) solution of the compound and complexes (Table 1) indicate that they are non-electrolytes in DMSO⁽³⁴⁾.

On the bases of the fore going studies, the ligand used in this study, coordinate to the metal ions in mono, bi or tridentate fashions from the oxygen or sulfur or both of them form mono and trinuclear complexes as shown in Fig 1.

Table (1): Analytical data and physical properties of the prepared complexes

No.	Chemical formula	Color	m. p. °C	Yield %	Λ (10 ⁻³ M) Ohm ⁻¹ .mol ⁻¹ .cm ²	μ _{eff}	Elemental micro analysis found / (Calc.)				
							C%	H%	S%	Sn%	M%
1	[Sn(OOCCH ₂ SNa) ₄]	White	>360	65	12	---	16.80 (16.82)	1.36 (1.40)	22.40 (22.43)	22.40 (22.43)	---
2	[Sn(SCH ₂ COONa) ₄]	Dark yellow	288	90	10	---	16.79 (16.81)	1035 (1.40)	22.39 (22.43)	22.39 (22.43)	---
3	[Sn ^(IV) Sn ₂ ^(III) (μ ² -OOCCH ₂ S) ₄]	Pale yellow	286	74	28	---	13.39 (13.41)	1.10 (1.12)	17.88 (17.87)	17.83 (17.87)	---
4	[Sn ^(IV) Sn ^(III) (μ ² -SCH ₂ COO) ₄]	Pale orange	>360	95	20	---	13.40 (13.41)	1.10 (1.12)	17.85 (17.87)	17.85 (17.87)	---
5	[Sn ₃ (μ ² -OOCCH ₂ S) ₄ Cl ₄]	White	290	85	12	---	11.16 (11.19)	0.89 (0.93)	14.89 (14.92)	14.89 (14.92)	---
6	[Sn ₃ (μ ² -SCH ₂ COO) ₄ Cl ₄]	Pale yellow	>360	92	18	---	11.17 (11.19)	0.90 (0.93)	14.90 (14.92)	14.90 (14.92)	---

7	[SnCo ₂ (μ ² -OOCCH ₂ S) ₄]	Dark brown	350	80	32	2.30	16.05 (16.04)	1.32 (1.34)	21.44 (21.46)	21.44 (21.46)	19.73 (19.76)
8	[SnNi ₂ (μ ² -OOCCH ₂ S) ₄]	Dark green	340	75	16	Dia	16.10 (16.10)	1.31 (1.34)	21.44 (21.47)	21.44 (21.47)	19.66 (19.69)
9	[SnCu ₂ (μ ² -OOCCH ₂ S) ₄]	Green	295	83	18	1.70	15.81 (15.85)	1.30 (1.32)	21.10 (21.13)	21.10 (21.13)	20.96 (20.98)
10	[SnCo ₂ (μ ² -SCH ₂ COO) ₄]	Brown	320	85	20	4.41	16.05 (16.09)	1.30 (1.34)	21.42 (21.46)	21.42 (21.46)	19.74 (19.76)
11	[SnNi ₂ (μ ² -SCH ₂ COO) ₄]	Greenish yellow	330	80	12	3.38	16.07 (16.10)	1.31 (1.34)	21.43 (21.47)	21.43 (21.47)	19.65 (19.69)
12	[SnCu ₂ (μ ² -SCH ₂ COO) ₄]	Light brown	360	75	10	1.98	15.81 (15.85)	1.36 (1.32)	21.10 (21.13)	21.10 (21.13)	20.96 (20.98)

Table (2): Electronic, infrared and ¹H-NMR spectral data of prepared complexes

No.	Band maxima (cm ⁻¹) λ _{max}	IR spectral band (cm ⁻¹)							¹ H-NMR δppm
		ν _{as} (COO)	ν _s (COO)	Δν(ν _{as} -ν _s)	ν(C-S)	ν(M-S)	ν(M-O)	ν(M-Cl)	
1	32150	1598s	1440s	258	---	340 _m	400 _m	---	3.415
2	228757	1578s	1428s	150	940s	342 _w	---	---	3.505
3	29000	1616s	1409 _m	216	---	345 _m	480 _m	---	3.165
4	32000	1622s	1407 _m	210	930s	340 _m	460 _m	---	3.650
5	30000	1600s	1440s	160	---	360 _m	480 _m	300 _m	3.810
6	29050,32000	1590s	1440s	150	920s	350 _m	470 _m	320 _m	3.810
7	15790,29520	1616s	1409s	205	930s	380 _m	470 _m	---	3.750
8	12500,125000	1622s	1407s	215	936s	360 _m	490 _m	---	3.710
9	13300	1617s	1407s	210	931s	375 _m	485 _m	---	3.800
10	12658,18182	1617s	1384s	233	932s	360 _m	480 _m	---	3.600
11	15460	1616s	1384s	232	935s	375 _m	490	---	3.600
12	12150	1618s	1384 _m	239	940s	370 _m	480	---	3.700

s= strong, m =medium, w =weak

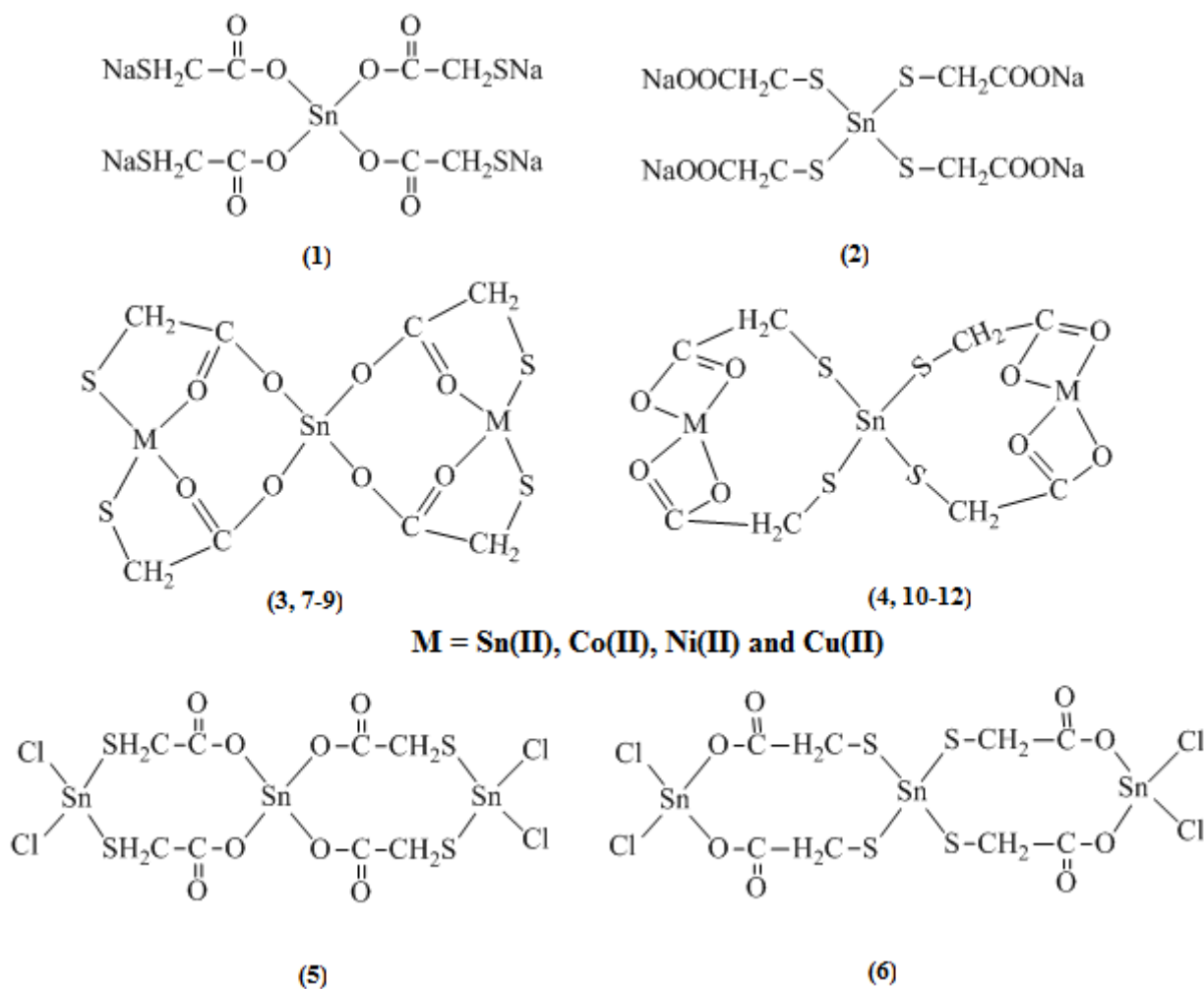


Figure (1): Suggested structure of the prepared complexes

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