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**Synthesis and characterization of some divalent transition metal complexes with tellurium containing 10-membered tetraazamacrocyclic ligands**

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**ABSTRACT**

A new series of 10-membered tellurium containing tetraazamacrocyclic complexes,  $[ML^1Cl_2]$ ,  $[ML^2Cl_2]$  and  $[ML^3Cl_2]$ , where  $[M = Mn(II), Co(II), Cu(II)]$ ;  $L^1, L^2$  and  $L^3 = 10$ -membered tellurium containing tetraazamacrocyclic ligands] have been prepared via the template condensation of 1,2-diaminoethane and diaryltellurium dichlorides,  $R_2TeCl_2$ , ( $R = p$ -hydroxyphenyl, 3-methyl-4-hydroxyphenyl,  $p$ -methoxyphenyl) in the presence of metal chlorides. These complexes have been characterized by elemental analyses, conductivity, magnetic susceptibility measurements, infrared, electronic absorption and proton magnetic resonance spectra. An octahedral geometry has been assigned to all the metal complexes.

**Keywords:** tellurium containing, tetraazamacrocycles, diaryltellurium dichlorides, 1,2-diaminoethane, template condensation, metal complexes.

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**INTRODUCTION**

The coordination chemistry of organotellurium ligands containing *hard* donor atoms such as N and O along with *soft* tellurium is interesting as such ligand framework can provide 'insight' into competitive coordination behaviour between the *hard* and *soft* donors towards a metal center [1, 2]. Such molecular systems may be important in transition metal catalyzed asymmetric synthesis [3,4] and as single source precursors in MOCVD processes [5-7]. Also, macrocycle complexes are significant as they have been found to act as possible models for biochemically important proteins and enzymes [8-12].

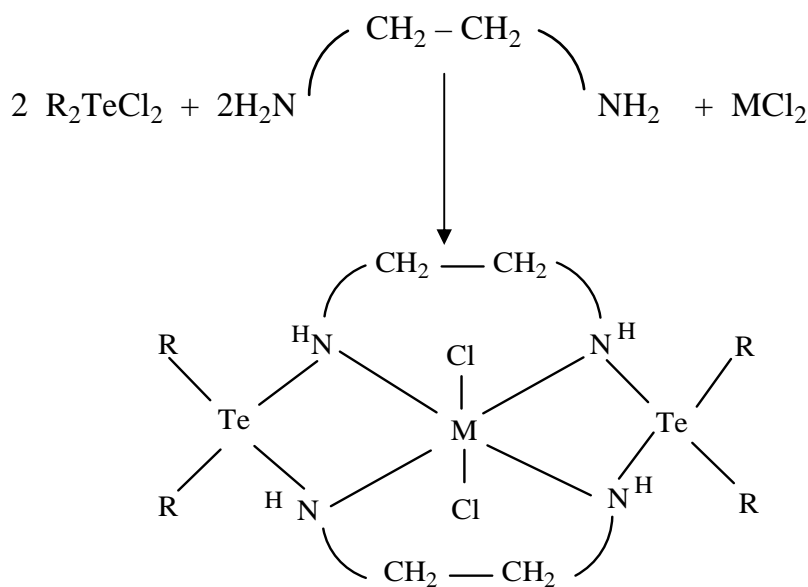
The development of tellurium containing macrocycles is evidenced in some recent publications [13-17]. Srivastava *et al* [18] reported the route of synthesis of metal complexes with tellurium containing macrocycles. Herein, we report the synthesis and characterization of divalent manganese, cobalt and copper complexes with three novel tellurium tetraazamacrocycles, ( $\text{Te}_2\text{N}_4$  system).

## EXPERIMENTAL SECTION

### Materials and Methods

The chemicals, tellurium tetrachloride (Aldrich), 1,2-diamionethane and  $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$  (SISCO Research Laboratories), phenol, *o*-cresol, anisole,  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ ,  $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$  (Loba Chemie) were of reagent grade. All the solvents were dried before use.

Bis(*p*-hydroxyphenyl)-, bis(3-methyl-4-hydroxyphenyl) and bis(*p*-methoxyphenyl) tellurium dichlorides were prepared by direct reactions of  $\text{TeCl}_4$  with phenol [19], *o*-cresol [20] and anisole [21] respectively. All the preparation were carried in dry conditions under the atmosphere of nitrogen.



where

- $L^1$  ;  $R = p\text{-hydroxyphenyl}$   
 $L^2$  ;  $R = 3\text{-methyl-4-hydroxyphenyl}$   
 $L^3$  ;  $R = p\text{-methoxyphenyl}$   
 $M = \text{Mn}^{II}, \text{Co}^{II} \text{ and } \text{Cu}^{II}$

Figure- 1

### ***Preparation of Complexes***

The 10-membered tellurium tetraazamacrocyclic transition metal complexes have been synthesized by the template condensation reaction of metal chlorides with diaryltellurium dichlorides and 1,2-diaminoethane in 1 : 2 : 2 molar ratio, as per fig. 1. A general method for the synthesis of these 1,6-diaryltellura – 2, 5, 7, 10-tetraazacyclodecane metal complexes is given below:

A saturated solution of 4.0 mmol of diaryltellurium dichloride in methanol was added slowly and with constant stirring to a methanolic (~ 5mL) solution of 4.0 mmol of ethylenediamine taken in a R.B. Flash. A distinct change in colour with slight turbidity was observed. The contents were refluxed for 2-3 h, followed by addition of 2.0 mmol solution of metal dichloride in about 10 mL methanol. An immediate change in colour was observed. The solution was the refluxed for 3-4 h and concentrated to about one third of original volume and cooled. This resulted in the separation of a coloured solid. Solid product was also obtained in some cases without concentration. This was filtered, washed with benzene/petroleum ether and dried in a vacuum desiccator over P<sub>4</sub>O<sub>10</sub>.

### ***Physical Measurements***

Elemental analysis for C, H, N were obtained from SAIF, Panjab University, Chandigarh on a CARLO ERBA Model 1106. Tellurium and chlorine contents were determined volumetrically [22]. Metals were estimated on Atomic Absorption Spectrophotometer (ECIL Model No. 4129) or gravimetrically [22]. The electrical conductivity in acetonitrile was obtained on a Systronic Type 305 conductivity bridge at 25 ± 2° C. The electronic spectra were recorded in dimethylsulphoxide at 22 ± 2°C on a Thermo Spectronic UV-1 spectrophotometer. The magnetic susceptibility data were obtained from National Physical Laboratory, New Delhi on a Gouy's balance (Model Johnson Matthey Alfa Products) using glycerin as a calibrant. The IR spectra (4000-400 cm<sup>-1</sup>) were recorded as KBr pellets on a Perkin Elmer Model 2000 FTIR Spectrometer at SAIF, Panjab University, Chandigarh. <sup>1</sup>H NMR spectra were recorded in DMSO – d<sub>6</sub> on BRUKER XWIN – NMR AVANCE 300 operating at 300.13 MHz, using tetramethylsilane as an internal reference. The NMR spectra were obtained from Kurukshetra University, Kurukshetra.

## **RESULTS AND DISCUSSION**

The molecular Formulae of the complexes [ML<sup>1</sup>Cl<sub>2</sub>], [ML<sup>2</sup>Cl<sub>2</sub>] or [ML<sup>3</sup>Cl<sub>3</sub>] have been assigned on the basis of the results of their elemental analyses (Table 1). These complexes are coloured, crystalline, air stable solids (except CuL<sup>1</sup>Cl<sub>2</sub>, which is hygroscopic) and are soluble only in polar donor organic solvents. Identification of metal complexes was made on the basis of their physico-chemical investigations. The formation of proposed macrocyclic skeleton is shown in figure 1.

The molar conductances of soluble complexes in acetonitrile at *ca* 10<sup>-3</sup> M show their non-electrolyte to weak-electrolyte nature [23] and hence suggest their formulation as [MLCl<sub>2</sub>].

### ***Magnetic and Electronic Spectral Studies***

The Mn<sup>II</sup> complexes show magnetic moments (Table 2) corresponding to five unpaired electrons (5.75 – 5.92 B.M.) at room temperature, close to the spin-only value of 5.92 B.M. Electronic

spectra of these complexes display two weak absorption bands as shown in Table 2, which are characteristic of octahedral geometry. [18, 24, 25].

**Table 1. Analytical data, physical properties, yields and molar conductance for metal complexes**

Complex	Empirical Formula (Formula Weight)	Colour	M.P °C (dec.)	Yield	Analysis found (calculated) %						$\Lambda_M$ at ca. $10^{-3}$ $M \text{ ohm}^{-1} \text{ cm}^2$ $\text{mol}^{-1}$
					C	H	N	Cl	Te	M	
[MnL <sup>1</sup> Cl <sub>2</sub> ]	C <sub>28</sub> H <sub>32</sub> Cl <sub>2</sub> N <sub>4</sub> O <sub>4</sub> Te <sub>2</sub> Mn (869.6)	Orange Pink	168- 170	69	38.20 (38.67)	3.23 (3.71)	6.15 (6.44)	7.90 (8.15)	29.01 (29.35)	6.08 (6.32)	*
[MnL <sup>2</sup> Cl <sub>2</sub> ]	C <sub>32</sub> H <sub>40</sub> Cl <sub>2</sub> N <sub>4</sub> O <sub>4</sub> Te <sub>2</sub> Mn (925.7)	Brown	190- 192	82	41.07 (41.52)	4.11 (4.36)	5.68 (6.05)	7.73 (7.66)	27.30 (27.57)	5.82 (5.93)	16.5
[MnL <sup>3</sup> Cl <sub>2</sub> ]	C <sub>32</sub> H <sub>40</sub> Cl <sub>2</sub> N <sub>4</sub> O <sub>4</sub> Te <sub>2</sub> Mn (925.7)	Brown	145- 150	77	41.00 (41.52)	4.01 (4.36)	5.57 (6.05)	7.59 (7.66)	27.42 (27.57)	5.64 (5.93)	28.5
[CoL <sup>1</sup> Cl <sub>2</sub> ]	C <sub>28</sub> H <sub>32</sub> Cl <sub>2</sub> N <sub>4</sub> O <sub>4</sub> Te <sub>2</sub> Co (873.6)	Grey	190- 194	78	38.13 (38.50)	3.14 (3.69)	6.04 (6.41)	7.86 (8.12)	29.21 (29.33)	6.42 (6.75)	*
[CoL <sup>2</sup> Cl <sub>2</sub> ]	C <sub>32</sub> H <sub>40</sub> Cl <sub>2</sub> N <sub>4</sub> O <sub>4</sub> Te <sub>2</sub> Co (929.7)	Pink Brown	158- 160	90	41.03 (41.34)	3.99 (4.34)	5.66 (6.03)	7.75 (7.63)	26.71 (27.45)	6.16 (6.34)	51.0
[CoL <sup>3</sup> Cl <sub>2</sub> ]	C <sub>32</sub> H <sub>40</sub> Cl <sub>2</sub> N <sub>4</sub> O <sub>4</sub> Te <sub>2</sub> Co (929.7)	Dark Green	110- 115	60	41.14 (41.34)	4.12 (4.34)	5.86 (6.03)	7.26 (7.63)	27.27 (27.45)	6.20 (6.34)	94.6
[CuL <sup>1</sup> Cl <sub>2</sub> ]	C <sub>28</sub> H <sub>32</sub> Cl <sub>2</sub> N <sub>4</sub> O <sub>4</sub> Te <sub>2</sub> Cu (878.2)	Dark Red	Hygro- scopic	94	37.95 (38.29)	3.26 (3.67)	6.09 (6.38)	7.82 (8.07)	28.81 (29.06)	7.06 (7.24)	*
[CuL <sup>2</sup> Cl <sub>2</sub> ]	C <sub>32</sub> H <sub>40</sub> Cl <sub>2</sub> N <sub>4</sub> O <sub>4</sub> Te <sub>2</sub> Cu (934.3)	Light Brown	192- 197	75	40.83 (41.14)	4.01 (4.32)	5.55 (6.00)	7.44 (7.59)	26.40 (27.31)	6.25 (6.80)	16.5
[CuL <sup>3</sup> Cl <sub>2</sub> ]	C <sub>32</sub> H <sub>40</sub> Cl <sub>2</sub> N <sub>4</sub> O <sub>4</sub> Te <sub>2</sub> Cu (934.3)	Light Green	120- 122	70	40.93 (41.14)	4.19 (4.32)	5.87 (6.00)	7.47 (7.59)	27.10 (27.31)	6.70 (6.80)	18.2

\* Not soluble in acetonitrile.

The magnetic moment values of cobalt (II) complexes lie in the range 5.01-5.17 B.M. corresponding to three unpaired electrons. The electronic absorption spectra of all the Co(II) complexes exhibit absorption in the region 9190-9710  $\text{cm}^{-1}$ , 14684-16727  $\text{cm}^{-1}$  and around 32000  $\text{cm}^{-1}$ , which may tentatively be assigned as shown in Table 2. These transitions suggest an octahedral environment [24, 27] around the cobalt ion.

The magnetic moments of Copper (II) complexes at room temperature lie in the range 1.77 – 1.90 B.M. corresponding to one unpaired electron. Electronic spectra of these complexes display bands in the range 10172 – 10822  $\text{cm}^{-1}$  ( ${}^2B_{1g} \rightarrow {}^2A_{1g}$ ), 17352 – 28011 ( ${}^2B_{1g} \rightarrow {}^2B_{2g}$ ) and 30250 – 32590 (CT). *Jahn Teller distortion* expected in  $d^9$  configuration actually disturbs the degeneracy, thus causing a split in energy level due to tetragonal distortion in geometry. This electronic spectral pattern of Copper (II) complexes under study suggests [18, 24, 28, 29] a distorted octahedral or tetragonal geometry.

### IR Spectra

The important IR bands and their assignments are tabulated in Table 3. The spectra are quite complex and an attempt has thus been made to draw the conclusions by comparing the spectra of metal complexes with those of corresponding constituent diarytellurium dichlorides and 1, 2-diaminoethane.

Table 2. Magnetic moment and electronic spectral data for metal complexes

Complex	Magnetic Moment (B.M.)	Electronic Absorption, cm <sup>-1</sup> Assignment		
[MnL <sup>1</sup> Cl <sub>2</sub> ]	5.75	28260 <sup>6</sup> A <sub>1g</sub> → <sup>4</sup> T <sub>2g</sub> (D)	32151 <sup>6</sup> A <sub>1g</sub> → <sup>4</sup> T <sub>1g</sub> (P) & (CT)	
[MnL <sup>2</sup> Cl <sub>2</sub> ]	5.92	18650 <sup>6</sup> A <sub>1g</sub> → <sup>4</sup> T <sub>1g</sub> (G)	32479 <sup>6</sup> A <sub>1g</sub> → <sup>4</sup> T <sub>1g</sub> (P) & (CT)	
[MnL <sup>3</sup> Cl <sub>2</sub> ]	5.82	25160 <sup>6</sup> A <sub>1g</sub> → <sup>4</sup> A <sub>1g</sub> (G)	35300 <sup>6</sup> A <sub>1g</sub> → <sup>4</sup> A <sub>2g</sub> (F) & (CT)	
[CoL <sup>1</sup> Cl <sub>2</sub> ]	5.01	9199 <sup>4</sup> T <sub>1g</sub> → <sup>4</sup> T <sub>2g</sub> (F)	16727 <sup>4</sup> T <sub>1g</sub> → <sup>4</sup> A <sub>2g</sub> (F)	32786 <sup>4</sup> T <sub>1g</sub> (F) → <sup>4</sup> T <sub>1g</sub> (P) & (CT)
[CoL <sup>2</sup> Cl <sub>2</sub> ]	5.17	9710 <sup>4</sup> T <sub>1g</sub> → <sup>4</sup> T <sub>2g</sub> (F)	14684 <sup>4</sup> T <sub>1g</sub> → <sup>4</sup> A <sub>2g</sub> (F)	32573 <sup>4</sup> T <sub>1g</sub> (F) → <sup>4</sup> T <sub>1g</sub> (P) & (CT)
[CoL <sup>3</sup> Cl <sub>2</sub> ]	5.15	9398 <sup>4</sup> T <sub>1g</sub> → <sup>4</sup> T <sub>2g</sub> (F)	14771 <sup>4</sup> T <sub>1g</sub> → <sup>4</sup> A <sub>2g</sub> (F)	32362 <sup>4</sup> T <sub>1g</sub> (F) → <sup>4</sup> T <sub>1g</sub> (P) & (CT)
[CuL <sup>1</sup> Cl <sub>2</sub> ]	1.77	10172 <sup>2</sup> B <sub>1g</sub> → <sup>2</sup> A <sub>1g</sub>	28011 <sup>2</sup> B <sub>1g</sub> → <sup>2</sup> B <sub>2g</sub>	30250 [CT]
[CuL <sup>2</sup> Cl <sub>2</sub> ]	1.85	10515 <sup>2</sup> B <sub>1g</sub> → <sup>2</sup> A <sub>1g</sub>	17352 <sup>2</sup> B <sub>1g</sub> → <sup>2</sup> B <sub>2g</sub>	32590 [CT]
[CuL <sup>3</sup> Cl <sub>2</sub> ]	1.90	10822 <sup>2</sup> B <sub>1g</sub> → <sup>2</sup> A <sub>1g</sub>	17561 <sup>2</sup> B <sub>1g</sub> → <sup>2</sup> B <sub>2g</sub>	30847 [CT]

Table 3. IR data (cm<sup>-1</sup>) for metal complexes

Complex	V <sub>N-H</sub>	N-H def.	N-H out of plane bending	V <sub>C-N</sub>
[MnL <sup>1</sup> Cl <sub>2</sub> ]	3220 m	1633 m	829 s	1171 s
[MnL <sup>2</sup> Cl <sub>2</sub> ]	3190 m	1625 m	813 s	1176 m
[MnL <sup>3</sup> Cl <sub>2</sub> ]	3260 m*	1652 m	822 s	1176 s
[CoL <sup>1</sup> Cl <sub>2</sub> ]	3195 m	1626 m	826 s	1173 s
[CoL <sup>2</sup> Cl <sub>2</sub> ]	3214 m	1627 s	812 s	1176 m
[CoL <sup>3</sup> Cl <sub>2</sub> ]	3250 m*	1649 m	823 s	1177 vs
[CuL <sup>1</sup> Cl <sub>2</sub> ]	3244 m	1636 vs	829 m	1173 m
[CuL <sup>2</sup> Cl <sub>2</sub> ]	3180 m	1627 m	812 s	1176 m
[CuL <sup>3</sup> Cl <sub>2</sub> ]	3250 m*	1651 m	822 s	1176 s

\*mixed with moisture band, s = strong, m = medium, vs = very strong.

The metal complexes under study did not show bands characteristic of free NH<sub>2</sub> group, instead the entire complexes exhibit a single sharp absorption band at around 3190-3260 cm<sup>-1</sup> (sometimes mixed with O-H) attributed to V<sub>N-H</sub> stretching vibration. The assignment of this sharp band is based on the fact that macrocyclic ligands which have coordinated secondary amino group, have bands in the vicinity of 3200 cm<sup>-1</sup> [18,30-32]. This contention finds support [30] from appearance of bands of medium to strong intensity at ~1640 cm<sup>-1</sup> and ~825 cm<sup>-1</sup> assigned as N-H deformation coupled with N-H out of the plane bending vibrations. Bands at

$\sim 1175\text{ cm}^{-1}$  may be reasonably assigned to C-N stretching vibration [18, 33, 34]. The above observation strongly suggest [18,30,33,34] that proposed macrocyclic framework is formed. The formation of tellurium containing macrocyclic ring is also supported by appearance of new weak intensity bands around  $420\text{-}410\text{ cm}^{-1}$  due to Te-N [18,35]. Evidence for formation of proposed macrocycles and coordination through N atoms is further supported by new medium to weak intensity bands around  $480\text{-}450\text{ cm}^{-1}$  assignable to M-N stretching [36]. The  $\nu_{\text{M-Cl}}$ , however could not be ascertained due to non-availability of far infrared data.

### <sup>1</sup>H NMR Spectra

The proton chemical shifts for some representative complexes which are soluble in DMSO -d<sub>6</sub> are compiled in Table 4.

**Table 4.** <sup>1</sup>H NMR data ( $\delta$  ppm) for metal complexes

Complex	-NH-	-CH <sub>3</sub> *	-OCH <sub>3</sub>	-CH <sub>2</sub> -	Phenyl	-OH
[CuL <sup>1</sup> Cl <sub>2</sub> ]	1.74 s (4H)	---	---	2.50 m (8H)	6.87 d (8H) 7.79 d (8H)	6.16 b (4H)
[CoL <sup>2</sup> Cl <sub>2</sub> ]	2.04 s (4H)	2.49 s (12H)	---	2.13 m (8H)	6.91 d (4H) 7.59 d, 7.67 s (8H)	9.97 b (4H)
[CuL <sup>2</sup> Cl <sub>2</sub> ]	1.78 s (4H)	2.49 s (12H)	---	2.07 m (8H)	6.93 d (4H) 7.59 d, 7.68 s (8H)	8.11 b (4H)
[MnL <sup>3</sup> Cl <sub>2</sub> ]	1.74 s (4H)	---	3.73 s (12H)	2.50 m (8H)	7.06 d (8H) 7.71 d (8H)	---
[CuL <sup>3</sup> Cl <sub>2</sub> ]	1.83 s (4H)	---	3.70 s (12H)	2.50 m (8H)	7.14 d (8H) 7.74 d (8H)	----

\* May be mixed with solvent peak, s = singlet, d = doublet, m = multiplet, b = broad.

The phenyl protons in metal complexes resonate at slightly up field side (6.87-7.79  $\delta$  ppm) as compared to parent diaryltellurium dichlorides [19,20,37], due to increase in electron density at the tellurium atom as a result of replacement of 2 Cl by 2 N atoms. Ethylenediamine, <sup>(a)</sup>H<sub>2</sub>N-(<sup>(b)</sup>CH<sub>2</sub>)-<sup>(a)</sup>NH<sub>2</sub> shows [38] two sets of four equivalent protons each at (a) = 1.19  $\delta$  ppm and (b) = 2.74  $\delta$  ppm. Metal complexes do not show any signal attributable to free -NH<sub>2</sub>, instead a broad singlet at around 1.74-2.04  $\delta$  ppm, which may be assigned to coordinated secondary amino group [39] is observed. This confirms the formation of proposed macrocycle skeleton. The deshielding of -NH- protons further suggests the donation of electron density to the metal-ions. The methylene protons in these metal complexes resonate at 2.07-2.50  $\delta$  ppm, as a multiplet as reported [18, 32, 34] for other tetraazamacrocycles derived from ethylenediamine. Also, the independence of aryl proton chemical shifts on the nature of metal-ion precludes the possibility of Te-M bond. Thus, <sup>1</sup>H NMR studies on these metal complexes support the formation of 10-membered tellurium tetraazamacrocycles and their tetra dentate ligation behaviour as predicted by IR studies.

Based on analyses, conductance, magnetic, electronic, infrared and proton magnetic resonance spectral studies, a distorted octahedral geometry involving four N atoms of tetraazamacrocycle and 2 Cl may be proposed for these metal complexes.

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