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## Synthesis, Characterization and Spectral Studies of the Chelate Complexes of $[C_5H_{10}Te(OH)_2]$ and $[C_8H_8Te(OH)_2]$

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### **Abstract:**

*The reactions of 1,1,2,3,4,5,6-heptahydro-1,1-dihydroxo tellurane  $C_5H_{10}Te(OH)_2$  (in situ) and 1,3-dihydro-2 $\lambda^4$ -benzotellurole-2,2-diyl dihydroxide  $C_8H_8Te(OH)_2$  (in situ) with oxalic acid, phthalic acid and EDTA which yield the chelate complexes. and were characterized through elemental analysis and IR spectroscopic studies.*

**Keywords:** Organotelluriums, Chelate, carboxylic acids.

### **1. Introduction:**

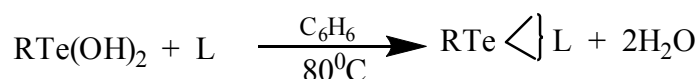
Chelates are of biological importance, for example haemoglobin in the red blood cells contains an iron porphyrin complex,<sup>(1)</sup> chlorophyll in green plant contains a magnesium porphyrin complex<sup>(2)</sup> and vitamin B-12 is cobalt corrin complex.<sup>(3)</sup> From structural point of view, the chelates (ring structures) are formed by the ligands containing two, three, four and six donor atoms i.e. by bidentate, tridentate, tetradentate and hexadentate ligands. The specific examples include oxalate ion, 2,2'-dipyridyl, acetylacetonate ion, ethylenediaminetetraacetic acid (EDTA)<sup>(4)</sup>. The ethylenediaminetetraacetic acid chelate complexes are of special mention because they not only form stable complexes<sup>(5-8)</sup> but metal-EDTA complexes, for example  $Ca^{2+}$ -EDTA complex are used for the treatment of lead and copper poisoning. Dermatitis caused by chromium, nickel salts is treated with EDTA cream.<sup>(4)</sup>

We have not come across the chelate complexes of organotellurium complexes with oxalate ion, phthalate ion and EDTA, hence in the present chapter we report the reactions of 1,1,2,3,4,5,6-heptahydro-1,1-dihydroxo tellurane  $C_5H_{10}Te(OH)_2$  (in situ) and 1,3-dihydro-2 $\lambda^4$ -benzotellurole-2,2-

diyl dihydroxide  $C_8H_8Te(OH)_2$  (*in situ*) with oxalic acid, phthalic acid and EDTA which yield the chelate complexes.

## 2. Experimental:

$C_5H_{10}TeI_2^{(9)}$ ,  $C_8H_8TeI_2^{(10)}$  were prepared by the literature methods and  $C_5H_{10}Te(OH)_2$  and  $C_8H_8Te(OH)_2$  were generated (*in situ*) by the reactions of  $C_5H_{10}TeI_2$  and  $C_8H_8TeI_2$  with freshly prepared AgOH (*in situ*) in deionised water. The chelate complexes of  $C_5H_{10}Te(OH)_2$  and  $C_8H_8Te(OH)_2$  were obtained by the following reaction in 1:1 molar ratio in dry benzene.



(R =  $C_5H_{10}$ ,  $C_8H_8$  & L = oxalic acid, phthalic acid and EDTA)

### 2.1 Synthesis of $C_5H_{10}Te(OH)_2$ and $C_8H_8Te(OH)_2$

To  $C_5H_{10}TeI_2$  (2.0g, 4.43 mmol) was added freshly prepared silver hydroxide (1.10 g, 8.86 mmol) in a mortar and ground with a little amount of deionised water to make a thick paste. It was ground for 45 minutes more after which the reddish-brown crystals of 1,1,2,3,4,5,6-heptahydro-1,1-diiodo tellurane disappeared. The paste was filtered with hot water and the filtrate was taken as  $C_5H_{10}Te(OH)_2$  (*in situ*). Similarly  $C_8H_8Te(OH)_2$  (*in situ*) was obtained by the reaction of  $C_8H_8TeI_2$  (2.0 g, 4.12 mmol) and freshly prepared silver hydroxide (1.03 g, 8.24 mmol) in deionised water.

### 2.2 Reaction of $C_5H_{10}Te(OH)_2$ with Oxalic acid

A mixture of  $C_5H_{10}Te(OH)_2$  (*in situ*) (1.03g, 4.45mmol) and oxalic acid (0.56g, 4.45mmol) in benzene (500 ml) was heated under reflux for ~8h using a Dean-Stark apparatus to remove the water formed in the reaction by azeotropic distillation. The mixture was filtered and reduced (~10ml)

to obtain the solid;  $C_5H_{10}Te \begin{array}{l} \diagup O(O)C \\ \diagdown O(O)C \end{array}$  [Yield: 0.62g (48.82 %), m.p. 170°C].

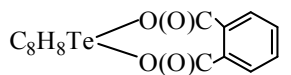
In the same way as above,  $C_5H_{10}Te(OH)_2$  (*in situ*) (1.03g, 4.45mmol) was reacted with phthalic acid (0.74g, 4.45mmol) and ethylenediaminetetra acetic acid (1.65g, 4.45mmol) to obtain their respective chelate complexes.

### 2.3 Reaction of $C_8H_8Te(OH)_2$ with Oxalic acid

A mixture of  $C_8H_8Te(OH)_2$  (*in situ*) (1.09g, 4.10mmol) and oxalic acid (0.52g, 4.10mmol) in benzene (500 ml) was heated under reflux for ~8h using a Dean-Stark apparatus to remove the water formed in the reaction by azeotropic distillation. The mixture was filtered and reduced (~10ml) to

obtain white solid;  $C_8H_8Te \begin{array}{l} \diagup O(O)C \\ \diagdown O(O)C \end{array}$  [Yield: 0.75g (57.25 %), m.p. 220°C(d)].

In the same way as above,  $C_8H_8Te(OH)_2$  (*in situ*) (1.09g, 4.10mmol) was reacted with phthalic acid (0.68g, 4.10mmol) to obtain the



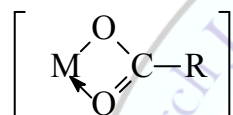
### 3. Results and Discussion:

The characterization data of chelate complexes of  $C_5H_{10}Te(OH)_2$  and  $C_8H_8Te(OH)_2$  are listed in **Table 3.1**. They are coloured solids, stable at room temperature and are not affected by the atmospheric moisture. They are insoluble in common organic solvents such as chloroform, acetone and dichloromethane.

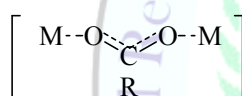
#### 3.1 Infrared Spectra:

The infrared spectra of chelate complexes of  $C_5H_{10}Te(OH)_2$  and  $C_8H_8Te(OH)_2$  in KBr have been recorded in the range of  $4000-400\text{ cm}^{-1}$  and the data are listed in **Table 3.2**. The various possible ways of bonding of carboxylate group to metal ions are as follows:

- (i) in ionic  $[M]^+ [OCOR]^-$
- (ii) as unidentate  $[M-OCOR]$
- (iii) as bidentate



- (iv) as bridging



The  $\Delta\nu\text{OCO}$  ( $\nu_{\text{asymOCO}} - \nu_{\text{symOCO}}$ ) is the widely accepted criterion for distinguishing between the above described different modes of bonding. Thus  $\Delta\nu\text{OCO}$  for unidentatetetracyclohexyl tin acetate is  $361\text{ cm}^{-1}$ <sup>(11)</sup>; for bidentate dimethyl tin diacetate is  $227\text{ cm}^{-1}$ <sup>(12)</sup>; for bidentate dimethyl tellurium oxalate is  $252\text{ cm}^{-1}$ <sup>(13)</sup> and in trimethyl tin acetate (containing bridging carboxylate group); its value is reported at  $153\text{ cm}^{-1}$ <sup>(14)</sup>. In the free carboxylic acids, used in the present investigation, the  $\nu_{\text{asymOCO}}$  absorption lies in the range of  $1750-1555\text{ cm}^{-1}$  and the  $\nu_{\text{symOCO}}$  absorption lies in the range  $1400-1300\text{ cm}^{-1}$ <sup>(7,12,13,15)</sup>. In newly synthesized chelate complexes of  $C_5H_{10}Te(OH)_2$  and  $C_8H_8Te(OH)_2$ ,  $\nu_{\text{asymOCO}}$  and  $\nu_{\text{symOCO}}$  absorptions appear at  $1632 \pm 10\text{ cm}^{-1}$  and  $1393 \pm 15\text{ cm}^{-1}$  respectively. The  $\Delta\nu\text{OCO}$  values are in the range of  $249 \pm 20\text{ cm}^{-1}$  (**Table 3.2**) which indicate bidentate nature of the carboxylate groups.  $\nu\text{TeCH}_2$  appear at  $522 \pm 41\text{ cm}^{-1}$ .

In the case of EDTA complexes, of  $C_5H_{10}Te(OH)_2$  the presence of absorptions at  $1700\text{ cm}^{-1}$  (**Table 3.2**) is due to the presence of free carboxylic acid groups' carbonyl stretchings while the band at  $1622\text{ cm}^{-1}$  (**Table 3.2**) is due to the complexed carboxyl groups of EDTA. In the absence of X-ray structure data of the EDTA complex, coupled with insoluble character of the complex, it is difficult to assign the exact stereochemistry of the complex, but EDTA acts as a bidentate ligand (as

discussed above) which is in agreement with the observations of Busch et al.<sup>(7)</sup>who, also, on the basis of infrared spectroscopic data only described the presence of bidentate EDTA in EDTA complexes of Pt(II) and Pd(II) obtained by the reaction of PtCl<sub>2</sub> and PdCl<sub>2</sub> with EDTA.

**Table 3.1** Characterization data of chelate complexes of C<sub>5</sub>H<sub>10</sub>Te(OH)<sub>2</sub> and C<sub>8</sub>H<sub>8</sub>Te(OH)<sub>2</sub>

Sr. No.	Complex	M.P (°C)	Colour	Yield (%)	Elemental Analysis (%) Obs. (Calcd.)			
					C	H	N	Te
1.		170	cream	48.82	29.40 (29.41)	3.47 (3.50)	-	44.64 (44.68)
2.		244	cream	74.53	43.10 (43.14)	3.84 (3.87)	-	35.27 (35.29)
3.		170	cream	96.19	33.83 (33.86)	4.12 (4.14)	5.26 (5.27)	23.98 (24.00)
4.		220(d)	cream	57.25	37.54 (37.55)	2.47 (2.50)	-	39.90 (39.92)
5.		260	grey	45.06	48.51 (48.53)	3.00 (3.03)	-	32.23 (32.25)

**Table 3.2** Infrared frequencies (cm<sup>-1</sup>) of chelate complexes of C<sub>5</sub>H<sub>10</sub>Te(OH)<sub>2</sub> and C<sub>8</sub>H<sub>8</sub>Te(OH)<sub>2</sub>

Sr. No.	Complex	$\nu_{\text{asym OCO}}$	$\nu_{\text{sym OCO}}$	$\Delta\nu \text{ OCO}$	$\nu \text{ TeCH}_2$
1.		1641(w)	1378(m)	262	510(w)
2.		1630(s)	1400(s)	230	515(m)
3.		1622(m)*	1396(s)	268	546(m)
4.		1632(m)	1392(m)	240	481(s)
5.		1637(w)	1408(s)	229	562(s)

\*1700cm<sup>-1</sup> (carboxylic acid carbonyl stretching of EDTA)

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