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Synthesis, characterization of Trinuclear containing Se(II) donar group of Co(II) complexes.

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Abstract:

Galaxy dihydrazone reacts with $Cocl_2$ in reflecting hot ethanolic solution (ethanol) to gives the compound of green product. Trinuclear complexes of general formula was prepared by a direct reaction of bis- (o-formyl pheny) selenide with glyoxal dihydrazone Co(II). The prepared compounds and complexes were characterised by IR, electronic spectra and NMR, conductivity measurement, magnetic measurements were determined spectrophotometrically. The electronic spectra and magnetic measurements indicate that the Co and Se complexes have squre planer and octahydral geometry.

Keywards: Selenium, antioxidant, bis(o-formylphenyl) selenide, [dibromo-bis-(dihydrazone cobalt(II)], anticancer agent.

1.Introduction:

Metal complexes play an important role in human health, plant and animal like due to their physico-chemical properties. Among trinuclear containing compounds selenium donor of Co(II) complexes as well as biological importance. Glyoxal dihydrazone (II) and bis-(o-formyl phenyl) selenide have been reported precoursur.

In the present work, the oxidation addition of bis-(o-formyl phenyl) selenide to dibromo- bis(dihydrazone) Co(II) in (2:1)) molar ratio have been reported, also reported the synthesis of trinuclear complexes of general formula [Se2CoMcBr2] (Mc = C32H24N8 as well as adducts with some neutral donor Ligands. The complex [Se2CoMcBr2] where prepared and characterized by physical, chemical, spectral data, magnetic and electronic spectra suggest.

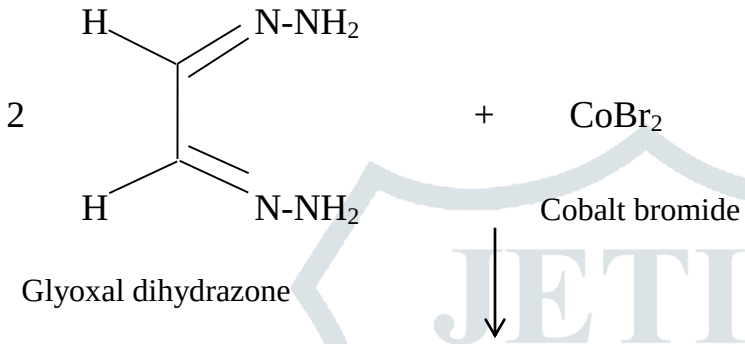
In view of this interesting result and as continuation of our comprehensive studies of transition and non-transition metal complexes with selenium containing ligand, we have prepared multinuclear complexes.

2.Experiment

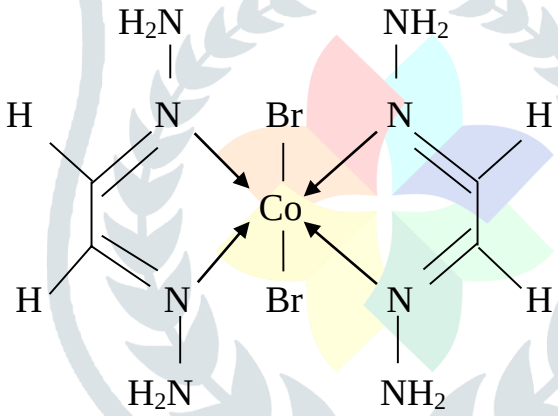
Prepration of [dibromo-bis-(dihydrazone) cobalt (II)]:-

About 1.72gm(0.02mole) of Glyoxal dihydrazone and 2.19gm (0.01 mole) of cobalt bromide was dissolved in distilled water and treated with hot ethanolic solution and was refluxed for 3 hrs. at 52°c . The green product was formed which was washed with ether and then dried under vacuum.

[Dibromo-bis-(dihydrazone) cobalt(II)]:-



Glyoxal dihydrazone

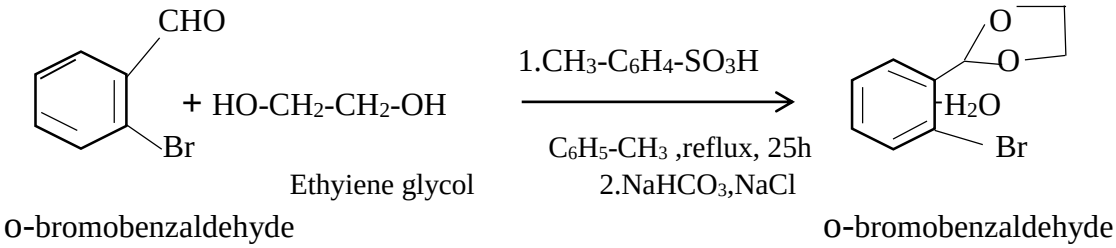


[dibromo-bis-(dihydrazone) cobalt (II)]

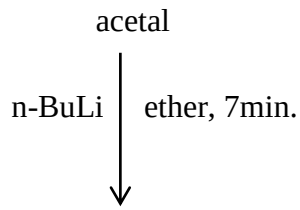
Prepration of Precursors

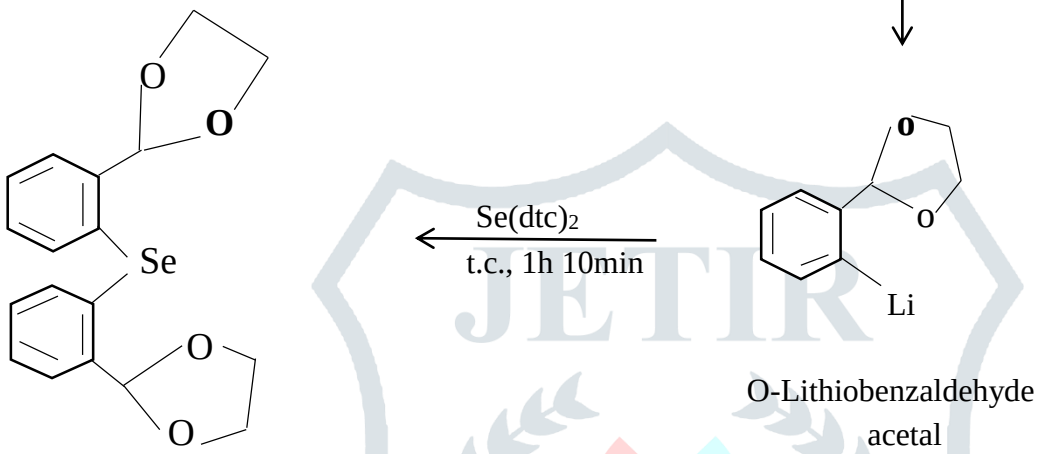
2.3 .Prepration of bis-(o-formyl phenyl) selenide

A mixture of 3.70 gm (0.01 mole) of O-bromobenzaldehyde was used as starting material. O-bromobenzaldehyde were prepared by reported produceres[18]. A mixture of 3.70 gm (0.01 mole) o-bromobenzaldehyde and 0.62 gm (0.01 mole) of ethyleneglycol in presence of toluene sulphonic acid and toluene and was refluxed for 25 hrs. at room temperature. The yellow product was obtained, which was washed with ether and then dried in vacuum. At the obtained product 2.29 gm (0.01 mole) of o-bromobenzaldehyde acetal and 0.64 gm (0.01 mole) of n- butyl lithium added in ether for 7 min. at 25°c. A mixture of treatment of 1.56 gm (0.01 mole) o-lithiobenzalde acetal with 2.63 gm (0.01) of selenium dithiocarbamate in ether led at 25°c followed by extraction to bis-(o-formyl phenyl) selenide acetal. Bis(o-formyl phenyl) selenide was formed by refluxing 2 hrs. 25°c then bis-(o-formyl phenyl) selenide acetal in concentrated HCl for removing protecting group.



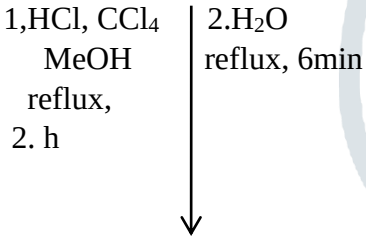
o-bromobenzaldehyde o-bromobenzaldehyde

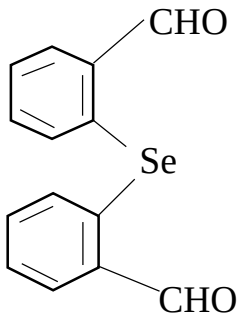




bis- (o-formyl phenyl) selenide acetal

O-Lithiobenzaldehyde acetal

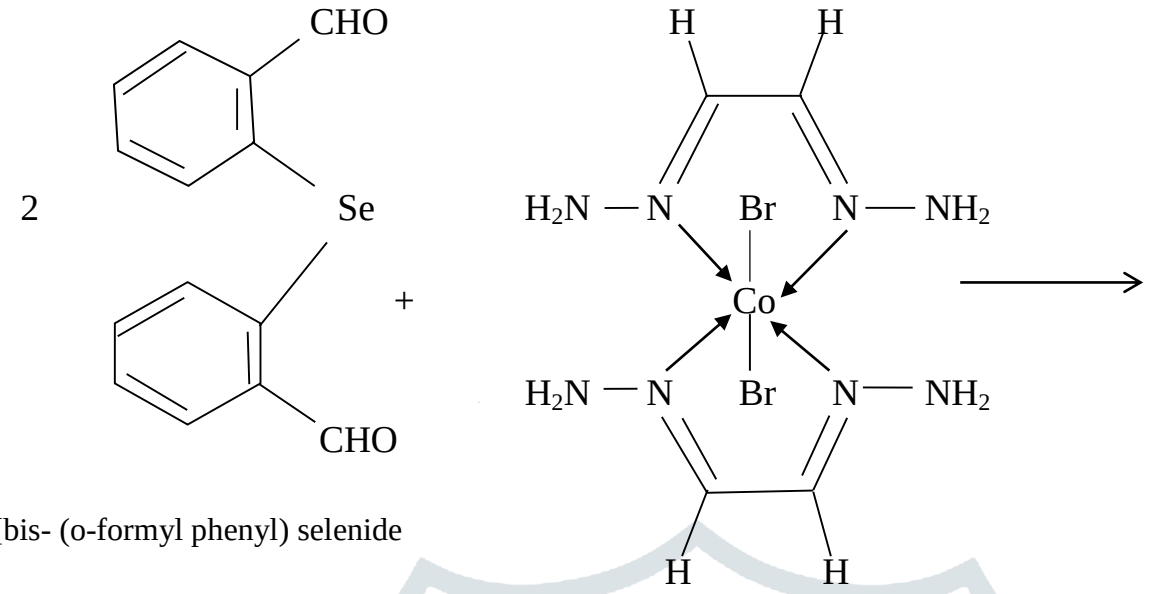




[bis- (o-formyl phenyl) selenide

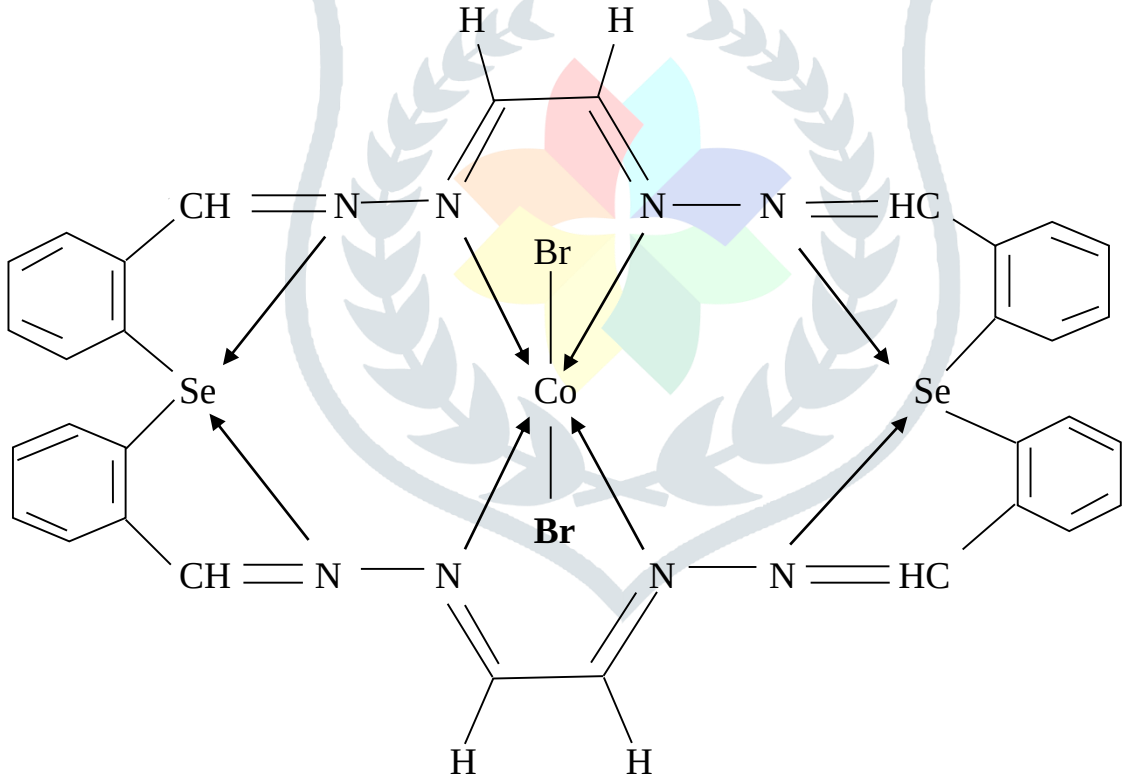
Prepration of trinuclear complexes :

5.78 gm (0.02 mole) bis (o-formyl phenyl) selenide and 3.91 gm (0.01 mole) of [dibromo-bis-(benzildihydrazone) cobalt(II)] were dissolved in distilled water and ethanol and was refluxed for 2 hrs. at 52°C. The green product was obtained which was washed with ether and then dried under vacuum .



[bis- (o-formyl phenyl) selenide

[dibromo-bis-(dihydrazone) cobalt (II)]



3. Characterization:

The analytical data of the compounds and complex are given in table(a, b, c). These data are in a good agreement bearing the proposed formulae. All compounds are air-stable at room temperature and the insoluble in the common organic solvent but soluble in DMF or DMSO. The elemental analysis for C, H N, Br, Se, Co were determined as

Molecular formula-$C_4H_{12}N_8Br_2Co$

Element	Calculated %	Found %
Carbon	12.28	12.30
Hydrogen	3.07	3.05
Nitrogen	28.64	28.66
Bromine	40.92	40.95
Cobalt	15.09	15.10

Table:-(a)

Molecular formula-$C_{14}H_{10}O_2Se$

Element	Calculated %	Found %
Carbon	58.11	58.12
Hydrogen	3.44	3.46
Nitrogen	11.09	11.10
Selenium	27.36	27.35

Table (b)

Molecular formula- $Se_2CoMcBr_2$

Element	Calculated %	Found %
Carbon	42.81	42.84
Hydrogen	02.68	02.65
Nitrogen	12.49	12.50
Bromine	17.83	17.85
Selenium	17.61	17.62
Cobalt	6.58	6.60

Table 2.4 (c)

Where Mc = $[C_{32}H_{24}N_8]$

The compounds where characterized by IR, spectroscopy, NMR (^1H, ^{13}C, ^{77}Se) and mass spectroscopy. Molar conductance of the complex was measured at room temperature. Infrared spectra of this prepared complex was recorded in KBr and CsI on Perkin-Elmer 457 spectrometer.

4. Result and Discusion:

After elemental analysis [(Table no.(a), (b), (c), stoichiometry of synthesized precursor and bichelating non macrocylic have been found to be $[C_{32}H_{24}N_8Se_2Br_2Co]$.

The μ_{eff} of synthesized complexes have been calculated to be 3.7 BM and was presumed due to presence of Central octahedral complex. It is excess value and presumably due to temperature independent paramagnetic nature.

4.1 Infrared spectra

There is no band establish in the region 3300-3100 cm^{-1} which can be assigned to the stretching vibrations of the $-NH_2$ groups of the precursor dihydrazones and suggestes metal ion assisted template condensation of the $-NH_2$ groups bearing the [bis-(o-formyl phenyl) selenide]. In former reported spectra of [bis-(diacetyldihydrazone)M(II)]$^{+2}$ complexes. The $-NH_2$ asymmetric and symmetric stretching mode of vibrations 3060-2870cm^{-1} have been detected like two strong bands in the region 3300 to 3200 cmi^{-1} in the region.

4.2 Electronic spectra and magnetic properties:-

To elucidate the structural problems, electronic spectra (Table: A) of the precursors & tricyclo macrocyclic on Shimadzu 160 A and Beckman - 20 spectrophotometers in order to complex were recorded respectively.

Electronic Spectral Bands (in cm^{-1}) and magnetic moments(in B.M.) of the trinuclear selenium(II) Se(II) macrocylic complexes.

Complexes	**$^3B_1g \rightarrow {}^3Eg$**	**^4B_2g**	**$^3T_1g(F)$**	**$^3T_1g(P)$+C.T**	**C.T.**	**μ_{Eff}/metal ion**
$Se_2CoMcBr_2$	87000	11500	16100	28300	-	2.76

TABLE -A

4.3 NMR:-

Compound bis-(o-formal phenyl) selenide was analized by(^1H,^{13}C,^{77}Se) NMR, IR spectroscopy and mass spectroscopy ^{1}H-NMR spectrum of the compound confirmed by the value of resonance peaks were ð (ppm) 8.58.

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