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SYNTHESIS AND SPECTRAL STUDIES OF Ni(II) COMPLEXES WITH 2-HEPTANONESEMICARBAZONE AND 2-HETANONETHIOSEMICABAZONE

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ABSTRACT: Complexes of Nickel(II) with 2-heptanonesemicarbazone (hsc) and 2-heptanonethiosemicabazone (htc) have been synthesized and characterized by elemental analysis, molar conductance measurements, magnetic moments, mass, IR and electronic/spectral studies. Synthesized complexes were found to have a general composition of $Ni(L)_2X_2$ (where $X=Cl^-$, $1/2SO_4^{2-}$, NO_3^-, ClO_4^-, NCS^-, CH_3COO^- and L=hsc or htc). Molar conductance indicates that all the complexes are non-electrolytes in nature whereas chloro complexes are 1:1 electrolytes. Metal complexes of 2-heptanonesemicarbazone were soluble in hot ethanol, methanol, acetone and DMSO and insoluble in chloroform, benzene and water, whereas metal complexes of 2-hepatanonethiosemicarbazone were soluble in ethanol, methanol and acetone, insoluble in chloroform and benzene. On the basis of IR and electronic spectral analysis thiocyanato, sulphato, perchlorato, acetato and nitrato complexes of were found to have six-coordinate octahedral geometry whereas chloro complexes were five-coordinate and having trigonal bipyramidal geometry.

INTRODUCTION: Transition metal complexes of semicarbazones and thiosemicabazones have received considerable attention because of their antibacterial, antifungal, antitumor, antiamoebic, antimalarial, antiviral, radioprotective, trypanocidal and anti-inflammatory activities[1-5]. Semicarbazones and thiosemicarbazones are among the most relevant nitrogen-oxygen/nitrogen-sulphur donor ligands [2, 3]. Thiosemicarbazone complexes have imine group (-N=CH-), which provides the biological activity and chelating properties favoring the central metal atom [10].

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Some significant bioactivities of transition metal complexes of semicarbazones (antiprotozoa, and anticonvulsant) and thiosemicarbazones (antibacterial, antifungal, antitumoral, antiviral) have been reported [3, 7]. The biological activity is considered to involve three kinds of mechanisms: (a) inhibition of ribonucleoside diphosphate reductaseenzyme (essential for DNA synthesis); (b) creation of lesions in DNA strand by oxidative rupture; (c) binding to the nitrogen bases of DNA or RNA, hindering or blocking base replication [1, 9].

It has been found that certainthiosemicarbazones are specific inhibitors of ribonucleotide reductase (an important metabolic target for the development of chemotherapeutic agents against cancer) [11]. Thiosemicarbazones and semicarbazones have the ability to produce some new and unique complexes with enhanced biological and analytical properties [4, 6]. Metal complexes of Ni(II) are found to act as a sensor [8] in many of the diseases for which thiosemicarbazones have been shown to effective[4]. The present paper reported the synthesis and characterization of Ni(II) complexes with 2-heptanonesemicarbazone (hsc) and 2-heptanonethiosemicabazone (htc).

MATERIALS AND METHODS:

All chemicals used were of A. R. grade and produced from sigma Aldrich, Bangalore, India. Metal salts were purchased from E. Merck, India and were used as received.

Synthesis of Schiff's base Ligand L_1 (hsc):

Ethanolic solution of semicarbazide hydrochloride (1.11g, 0.01mol) and sodium acetate (0.82g, 0.01mol) were dissolved in 50 mL of distilled water followed by the addition of 2-heptanone (1.42mL, 0.01mol). The reaction mixture was refluxed for about an hour on a water bath. White colour precipitate was formed. The precipitate was filtered and washed with ethanol. The same was dried over P_4O_{10}. The purity of ligand was checked by TLC.

Synthesis of Schiff's base Ligand L_2 (htc):

Ethanolic solution of thiosemicarbazide (0.91g, 0.01mol) and ethanolic solution of the 2-heptanone (1.42 mL, 0.01 mol) were mixed together in equimolar ratio (1:1), 2-3 drops of glacial acetic acid was added and the contents were refluxed on a water bath for about one hour and then concentrated. On cooling the reaction mixture in an ice bath, a yellowish coloured product was separated out. The purity was checked by TLC. **Table 1** is showing analytical data of the Schiff's base Ligands (hsc&htc).

TABLE 1: ANALYTICAL DATA OF THE SCHIFF'S BASE LIGANDS (HSC&HTC)

				Elemental analysis found /(calculated)		
Ligand	Colour	Yield%	M.P (^{0}C)	C%	H%	N%
L_1 = hsc ($C_8H_{17}N_3O$)	White	85	127	56.14 (56.10)	9.94 (9.89)	24.56 24.53)
L_2 =htc ($C_8H_{17}N_3S$)	Yellow	80	77	51.4 (50.9)	9.1 (9.0)	22.5 (23.0)

Synthesis of Complexes:

Hot ethanolic solution of corresponding metal salts (0.05 mol) ($NiCl_2$, $NiSO_4.6H_2O$, $Ni(NO_3)_2$, $Ni(CH_3COO)_2$, $Ni(SCN)_2$ and $Ni(ClO_4)_2$) were mixed with hot ethanolic solution schiff's base ligand L_1& L_2 (0.1mol). The mixture was refluxed for about 3 hours on a water bath. On cooling metal complexes of different colors were separated out. The obtained complexes were washed with 50% ethanol and dried.

Physical Measurements:

Elemental analysis was performed on a Carlo-Erba 1106 elemental analyzer. Molar conductance was measured on the Elico (CM82T) conducting bridge. Magnetic susceptibility was measured at room temperature on a Gouy balance using $CuSO_4.5H_2O$ as a calibrant. A mass spectrum was recorded on JEOL, JMS.DX-303 mass spectrometer. IR spectra were recorded on a FTIR spectrum BX-II spectrophotometer. The electronic spectra were recorded in DMSO on Shimadzu UV mini-1240 spectrophotometer.

RESULT AND DISCUSSION: Metal complexes were synthesized by reacting ligands 2-heptanonesemicarbazone (hsc) or 2-heptanonethiosemicarbazone (htc) with metal ion in 2:1 ratio in ethanolic medium. Elemental analysis of the complexes reveal that the complexes have general composition, $[Ni(L)_2X_2]$ (where L=hsc or htc and X=Cl^-, $1/2SO_4^{2-}$, NO_3^-, ClO_4^-, NCS^-, CH_3COO^-). Chloro complexes of semicarbazone or thiosemicarbazone of heptanone are 1:1 electrolytes and may be formulated as $[NiL_2X]X$.

The ligands behaved as bidentate and coordinate through sulphur/oxygen and nitrogen donor atoms. Table II reveals the color, composition and magnetic moment of the complexes of Ni(II) with 2–heptanonesemicarbazone or 2-heptanonethiosemicarbazone.

These complexes exhibit different stereochemistry, varying from five-coordinate square pyramidal or trigonal bipyramidal to six-coordinate octahedral.

All the complexes were found to be soluble in DMSO. Magnetic moment of all complexes of present study lies in the range of 2.95-3.05 B.M., which correspond to two unpaired electrons. Metal complexes of 2-heptanonesemicarbazone were soluble in hot ethanol, methanol, acetone and DMSO and insoluble in chloroform, benzene and water, whereas metal complexes of 2-hepatanonethiosemicarbazone were soluble in ethanol, methanol and acetone, insoluble in chloroform and benzene.

TABLE 2: COLOUR, COMPOSITION AND MAGNETIC MOMENT OF THE COMPLEXES OF Ni(II) WITH 2-HEPTANONESEMICARBAZONE AND 2-HEPTANONETHIOSEMICARBAZONE

Complex	Colour	Yield	%Ni	% C	%H	%N	μeff(B.M)
$Ni(hsc)_2Cl_2$	Light Green	70%	12.34 (12.32)	40.85 (40.87)	7.23 (7.21)	17.87 (17.85)	3.01
$Ni\ (hsc)_2SO_4$	Blue	70%	11.69 (11.68)	38.70 (38.71)	6.85 (6.89)	16.93 (16.93)	3.02
$Ni(hsc)_2(NO)_2$	Purple	75	11.09 (11.68)	36.64 (36.62)	6.48 (6.42)	21.37 (21.28)	3.00
$Ni(hsc)_2(CH_3COO)_2$	Greenish white	80%	11.19 (11.18)	46.33 (46.32)	7.72 (7.73)	16.21 (16.20)	3.00
$Ni(hsc)_2(ClO_4)_2$	Green	65%	9.69 (9.68)	32.10 (32.11)	5.68 (5.67)	14.04 (14.04)	2.95
$Ni(hsc)_2\ (NCS)_2$	Blue	60%	11.98 (11.97)	44.60 (44.61)	7.02 (7.00)	23.14 (23.15)	3.05
$Ni(htc)_2Cl_2$	Green	75%	11.55 (11.56)	38.24 (38.25)	6.77 (6.75)	16.73 (16.70)	3.00
$Ni(htc)_2SO_4$	Brown	80%	10.98 (10.93)	36.36 (36.37)	6.43 (6.40)	15.90 (15.91)	2.92
$Ni(htc)_2(NO_3)_2$	Purple	70%	10.443 (10.40)	34.53 (34.51)	6.11 (6.12)	20.14 (20.13)	2.98
$Ni(htc)_2(CH_3COO)_2$	Green	85%	10.54 (10.50)	43.63 (43.68)	7.27 (7.29)	15.27 (15.30)	3.07
$Ni(htc)_2(ClO_4)_2$	Green	65%	9.20 (9.21)	30.47 (30.49)	5.39 (5.40)	13.33 (13.30)	2.99
$Ni(htc)_2\ (NCS)_2$	Blue	75%	11.24 (10.25)	41.86 (40.87)	6.58 (6.58)	21.70 (21.70)	3.00

Mass Spectra:

The proposed formula of ligand L_1 and L_2 were confirmed by electronic impact mass spectrum. The ligand L_1 showed a molecular ion peak at m/z =171amu corresponding to species $[C_8H_{17}N_3O]^+$ and ligand L_2 showed a molecular ion peak at m/z=187 amucorresponding to the species $[C_8H_{17}N_3S]^+$.

IR & Electronic Spectra:

For first Ligand 2-heptanonesemicarbazone, the characteristic absorption of the carbonyl group was observed at 1900-1700 cm^{-1} [12, 13]. The amide band in semicarbazone has been observed at 1556-1588 cm^{-1}. The band at 1650-1600 cm^{-1} in 2-heptanonesemicarbazone apparently has large contribution from ν(C=N) [12, 14].

In all metal-ligand complexes, coordination through 'O' increases double bond character of the ν(C=N) band as compared to the ligand, which shows that 2-heptanonesemicarbazone behaves as bidentate ligand.

The second ligand, 2-heptanonethiosemicarbazone may exihibits thione-thiol tautomerism as it contains a thioamide –NH-C=S functional group. In the IR spectrum of ligand,ν(S-H) band at 2560 cm^{-1} is absent but ν(N-H) band at ca.3214 cm^{-1} is present, which indicats ligand remains as the thione-thiol tautomer in the solid state. In thiosemicarbazone the position of ν(C=N) band appeared at 1600 cm^{-1}, which is shifted towards lower wave number in the complexes indicating coordinated via the azomethine nitrogen [12,15,16].

The spectra of $[Ni(L)_2(NCS)_2]$ complexes, under the study, show three bands at 9220-10520cm^{-1}, 13800-19230 cm^{-1} and 23800-29410 cm^{-1}, (Table

III) which clearly rule out tetrahedral geometry for these complexes. The thiocyanato complexes show only one thiocyanatoν(C=N) absorption at 2100 cm^{-1}suggesting that both the NCS groups being in the same environment and coordinated through nitrogen atom[17, 18].

Electronic Spectra of these complexes are also characteristic of pseudo octahedral Ni(II), with ν_1 at $^3A_{2g}(F)\rightarrow{}^3T_{2g}(F)$, ν_2 at $^3A_{2g}(F)\rightarrow{}^3T_{1g}(F)$ and ν_3 at $^3A_{2g}(F)\rightarrow{}^3T_{1g}(P)$, laying at 9793-l0400 cm^{-1}, 17968 —19050 cm^{-1} and 25700-26315 cm^{-1} respectively, the ν_2/ν_1 and ν_3/ν_1 ratio in these complexes is 1.945-1.727 and 2.471-2.687, well within the characteristic range[19] (1.5-2.9) for octahedral nickel(II).

The electronic spectra of [Ni(hsc)$_2$X$_2$] (X-sulphate, perchlorate, acetate) complexes display absorption bands at 9220-9880 cm^{-1}(ν_1), 15873-19230cm^{-1}(ν_2) and 25641-29410 cm^{-1}(ν_3), which may arise from both six-coordinate and five-coordinate complexes of nickel(II) and ν_2/ν_1 ratio is found in the range of 1.682-1.946, which suggests octahedral geometry for the complexes. The sulphatocomplexes show IR S-O stretching frequencies at 1270, 1153, 1097 and 977 cm^{-1}, suggesting that the ligand behaves asbidentate chelating agent coordinating through azomethine nitrogen and thiolate sulphur [20, 21].

The perchloratocomplexes show absorption bands at 1150 cm^{-1}(ν_1), 1020 cm^{-1}(ν_2) and 970 cm^{-1}(ν_4) cm^{-1}suggesting unidentatebehavior of perchlorato group.The acetato complex under study shows infrared carboxyl stretching frequencies at 1585 cm^{-1}for ν(C=O)and 1370cm^{-1} forν(C-O). The 210cm^{-1}difference between ν(C=O) and ν(C-O) suggests a unidentate [22] behaviour of both the acetato groups in this complex.

IR spectra of nitrato complexes show bands corresponding to both coordinated and uncoordinated nitrato [23]. The coordinated nitrato show infrared bands at 1505-1490 cm^{-1}(ν_1), 1325-1275 cm^{-1} (ν_5), 1045-1020 cm^{-1} (ν_2), and 808-803 cm^{-1} (ν_6). The complexes under the study show infrared bands at 1510-1468 cm^{-1}(ν_1) 1383 cm^{-1} (ν_3), 1230-1300 cm^{-1} (ν_5), 1040 cm^{-1} (ν_2) and 870 cm^{-1} (ν_6). The separation of >200 cm^{-1} between ν_1 and ν_5 indicates the bidentate behaviour of nitrato group [23]. The broad absorption band at 1383 cm^{-1}is assigned toν_3 of uncoordinated nitrate [23]. [Ni(htc)$_2$X$_2$] (X-sulphate, acetate, perchlorate) complexes display absorption bands at 9500-l0520cm^{-1} (ν_1), 15873-19050 cm^{-1} (ν_2), and 23800-26310 cm^{-1} (ν_3) (Table III), these spectra are identical to 2-heptanone, which suggest octahedral geometry [23] for these complexes.

Chloro complexes of semicarbazones and thiosemicabazones of heptanoneare 1:1 electrolytes and may be formulated as [NiL$_2$X]X, these complexes show electronic spectral bands at 10280-10400 cm^{-1} (ν_1) , 17625-18150 cm^{-1} (ν_2) and 24400-26315 cm^{-1} (ν_3), which may be assigned to $^1E'(F)\rightarrow{}^3E''(F)$, $^3E'\rightarrow{}^3E''(P)$ and $^3E'\rightarrow{}^3A'_2(P)$ transitions respectively (Table III). $^3E'\rightarrow{}^3A''_1$, $^2A''_2$ transition may be mixed with $^3E'\rightarrow{}^3A'_2(P)$ transition [24]. Thus, five coordinate trigonal bipyramidal geometry may be suggested for these complexes.

TABLE 3: ELECTRONIC SPECTRAL BANDS (cm^{-1})Ni(II) COMPLEXES OF 2-HEPANONESEMICARBAZONE AND 2- HEPTANONETHOSEMICARBAZONES

Complex	$^3T_{2g}(F)\leftarrow{}^3A_{2g}(F)$ (ν_1)	$^3T_{1g}(F)\leftarrow{}^3A_{2g}(F)$ (ν_2)	$^3T_{1g}(F)\leftarrow{}^3A_{2g}(F)$ (ν_3)
$Ni(hsc)_2SO_4$	9220	17860	27250
$Ni(hsc)_2(NO_3)_2$	10362	16129	28580
$Ni(hsc)_2(CH_3COO)_2$	9880	19230	29410
$Ni(hsc)_2(ClO_4)_2$	9434	15873	25641
$Ni(hsc)_2(NCS)_2$	10400	17968	25700
$Ni(htc)_2SO_4$	10520	16030	26310
$Ni(htc)_2(NO_3)_2$	10470	13800	24390
$Ni(htc)_2(CH_3COO)_2$	9700	16130	23800

$Ni(htc)_2(NCS)_2$	9753	19050	26315
$Ni(htc)_2(CIO_4)_2$	9500	15853	25641
	$^3E' \rightarrow ^3E''(F)$	**$^3E' \rightarrow ^3E''(P)$**	**$^3E' \rightarrow ^3A'_2(P)$**
$Ni(hsc)_2Cl_2$	10280	18510	24400
$Ni(htc)_2Cl_2$	10400	17625	26315

Ligand Field Parameters:

Various field parameters are calculated[25] for the complexes and listed in Table-IV. The nephelauxetic parameter ß = B (complex)/B (free ion), where B free ion for Nickel(II) is 1041 cm^{-1}. The value of ß lies in the range 0.84-0.99. These values indicate the appreciable covalent character of metal ligands sigma bond.

TABLE 4: LIGAND FIELD PARAMETERS OF Ni(II) COMPLEXES OF 2-HEPTANONESEMICARBAZONE AND 2-HEPTANONETHIOSEMICARBAZONES

Complex	Dq(cm^{-1})	B(cm^{-1})	β	P(cm^{-1})	v_2/v_1	v_3/v_1	LFSE KJ/mole
$Ni(hsc)_2Cl_2$	1028	1048	0.97	15720	1.765	2.373	147.32
$Ni(hsc)_2SO_4$	922	940	0.87	14100	1.937	2.955	132.13
$Ni(hsc)_2(NO_3)_2$	1030.2	1051	0.97	15765	1.565	2.774	147.64
$Ni(htc)_2(CH_3COO)_2$	988	1008	0.93	15120	1.946	2.976	141.59
$Ni(hsc)_2(NCS)_2$	1040	1061	0.98	15915	1.727	2.471	149.04
$Ni(htc)_2SO_4$	1052	1073	0.99	16095	1.533	2.500	150.76
$Ni(htc)_2(NO_3)_2$	1047	1068	0.84	16020	1.318	2.329	150.05
$Ni(htc)_2(CH_3COO)_2$	970	989	0.91	14835	1.662	2.453	139.01
$Ni(htc)_2(NCS)_2$	979.3	999	0.92	14985	1.945	2.687	140.34
$Ni(htc)_2(CIO_4)_2$	950	969	0.89	14535	1.670	2.699	136.14

CONCLUSION: The Ni(II) complexes of 2-heptanonethiosemicarbazone and 2-heptanonesemicarbazone have been synthesized and studied. The sulphato, perchlorato, acetato, nitrato and thiocyanato complexes of Ni(II) were found to have six-coordinate octahedral geometry, while chloro complexes are assigned to have five-coordinate trigonal bipyramidal geometery.

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