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Synthesis characterization and antimicrobial studies of Fe (III) complexes of thiaosemicarbazide

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ABSTRACT

Some Schiff base derived from 4 -thiosemicarbazone -3-methyl–1-[4-nitrophenyl]-2-pyrazoline -5-one with benzoyl chloride, acetyl chloride, propionyl chloride, p-toluyl chloride. The structures of new compounds were established on the basis of elemental IR and ^{13}*C-NMR data, the compounds were evaluated for their antibacterial activities.*

Keywords: Thiosemicarbezide, TGA, Antimicrobial activity, Spectral studies

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INTRODUCTION

In the Synthetic chemistry, Thiosemicarbazide (TSC) is interesting molecule due to various organic revolutions in which they can take part. The chemistry of thiosemicarbazone complexes of the transition metal ions has been receiving potential attention because of the straight functional mobility of these complexes on the bio system [1]. The anti-proliferative properties of thiosemicarbazones have been attributed to their ability to chelate metal ions because of the presence of an NNS (Nitrogen–Nitrogen–Sulfur) tridentate set of donor atoms that bind not only iron, but also copper, nickel, zinc [2-5]. Most of the derivatives of the thiosemicarbazone and their metal complexes are well known for their wide range of biologically activities that include cytotoxic, anti-bacterial, anti-fungal, anti-tumor, anti-malarial and anti-leukemic [6-11]. We are reporting synthesis, characterization, TGA and Anti-microbial properties of thisemicarbezone adduct of Fe (III) derived from B_tMNPO, AtMNPO, P_tMNPO, and TtMNPO.

MATERIALS AND METHODS

Experimental

All the chemicals used in the present study were of A. R. grade. Melting points were taken in open glass capillaries. The ligands were analyzed for Carbon, Hydrogen, Nitrogen and Sulfur were estimated on a Perkin Elmer, Series II, 2400 C H N S analyzer (CSIR, Bhavnagar, India). The infrared spectra of the ligands were recorded on a FT-IR in KBr pellets (Gujarat Laboratory, Ahmedabad, India). The ^{13}C-NMR spectra in DMSO of all ligands were recorded on a Bruker DRX - 200 FT - NMR spectrophotometer.

Synthesis of ligands

The following procedure has been used in the preparation of all ligands B_tMNPO, A_tMNPO, P_tMNPO, and TtMNPO.

The Acylthiosemicarbazones were prepared by refluxing 1: 1 mole of 4 – Acyl - 3 - methyl - 1 – [4´ - nitrophenyl] - 2 - pyrazolin - 5 - one and thiosemicarbazide hydrochloride in ethanol for two hours on water bath. The resulting mixture was allowed to stand overnight. The solid product thus obtained was collected by filtration, washed with water and air dried. The acylthiosemicarbazones were then recrystallized in ethanol.

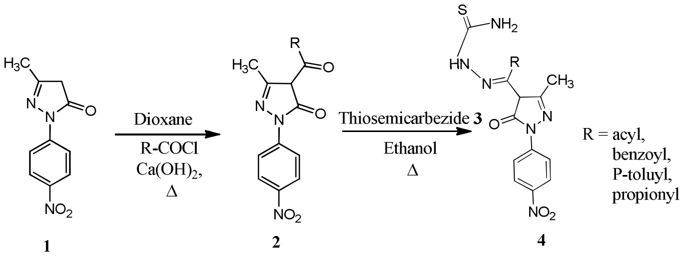
Synthesis of the Complexes:

The following general procedure was used in the synthesis of metal complexes. Metal salt was dissolved in a minimum amount of hot ethanol. The hot ethanolic ligand solution in slight excess over the metal ligand ratio 1:3 for all metal complexes was added drop wise with constant stirring. To the resulting mixture 2 gm. of sodium acetate was added and then the mixture was refluxed for 2 hours. The resulting mixture thus obtained was then concentrated to half of its original volume. The product was filtered and washed several times with hot water and finally with hot ethanol. The product was dried at 45 $^{\circ}$C. The yields of the complexes were almost quantitative.

Table 1: Physical properties of ligands

Ligands	Melting points	Color	carbon	Hydrogen	Nitrogen	Sulfur	Yields%
B$_t$MNPO	210	Brown	54.50 (54.54)	3.99 (4.04)	21.99 (21.21)	8.06 (8.08)	80
A$_t$MNPO	224	Dark Brown	46.62 (46.70)	4.22 (4.19)	25.20 (25.14)	9.45 (9.58)	76
P$_t$MNPO	217	Pale Yellow	48.31 (48.28)	4.55 (4.60)	24.21 (24.14)	9.15 (9.20)	78
T$_t$MNPO	229	Dark Brown	55.55 (55.61)	4.40 (4.39)	20.38 (20.49)	7.75 (7.80)	75

RESULTS AND DISCUSSION



FT-IR Spectra

The FT-IR Spectra of ligands B$_t$MNPO, AtMNPO, P$_t$MNPO, and TtMNPO Show very complicated spectra especially in the 1500-1725 cm^{-1} region. This may be because of the presence of C = O, C = S, - NH$_2$ and more than one C = N groups in the ligands. All the ligands show strong band in the region 1710 – 1735 cm^{-1}, due to $\upsilon_{c=o}$(Stretching) of pyrazolin ring. The absence of any bands 3500cm^{-1} for $\upsilon_{o\text{-}H}$ may indicate the existence of keto form. All these observations suggest that ligands studied in the present work may have structure – I at least in the solid state

Table 2: IR spectral data of the Ligands

Assignment	B$_t$MNPO	A$_t$MNPO	P$_t$MNPO	T$_t$MNPO
υ_{O-H} **(Coordinated Water)**	-	-	-	-
υ_{NH_2}	3450-3290 (s, s)	3450-3290 (s, s)	3450-3290 (s, s)	3450-3290 (s, s)s)
υ_{N-H}	3300 (w, s)	3305 (w, s)	3310 (w, s)	3331 (w, s)
Aryl υ_{C-H}	3078 (m, br)	3072 (m, br)	3120 (m, br)	3119 (m, br)
Saturated υ_{C-H}	2925 (w,br)	3924 (w, br)	2926 (w, br)	2922 (w, br)
$\upsilon_{C=O}$ **unsifted**	1715 (s, s)	1735 (s, s)	1710 (s, s)	1710 (s, s)
$\upsilon_{C=N}$	1628 (s, s)	1628 (s, s)	1638 (s, s)	1628 (s, s)
$\upsilon_{C=N}$ **(Pyrazolin Ring)**	1591 (s, br)	1591 (s, br)	1593 (s, br)	1591 (s, br)
υ_{O-H} **Bonding Mode of** H_2O	-	-	-	-
$\upsilon_{C=C}$ **(Phenyl Ring)**	1508 (s, s)	1497 (s, s)	1506 (s, s)	1510 (s, s)
Pyrazolin Ring Stretch	1436 (w, sh)	1425 (w, sh)	1443 (w, sh)	1437 (w, sh)
υ_{NO_2} **(4 - Nitrophenyl Ring)**	1342 (s, s)	1323 (s, s)	1331 (s, s)	1330 (s, s)
Pyrazolin Ring Breathing	1110 (w, sh)	1099 (w, sh)	1111 (w, sh)	1103 (w, sh)
υ_{N-N} **(Stretch)**	1053 (s, s)	1067 (s, s)	1064 (s, s)	1058 (s, s)
CH_3 **Rocking**	1001 (w, sh)	1012 (w, sh)	1004 (w, sh)	1015 (w, sh)
C - C_6H_5	939 (m, s)	964 (m, s)	955 (m, s)	945 (m, s)
$\upsilon_{V=O}$	-	-	-	-
υ_{O-H} **Rocking Mode of** H_2O	-	-	853 (m, s)	-
$\upsilon_{C=S}$ **Stretch**	870 (s, s)	839 (m, s)	-	852
C – CH_3 **Stretch**	852 (m, s)	794 (s, s)	794 (s, s)	839 (m, s)

^{13}C NMR Spectra

Table 3: ^{13}C NMR Spectral data of 2-pyrazoline-5-one derivatives

^{13}C	**B$_t$MNPO**	**A$_t$MNPO**	**P$_t$MNPO**	**T$_t$MNPO**
1	14.6	14.4	14.3	14.1
2	154.5	156.3	157.0	156.2
3	24.8	24.6	24.6	24.7
4	167.2	166.8	169.1	167.3
5	121.4	123.1	120.8	120.8
6	119.2	117.3	117.2	117.1
7	142.67	144.6	144.2	144.1
8	140.7	140.2	140.5	127.9
9,10	128.2	-	-	128.4
11	154.5	156.5	154.2	-
12	182.32	180.5	183.0	-
13,14,15	130.5	-	-	139.1
16,17,18	133.5	-	-	130.5

Biological activity

The antimicrobial activities of the synthesized thiosemicarbazide ligands,
(I) [B$_t$MNPO] (II) [A$_t$MNPO]
(III) [P$_t$MNPO] (IV) [T$_t$MNPO]

and their complexes of against the bacterial strains such as *Escherichia coli* and *Bacillus subtilis* and Yeast strains such as *Saccharomyees Cerevisiae* and fungal strains such as *Aspegillus niger* following the literature procedures. The effect of the ligands (I-IV) and their metal complexes in the growth media were investigated by standard microbiological parameters. Concentration of the test compounds were kept constant (500 ppm) during all the experiments. The bacterial, fungal and yeast cultures were maintained on Nutrient-agar, Potato dextrose-agar and YEDP culture-tubes (slants) respectively and were sub cultured every fortnight and stored at 0-5 ^{o}C temperature.

Most of the compounds were active against microorganisms the results are as under table no:4

Table 4: Antibacterial Activity of the Ligands (Control-DMF)

Compounds	*E-coli*	*B-Subtillis*	*Saccharonyces Cerevisiae*	*Aspegillus niger*
B$_t$MNPO	+	+	+	+
A$_t$MNPO	-	+	+	+
P$_t$MNPO	-	-	-	-
T$_t$MNPO	-	+	+	+

CONCLUSION

The new synthesized Fe (III) complexes of Thiosemicarbazide would be good active against micro-organisms, the results shown in table. **4.** This synthesis was confirmed by elemental analysis, IR and ^{13}C NMR.

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