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SYNTHESIS, CHARACTERISATION AND ANTIMICROBIAL ACTIVITY OF ZINC (II) WITH BENZOTHIAZOLE-2-ALDEHYDE THIOSEMICARBAZIDE

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ABSTRACT

Benzothiazole-2-aldehyde thiosemicarbazide(BTATSC) has been synthesized. Complexes ofbenzothiazole-2-aldehyde thiosemicarbazide with Zn (II) has been synthesized in ethanolmedium. The formation of the complexes is endothermic process. The ligand was characterizedby melting point, elemental analysis, absorption spectra and screened for antimicrobial activity while the complexes were characterized by melting point, absorption spectra. A simple & sensitivespectrophotometric method was developed for transition metal complexes of BTATSC. Theoptimum condition for complete colour development has been established. The stability constant,dissociation constant & change in free energy of Zn (II) complex with benzothiazole-2-aldehydethiosemicarbazide was determined. Composition of the metal & ligand has been determined byJob's variation & mole ratio method indicate that the M:L is 2:1. Tolerance limit of diverse ionsin the determination of Zn (II) with benzothiazole-2-aldehyde thiosemicarbazide wasinvestigated**.**

Keywords: Benzothiazole-2-aldehyde thiosemicarbazide,Zn(II), Spectrophotometry,Antimicrobial activity.

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INTRODUCTION

Zinc ions are the most common transition-metal ionsin protein crystal structures in the Protein Data Bank[1-2]and are the secondmost common metal ions overall after magnesium. Zn^{+2}ionscan play a largely catalytic role or having a largely structural role inproteins[3-6]but they aresometimes also found to have nonbiological functions ascrystalpacking mediators. The zinc finger is the mostcommonly observed zinc-binding motif in the PDB[7]. It is present in protein domains with diverse functions such as binding DNA, RNA, proteins or lipids[8].The importance of zinc for stabilization of protein loops in enzymes, zinc fingers etc., has generated new interest in the field of Zn coordination chemistry[9-15].

Thiosemicarbazones are compounds that have been studied for a considerable period of time for their biological properties.Tracesof interest date back to the beginning of the 20th century but the first reports on their medical applications began to appear in the fifties as drugs against tuberculosis and leprosy[16-17]. In the Sixties their antiviral properties were discovered and a huge amount of research was carried out that eventually led to the commercialization of methisazone, marboran, to treat smallpox[18]. In this period one of the first antitumor activity results was published[19]. Recently Triapine(3-aminopyridine-2-carboxaldehyde thiosemicarbazone) has been developed as an anticancer drug and has reached clinical phase II on several cancer types[20-21]. Thiosemicarbazones are of considerable interest because of their chemistry and potentially beneficial biological activity, such as antibacterial[22-26], antifungal[27],antiviral[28], antiamoebic[29], antimalarial [30-31]and antitumor activity [32]. Thiosemicarbazone have been frequently employed for the quantitative determination of inorganic ion[33]. Thiosemicarbazones act as Iron chelators, interferes with DNA synthesis toprevent their production led to a lot of interest in their complexation as well as theirpharmaceutical importance[34].

MATERIALS AND METHODS

All chemical and solvents used were of analytical grade.An Elico pH meter LI-610 is used for the pH measurements.An Elico UV-visible spectrophotometer model UV-SL-164 equipped

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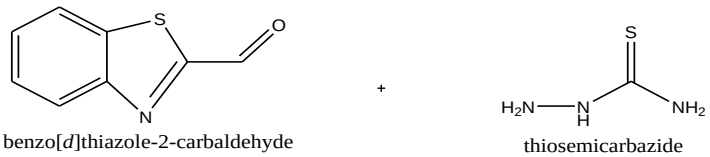
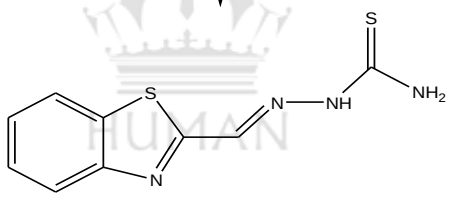
with 1 cm quartz cell used for spectrophotometric measurements taken on the instrument. Elemental analysis and antimicrobial activity was done in Laboratory approved by Central Government for AGMARK.

SYNTHESIS AND CHARACTERISATION OF BENZOTHIAZOLE-2-ALDEHYDE THIOSEMICARBAZID (BTATSC)

Synthesis of Benzothiazole-2-aldehyde thiosemicarbazide



benzo[*d*]thiazole-2-carbaldehyde thiosemicarbazide



(*E*)-1-((benzo[*d*]thiazol-2-yl)methylene)thiosemicarbazide

The crude product is crystallized in methanol. The recrystallized product has melting point is 180^0C and molecular weight by formula is 208.00.

Characterization of Benzothiazole-2-aldehyde thiosemicarbazide (BTATSC)

Absorption Spectra of (BTATSC)

Absorption Spectra of BTATSCwas recorded against a blank solution containing buffer (pH 5).Absorption spectra were recorded in the wave length range220-500nm. BTATSCshows an absorption maximum at 300 nm. At 280 nm wavelength the molar absorptivity of BTATSCis 0.99794×10^3 $L.mol^{-1}.cm^{-1}$.

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Elemental Analysis of BTATSC

The elemental analysis of BTATSCwas done in Laboratory approved by Central Government for AGMARK. Table 1.

Antimicrobial Activity ofBTATSC

Antimicrobial Activity of BTATSChas been done in the Laboratory approved by Central Government through AGMARK. Table 2.

Characterization ofZn (II)-BTATSC

Absorption Spectra of Zn (II)-BTATSCwas recorded against a blank solution containing buffer (pH 5). Absorption spectra were recorded in the wave length range 220-500nm. The complex shows an absorption maximum at 280 nm

Effect of Reagent concentration

Effect of Reagent concentration was studied by taking varying amount of reagent and fixed amount of transition metal.

Validity of Beer's Law and Composition of Complex

For the study of Beer's law the solutions were prepared which containing different amounts ofZn (II), same amount of BTATSCand 1 ml of pH 5.The composition of the Zn (II)-BTATSC complex is found to be 1:2. It was determined by studying Job's method. The ratio of metal ion to ligand molecule in the coloured complex was found to be 1:2.

Physico-chemical Characteristic of Zn (II) - BTATSC

Physico-chemical and Analytical Characteristic of transition metal complex of BTATSC was studied and given in Table 4 and Tolerance limit of diverse ions in the determination of BTATSC shown in Table No. 5.

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RESULTS AND DISCUSSION

Table No. 1. Elemental Analysis of Benzothiazole-2-aldehyde thiosemicarbazide

Sr.No.	Chemical Analysis	Percentage Found	Percentage Expected
1)	Carbon	51.92 %	55.47 %
2)	Hydrogen	03.85 %	04.16 %
3)	Sulphur	30.77 %	31.80 %
4)	Nitrogen	13.46 %	12.95 %

Table No. 2.Antimicrobial Activityof Benzothiazole-2-aldehyde thiosemicarbazide

Sr.No.	Antimicrobial	Activity
1)	*KlebsiellaPneumoniae*	Nil
2)	*VibriaeCholerease*	Nil
3)	*Bacillus Megaterium*	Nil
4)	*Salmonallatyphi*	Nil
5)	*ShigellaFlexneri*	Nil

Table3.Experimental Result & Physical data of Benzothiazole-2-aldehyde thiosemicarbazide& its Complexes

Code No	Compound M.P. (^{0}C)	Colour	Molecular Weight by Formula gm/mole	Yield
BTATSC	160^{0}C	Yellowish	208.00	82 %
Zn (II)-BTATSC	178^{0}C	Blackish Yellow	273.38	91 %

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Table. 4. Physico-Chemical and Analytical Characteristic ofZinc(II) complex of Benzothiazole-2-aldehyde thiosemicarbazide

Sr.No.	Characteristics	Zn (II)-BTATSC
1)	Absorption Spectra	280 nm
2)	Molar absorptivity	0.99794×10^3 $L.mol^{-1}.cm^{-1}$
3)	pH range (optimum)	5.0
4)	Reagent required for maximum complexation	0.200ml
5)	pKa	2.99287×10^8
6)	Beer's law validity range (ppm)	6.0 ppm
7)	Composition of complex (M : L)	1:2
8)	Stability Constant	5.3745×10^7
9)	Dissociation Constant	2.982630×10^{-8}
10)	Change in free energy	-37.16535KJ/mole
11)	Sandell's Sensitivity (µg /cm^2)	0.004241 µg /cm

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Table No.5.Tolerance limit of diverse ions of Zinc(II) Complex of Benzothiazole-2-aldehyde thiosemicarbazide

Sr. No.	Metal ion	Salt	Interference
			Zn(II)- BTATSC
1)	Mg (II)	$MgSO_4$	28
2)	Ca(II)	$CaCl_2.2H_2O$	158
3)	Cd (II)	$CdCl_2$	28
4)	Mn (II)	$MnCl_2$	10
5)	Co (II)	$CoSO_4$	40
6)	Ce (IV)	$Ce(SO_4)_2$	Interferes
7)	Ba (II)	$BaCl_2$	Interfere
8)	Cr (III)	$K_2Cr_2O_7$	09
9)	Hg (II)	$HgCl_2$	26
10)	Ti (V)	K-titanyl oxalate	39
11)	Ni (II)	$NiCl_2$	41
12)	Sn (II)	$SnCl_2$	02
13)	Na (I)	NaCl	55
14)	Pb (II)	$PbSo_4$	Interfere
15)	V (v)	V_2O_5	89
16)	Zn (II)	$ZnSO_4$	25
17)	Al (III)	$AlCl_3$	41
18)	Pd (II)	$PdCl_2$	Interfere
19)	K(II)	KCl	73

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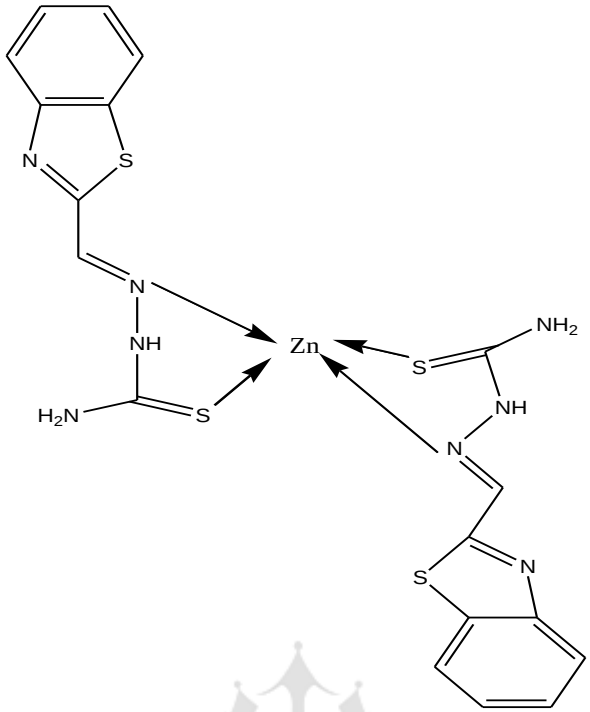
Structure of Zn (II)- BTATSC



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