Synthesis and Characterization U (II) with O-methylzimtaldehyde Thiosemicarbazones [O-MeZimTSC] and its Biological Activity

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Summary

Synthesis of O-MeZimTSC, Melting Point,Elemental analysis, X-RD, Effect of diverse ion and Antimicrobial activity are studied. A simple ,sensitive and specific spectrophotometric method for the determination of U(II) is developed based on the colour reaction between uranium(II) and O-Methylzimtaldehyde thiosemicarbazone[O-MeZimTSC]. X-ray diffraction pattern with powder X-ray diffractiom was studied. . NMR , Effect of the diverse ion have been studied respectively. Stability constant of the complex,Dissociation constant and Change in free energy are determined.. Composition of the metal and ligand has been determined by Job's variation and mole ratio methods. The optimum conditions for complete colour development have been established by studying parameters like effect of medium. Reagent concentration, time period have been studied. Application of this O-MeZimTSC for antimicrobial activity have been performed .

Keywords—Uranium(II),O-Methylzimtaldehyde thiosemicarbazide, Spectrophotometry, Antimicrobial samples.

Introduction

Uranium is very heavy metal. The uranyl UO_2^{2+} is stable in acid solution while the diuranate anion $U_2O_7^{2-}$ is stable in alkali medium.It is mined as oxide ores uranite and Pitchblende. It is used in the production of energy in nuclear reactor. Uranium gives coloured products with many organic reagents.In different environmental samples,several techniques are developed for the determination of Uranium,likeSpectroscopy[2],

Neutronactivation[3,4]Spectrophotometry[5],Molecular fluorescence spectroscopy[6], Complexiметric titration[7], Pulse polorography[8-9],Gas chromatography[10-12], Radioactivation[13-14]and inductively coupled plasma emission spectrometry[15].Mixed ligand complexes ofuranium with sudan violet and selected ligands have been reported[16].

Thiosemicarbazones are important organic analytical reagents for the determination of metal ions in microgram quantities. They form coloured complexes with many metal ions and act as good chelating agents. In addition to the analytical utility[17-23], these reagents are found to be biologically active. These compounds contain an azomethine nitrogen atom.Further the metal complexes formed with these reagents are of great medicinal value in the treatment of diseases like influenza, protozon, smallpox and certain kinds of tumours. These reagents inhibit tumour growth and increase the activity of some drugs.In the treatment of cancer the active species is a metal chelate of thiosemicarbazones. Complexes of transition metal with thiosemicarbazides and their Schiff base have been studied extensivel both of the hydrogen atoms of N_4 atom are substituted with alkyl or aryl groups have not been investigated in detail[24]. A large number of thiosemicarbazides have been found to have good antibacterial[25], antifungal[26-27], herbicidal and antiacetylcholinesterase activities. The pharmacological importance of metal complex with heterocyclic thiosemicarbazones[28].

Materials and Methods

An Elico UV- visible spectrophotometer model UV-SL 164 equipped with 1 cm quartz cell is used for spectrophotometric measurements. An Elico pH meter LI-610 is used for pH measurements. The chemicals used are of analytical reagent grade. Perkins Elmer 221 IR spectrophotometer using KBr pellets techniques is used for IR studies.X-RD was taken on PW 3710 diffractometer using CuK2 radiation has been taken on the instrument BRUKER AC 300F NMR spectrophotometer 300HZ with $CDCl_3$ solvent. Elemental analysis and antimicrobial activity was done in laboratory approved by Central Government for AGMARK.

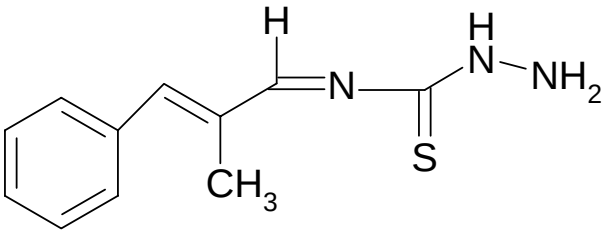
Synthesis and Characterisation of O-MeZimTSC

Synthesis of O-MeZimTSC

O-Methylzimtaldehyde thiosemicarbazones (O-MeZinTSC) is prepared by conducing

O-Methylzimtaldehyde with thiosemicarbazide in methanol medium for three hours .

The structure of the reagent is



The crude product is crystallized in methanol. The recrystallized product has melting point is 210-215^{o}C and molecular weight by formula is 219.00.

Characterisation of O-MeZimtSC

Elemental Analysis of O-MeZimTSC

The elemental analysis of O-MeZimTSC was done in laboratory approved by Central Government for AGMARK. It shows the result of elemental analysis in Table 1.

X-RD of O-MeZimTSC

X-RD spectra of O-MeZimTSC was taken on PW 3710 diffractometer using CuK2 radiation (Y= 1.54056A^0) The X-RD diffraction of O-MeZimTSC was recorded at angle 20 from 10.905 to 86.780. The data of X-ray diffraction of O-MeZimTSC were presented in Table 2. And X-ray spectrum in fig 1. For the determination of structure Hesse-Lipson procedure is used[30]**.**

Absorption Spectra of O-MeZimTSC

The absorption spectra of O-MeZimTSC was recorded against a blank solution containing buffer (pH=5) and is shown in fig 2. Absorption spectra was recorded in the wave length range 220-520 nm. The complex shows an absorption maximum at 290 nm. At 290 nm wavelength the molar absorptivity of O-MeZimTSC is 3 .6488x10^3L. mol^{-1} cm^{-1}.

Infrared spectra of O-MeZimTSC

IR spectra of O-MeZimTSC was taken in the range of 4000 cm^{-1} to 200 cm^{-1} on Perkin Elmer 221 IR Spectrophotometer using KB r pellet technique. The characteristic bands observed are as in Table 3. Fig 3 shows IR spectra of O-MeZimTSC.

NMR Spectra of O-MeZimTSC

NMR spectra of O-MeZimTSc has been taken from Government of Central Instrumentation laboratory. Instrument used BRUKER AC 300F NMR spectrophotometer 300HZ with $CDCl_3$ solvent. The characteristic chemical shift and the type of proton given in Table 4 .The NMR spectra of O-MeZimTSC is as shown in fig 4. From the NMR spectra and the table it is observed that the aromatic proton tallies with the structure of O-MeZimTSC.

Antimicrobial Activity of O-MeZimTSC

Antimicrobial Activity of O-MeZimTSC has done in the laboratory approved by Central Government through AGMARK .The result are noted in Table 5.

Physico-chemical Characteristic of U(II) –O-MeZimTSC

Physico- chemical and Analytical Characteristic of U(II) –O-MeZimTSC was studied and given in Table 6.

Table No. 1. Elemental Analysis of O-MeZimTSC

Sr. No.	Chemical Analysis	Percentage Found	Percentage Expected
1)	Carbon	58.82	60.23
2)	Hydrogen	04.36	04.93
3)	Sulphur	13.58	14.62
4)	Nitrogen	17.26	19.16

Table NO.2 XRD for O-MeZimTSC (Powder method)

2	hkl	Sin^2 Observed	Sin^2 Calculated	d(A^0) Observed	d(A^0) Calculated
10.450	100	0.01175	0.00810	7.1035	8.5569
13.860	110	0.01454	0.01758	6.3841	5.8048
15.320	110	0.01774	0.01758	5.7788	5.8048
17.970	111	0.02436	0.03097	4.9321	4.3578
18.895	111	0.02692	0.03097	4.6927	4.3578
19.025	111	0.02742	0.03097	4.6609	4.3578
19.765	111	0.02944	0.03097	4.4881	4.3578
20.745	200	0.0324	0.03246	4.2782	4.2782
21.750	200	0.0355	0.03246	4.0828	4.2782
23.625	210	0.0419	0.04190	3.7628	3.7622
24.545	210	0.0463	0.04190	3.6238	3.7622
26.615	211	0.0529	0.05555	3.3465	3.2676
27.270	211	0.0555	0.05555	3.2676	3.2676
27.935	211	0.0582	0.05555	3.1913	3.2676
30.915	220	0.0710	0.07039	2.8901	2.9102
35.800	310	0.0944	0.07039	2.5061	2.6826

a=8.55639 b=7.90075 c=6.5938

Structure of Powder Crystal

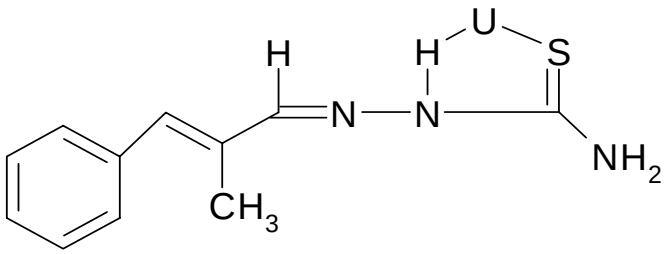


Table No.3 Infrared spectra of O-MeZimTSC

Sr.No.	Frequency Wavenumber	Expected Element
1)	710 780 790 850	Strong CH_2
2)	1120 1190	C=S, Strong
3)	1190 1250 1330	Strong CS, NH
4)	1560	NH. Medium

5)	1690	NH. Medium, NH_2
6)	1850 1980	Benzene ring
7)	2030	N=C=, Strong
8)	2260	N=C= , Strong.
9)	2620 2800 2860 2900 3080	-C- , H, Strong
10)	2860	Intermolecular H bonding
11)	2900	O-H
12)	3360 3760	NH_2=NH , Medium.
13)	3800	Variable free energy.

Table No. 4 NMR Data of O-MeZimTSC

Sr.No.	Types of Proton	Groups	Chemical Shift (ppm)
1)	Aromatic	Ar-H	8.0001 7.7681 7.3864 7.3722 7.3372 7.3236 7.3090 7.2949 7.2793 7.2624 7.2151 7.1694 7.1572
		Allyic Proton CH	6.9743 6.7970 6.5796
2)	Benzylic	Ar-OH	2.0659 2.1147
3)	Aldehydic	R-CHO	10.206

Table No. 5 Antimicrobial activity of O-MeZimTSC

Sr.No.	Antimicrobial	Activity
1)	Klebsiella Pneumoniae	Nil
2)	Vi briae Cholerease	Nil
3)	Salmonalla typhi	Nil

Table No. 6 Physico-Chemical and Analytical Characteristic of U(II)-O-MeZimTSC

Sr.No.	Characteristics	Results
1)	Absorption Spectra	350 nm
2)	Molar absorptivity (L. mole^{-1}.cm^{-1})	1.0667x10^{5}
3)	pH range (optimum)	7.0
4)	Reagent required for maximum complexation	2.0 ml
5)	pKa	6.884x10^{8}
6)	Beer's Law validity range (ppm)	0.66-6.09
7)	Composition of complex (M:L) obtained in Job's and Molar ratio method	1:1
8)	Stability Constant	2.65041x10^{7}
9)	Dissociation Constant	3.773x10^{-8}
10)	Change in free energy	-42.35 KJ/mole
11	Sandell's sensitivity (mg/cm^{-2})	0.00223145 µg. cm^{-2}

Table No. 7 Tolerance limit of diverse ions in the determination of Uranium (II)

Sr. No.	Metal ion	Sait	Interference U(II)
1)	Cr(II)	$K_2Cr_2O_7$	59.00
2)	Ti (II)	$TiCl_2$	55.00
3)	Fe(III)	Amm. Ferrous Sulphate	31.47
4)	Citrate	Citric Acid	59.00
5)	Sn (II)	$SnCl_2$	78.67
6)	Pb (II)	$Pb(NO_3)_2$	90.48
7)	Mg (II)	$MgCl_2$	70.81
8)	Co(II)	$CoSO_4$	82.61
9)	Hg(II)	$HgCl_2$	90.48
10)	CH_3COO^-	CH_3COONa	None
11)	SCN	NH_4SCN	None

Fig 1. X-RD Spectra of O-MeZimTSC

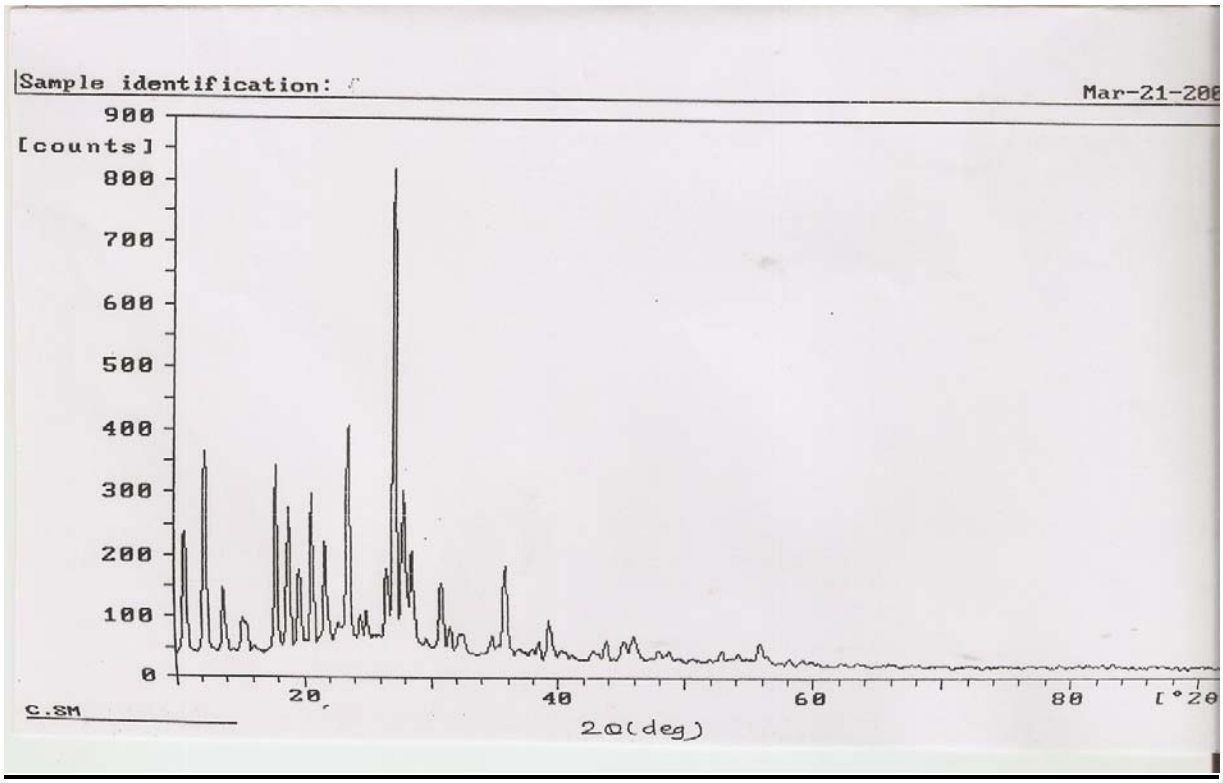


Fig 2. Absorption Spectra of O-MeZimTSC

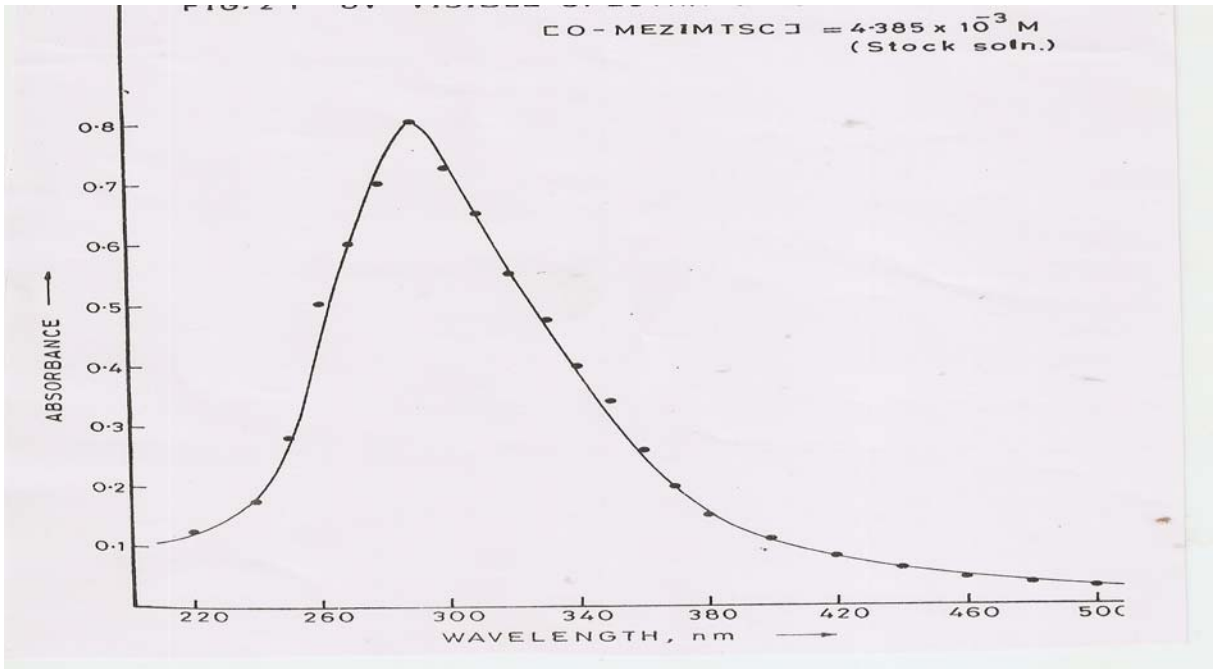


Fig 3. IR Spectra of O-MeZimTSC

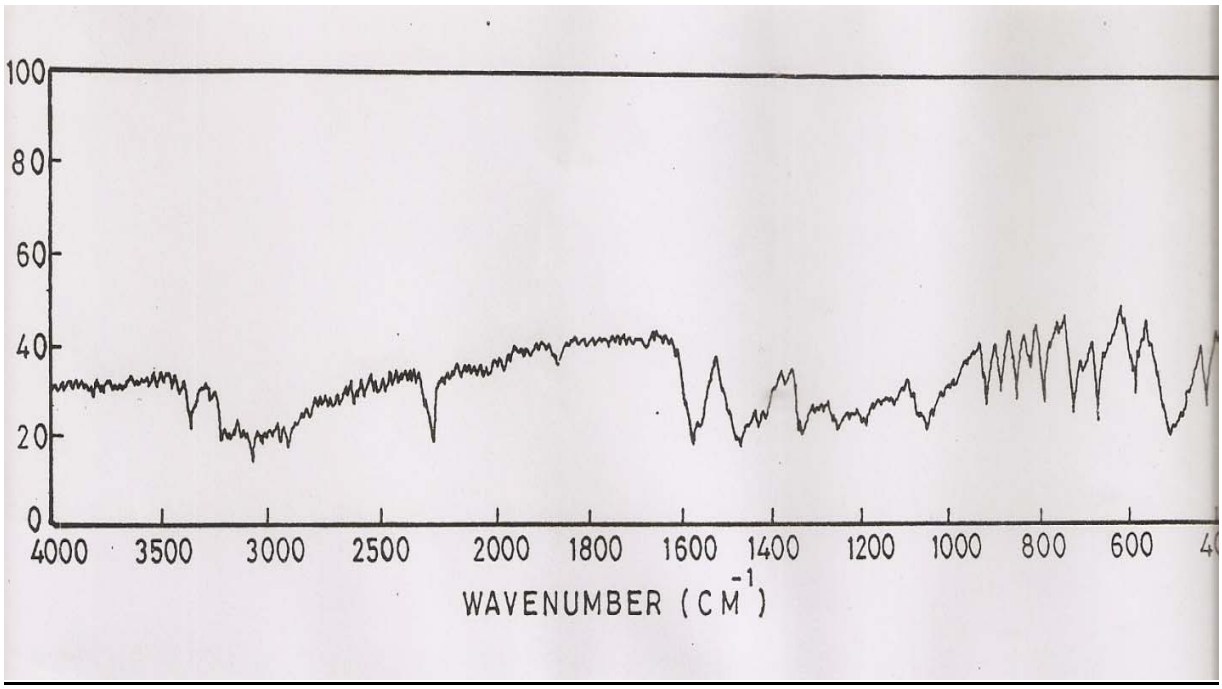
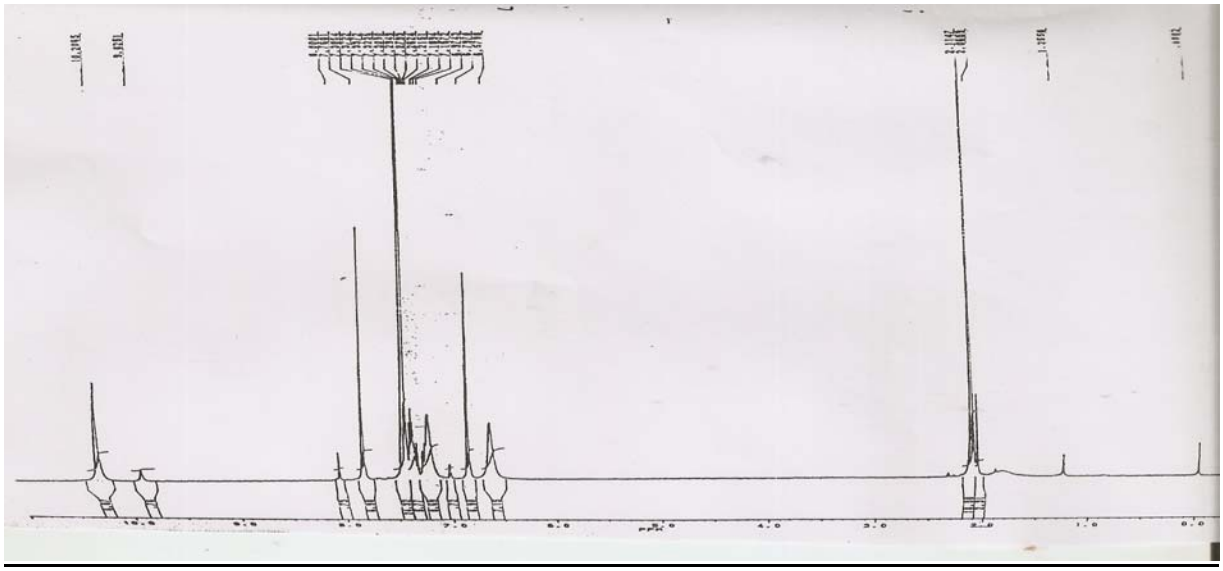


Fig 4. NMR Spectra of O-MeZimTSC



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