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Synthesis and Photometric Determination of Palladium (II) with Furfuraldehyde-2-Salisaldehyde Thiosemicarbazone (F2STSC) and its Biological Activities

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ABSTRACT

Furfuraldehyde-2-salicylaldehyde thiosemicarbazide (F2STSC) has been synthesized. 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 Pd (II) is developed based on the color reaction between palladium (II) and Furfuraldehyde-2-salicylaldehyde thiosemicarbazide (F_2STSC). X-ray diffraction pattern with powder X-ray diffraction was studied. NMR, the effect of the diverse ion have been studied respectively. The stability constant of the complex, Dissociation constant, and Change in free energy is determined. The composition of the metal and ligand has been determined by Job's variation and mole ratio method. The optimum condition for complete color development has been established by studying parameters like an effect of the medium, Reagent concentration, time period have been studied. Application of this F2STSC for antimicrobial activity has been performed.

INTRODUCTION

Thiosemicarbazones are an important organic analytical reagent for the determination of metal ions. They found color complexes with many metal ions and act as good chelating agents. In addition to analytical utility[1-7], these reagents are found to be biologically active A large number of thiosemicarbazide have been found to have good antibacterial[8], antifungal[9,10]. The pharmacological importance of metal complex with heterocyclic thiosemicarbazones[11]. Thiosemicarbazone derivatives have demonstrated the wide range of biological activity viz antimicrobial[12-17], antitumour[18-19], sodium channel blocker[20], anticancer[21-22], antitubercular[23], antiviral[24]. Thiosemicarbazones has many variable bonding modes promising biological implications and structural diversity[25]. Thiosemicarbazone of transition metal complexes has potentially chemotherapeutic properties of both ligands and complexes as antitumor and antibacterial agents[26]. Pharmacological potential of thiosemicarbazone as an antitumor agent is one of the more promising areas of research. Thiosemicarbazone complexes differ from the free ligand with respect to their biological properties. Thiosemicarbazones reduced by complexing to the metal cation. Synthesis of transition metal complexes with thiosemicarbazones. Recently triapine (3-aminopyridine-2-carboxaldehyde thiosemicarbazone) has been developed as a several cancer types[27]. Study of antiproliferative activity of Pd(II) complex of 8 ethyl-2-hydroxy tricycle tridecane-13-one-thiosemicarbazone has been studied[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 studied. X-RD was taken on PW 3710 diffractometer using CuK_2 radiation has been taken on the instrument BRUKER AC 300 F NMR spectrophotometer 300 HZ with $CDCl_3$ solvent. Elemental analysis and antimicrobial activity were done in the laboratory approved by Central Government for AGMARK.

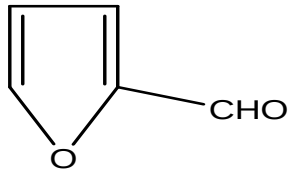
Synthesis and Characterisation of F2STSC

Synthesis of F2STSC

Furfuraldehyde-2-salicylaldehyde thiosemicarbazide (F2STSC) was synthesized in two steps.

Step I: Synthesis of Furfuraldehyde thiosemicarbazone

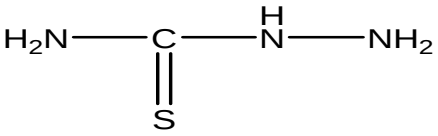
Furfuraldehyde thiosemicarbazone was synthesized by refluxing[222] 2 g of furfuraldehyde and 2 g of thiosemicarbazide in 100 ml round bottom flask in 50 ml alcohol for one and half hour. After one and half hour brownish product was obtained. The product was filtered and recrystallized in alcohol. Obtained dried compound is furfuraldehyde thiosemicarbazone



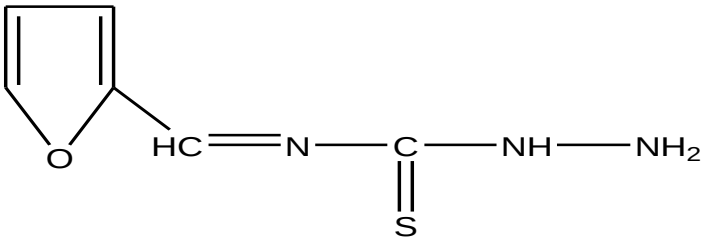
furan-2-carbaldehyde

Furfuraldehyde

+



thiosemicarbazide



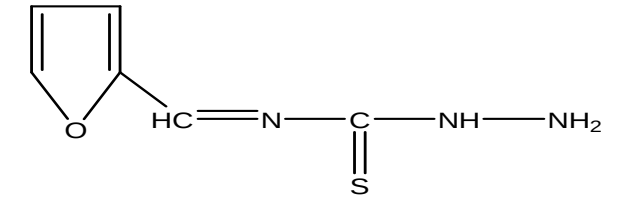
4-((furan-2-yl)methylene)thiosemicarbazide

Furfuraldehyde thiosemicarbazide

Brownish Product

Step II Synthesis of furfuraldehyde-2-salicylaldehyde thiosemicarbazone (F2STSC).

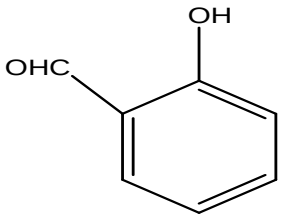
Furfuraldehyde thiosemicarbazone was refluxed with 2 gm of salicylaldehyde for 1 hrs. After the one-hour brownish yellow product was obtained. It was filtered and washed with alcohol and dried. The final product was furfuraldehyde-2-salicylaldehyde thiosemicarbazone (F2STSC).



4-((furan-2-yl)methylene)thiosemicarbazide

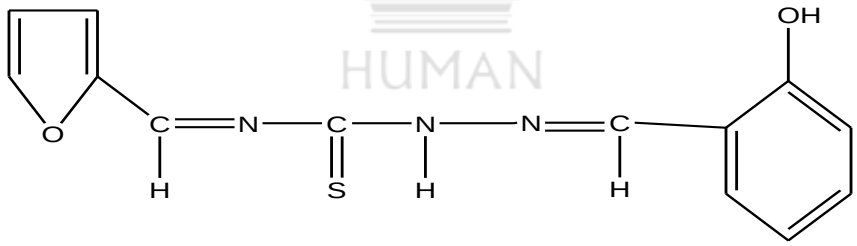
Furfuraldehyde thiosemicarbazide

+



2-hydroxybenzaldehyde

Salisaldehyde



Furfuraldehyde-2-salisaldehyde thiosemicarbazone

(F2STSC)

Brownish Yellow Product

Melting point 273.00 and its molecular weight is 273.00

Characterisation of F2STSC

Elemental Analysis of F2STSC

The elemental analysis of F2STSC was done in the laboratory approved by central Government for AGMARK. It shows the result of elemental analysis in **Table 1**

X-RD of F2STSC

X-RD spectra of F2STSC was taken on PW 3710 diffractometer using CuK_2 radiation (Y= 1.54056 A^0). The X-RD diffraction of F2STSC was recorded at angle 20 from 10.905 to 56.780. The data of X-ray diffraction of F2STSC were presented in **Table 2** and X-ray spectrum in Fig 1. For the determination of structure Hesse-Lipson procedure is used.

Absorption Spectra of F2STSC

The absorption spectra of F2STSC was recorded against a blank solution containing the buffer (pH=5) and is shown in **Fig 2**. Absorption spectra were recorded in the wavelength range 340-510 nm. The complex shows an absorption maximum at 290 nm. At 290 nm wavelength the molar absorptivity of F2STSC is 3.0811×10^3 $L.mol^{-1}.cm^{-1}$.

Infrared Spectra of F2STSC

IR Spectra of F2STSC was taken in the range of 4000 cm^{-1} to 200 cm^{-1} on Perkin Elmer 221 IR Spectrophotometer using KBr pellet technique. The characteristic bands observed are as in Table 3**. Fig. 3** shows IR spectra of F2STSC

NMR Spectra of F2STSC

NMR spectra of F2STSC have been taken from Government of Central Instrumentation laboratory. The instrument used BRUKER AC 300F NMR Spectrophotometer 300 HZ with the $CDCl_2$ solution. The characteristic chemical shift and the type of proton given in **Table 4**. The NMR spectra of F2STSC 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 F2STSC**.**

Antimicrobial Activity of F2STSC

Antimicrobial activity of F2STSC has been done in the laboratory approved by Central Government through AGMARK. The result is noted in **Table 5**

Physico-chemical Characteristic of F2STSC

Physico-chemical and analytical characteristic of F2STSC was studied and given in **Table 6**

Table 1 Elemental Analysis of F2STSC

Sr. No.	Chemical Analysis	Percentage Found	Percentage Expected
1	Carbon	47.82	57.12
2	Hydrogen	05.21	04.05
3	Sulphur	09.22	11.70
4	Nitrogen	17.16	15.38
5	Oxygen	20.59	11.73

Table 2 XRD for F2STSC (Powder Method)

2 θ	**hkl**	**Sin^2 Observed**	**Sin^2 Calculated**	**d(A^0) Observed**	**d(A^0) Calculated**
10.160	----	0.027840	----	8.6992	----
12.545	110	0.011937	0.19229	7.0502	5.5555
16.010	110	0.019393	0.19223	5.5313	5.5555
16.150	111	0.019731	0.02752	5.4836	4.6429
18.930	200	0.027032	0.02704	4.6841	4.6840
20.005	200	0.030168	0.02704	4.4348	4.6840
20.535	200	0.031770	0.02704	4.3215	4.6840
22.930	210	0.039509	0.03950	3.8753	3.8752
23.530	210	0.041574	0.03950	3.7778	3.8752
25.260	211	0.047809	0.04781	3.5228	3.5228
26.145	211	0.051158	0.04781	3.4056	3.5228
26.620	----	0.053004	----	3.3459	----
27.460	----	0.056337	----	3.2454	----
29.845	220	0.663124	0.07690	2.9912	2.7775
32.705	310	0.079268	0.07331	2.7359	2.8448
32.875	310	0.080071	0.07331	2.7221	2.8448

a = 9.3682 **b= 6.8990** **c = 8.250**

Table 3 Infrared Spectra of F2STSC

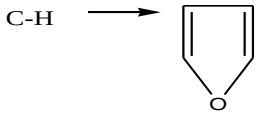
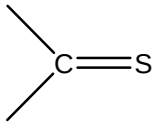
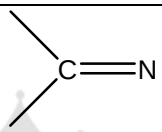
Sr. No.	Frequency Wavenumber	Expected Element
1	740	4 adjacent aromatic 
2	800	
3	820	
4	920	
5	1030	
6	1230	CS, -NH
7	1340	
8	1360	
9	1430	
10	1460	 Conjugated cyclic
11	1510	Benzene
12	1580	
13	1580	Unsaturated comp. C=N
14	1660	
15	1660	C=S, Stretch, Sulphur compounds
16	1850	
17	2000	N=C=N
18	2280	
19	2505	
20	3700	Free OH, O-H stretch

Table 4 NMR Data of F2STSC

Sr. No.	Types of Proton	Groups	Chemical Shift (ppm)
1	Aromatic proton	Ar-H	7.6919 7.8638 7.5502 7.4638 7.4186 7.3932 7.3256 7.2754 7.2501 7.2247 6.9325 6.9050 6.8799 6.8549
2	Primary proton	R-CH_3	8.3036
3	Aldehyde	R-CHO	9.6055
4	Secondary proton	R_2CH_2	1.2555
5	Alcohols	HC-OH	3.1239

Table 5 Antimicrobial Activity of F2STSC

Sr. No.	Antimicrobial	Activity
1	*Klebisiella pneumoniae*	Nil
2	*Vibriae cholerease*	Nil
3	*Salmonalla typhi*	Nil

Table 6 Physico-chemical and Analytical Characteristic of F2STSC

Sr. No.	Characteristics	Results
1	Absorption spectra	430 nm
2	Molar absorptivity	3.0811×10^3 L. $mol^{-1}.cm^{-1}$
3	pH range (optimum)	5
4	Reagent required for maximum complexation	1.0 ml
5	pKa	7.844×10^8
6	Beers Law validity range (ppm)	2.3537×10^{-5} M
7	Composition of complex (M:L) obtained in Job's and Mole ratio method	1:2
8	Stability constant	4.411894×10^7
9	Dissociation constant	2.2666×10^{-8}
	Degree of dissociation	0.060869
10	Change in free energy	-43.61 KJ/mol
11	Sandell's sensitivity (mg/cm^{-2})	0.034539 $\mu g.cm^{-2}$

Table 7 Tolerance limit of diverse ions in the determination of F2STSC-Pd (II)

Sr. No.	Metal ion	Salt	Interference F2STSC-Pd (II)
1	Fe (III)	$FeCl_3$	882.5
2	Co (II)	Co (SO_4)	127.5
3	Sn (II)	$SnCl_2$	125.0
4	Mg (II)	$MgCl_2$	127.5
5	Ni (II)	$NiSO_4.2H_2O$	None
6	Salicylate	Salicyclic acid	None
7	SCN	NH_4SCN	None

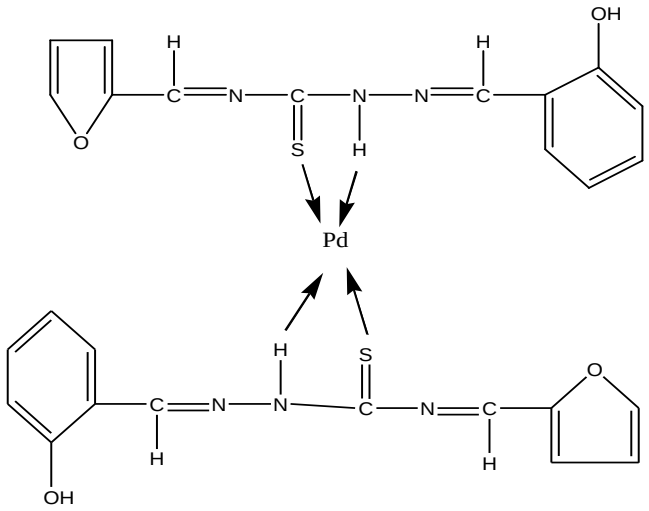


Fig 1 X-RD Spectra of F2STSC

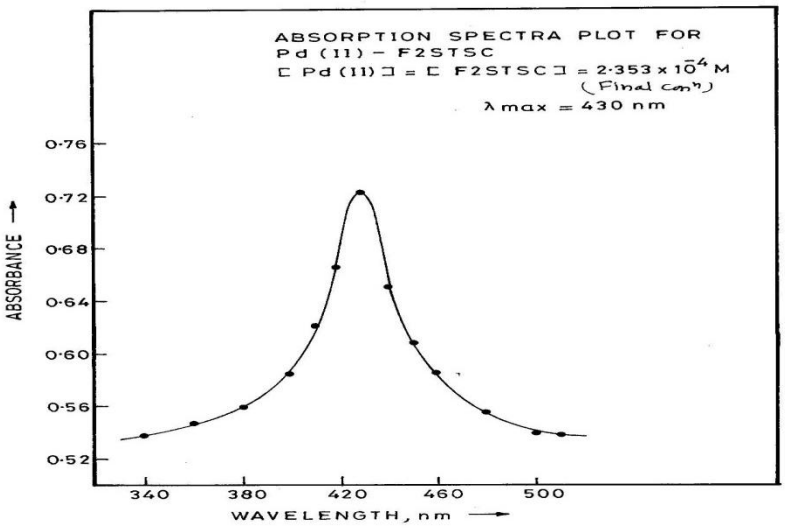


Fig 2 Absorption Spectra of F2STSC

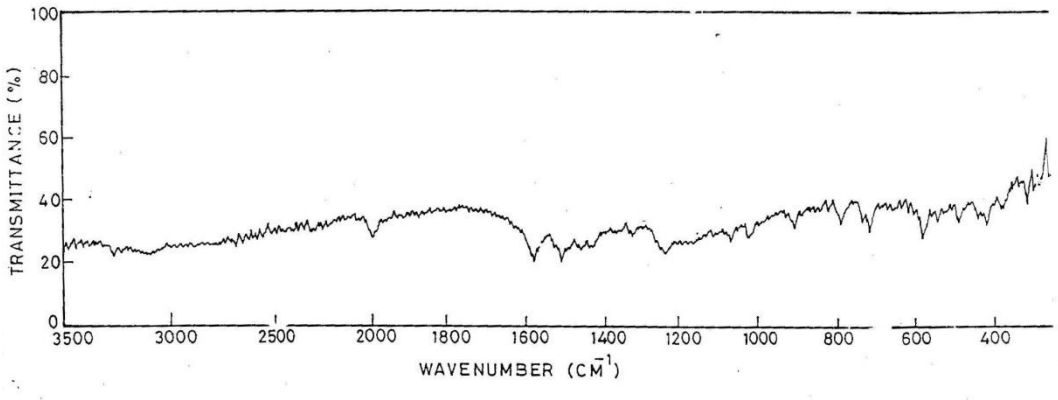


Fig 3 IR Spectra of F2STSC

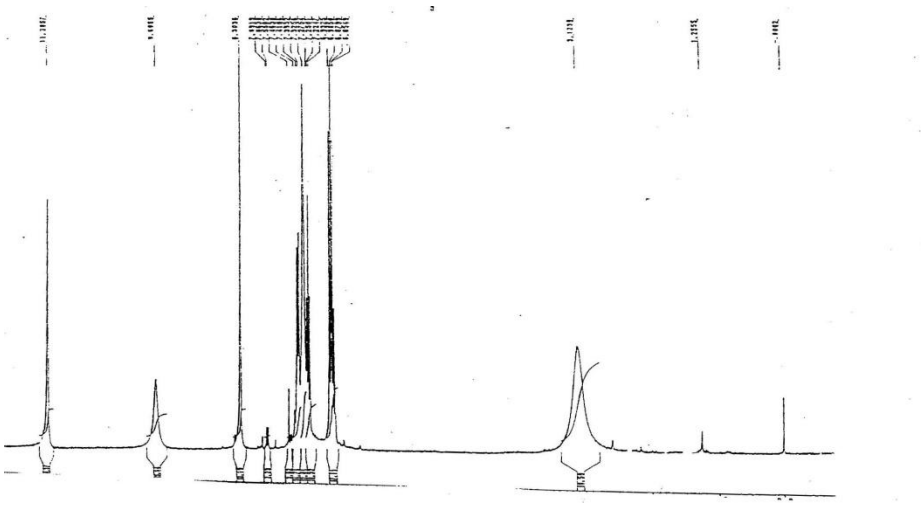


Fig 4 NMR Spectra of F2STSC

CONCLUSION

Absorption of F2STSC at 430nm. Its molar extinction coefficient is 3.0811 x 10^3 L.mol^{-1} cm^{-1}. F2STSC is suitable for the determination of Pd(II) metal. The composition of the complex (M:L) obtained in Job's and Mole ratio method 1:2. Sandell's sensitivity is 0.034539µg.cm^{-2} .

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