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Infrared Absorption Studies on Some New Potential Antimicrobial Diazotization Product of 4-aryl-Thiosemicarbazides

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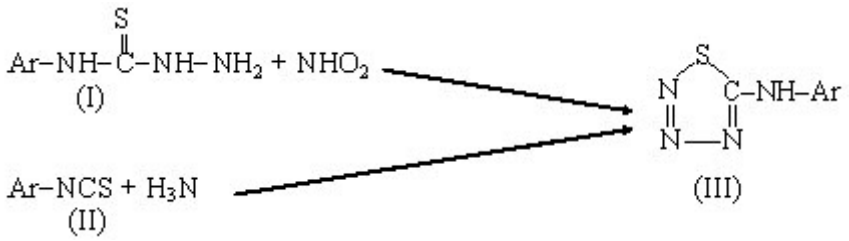
ABSTRACT

The infrared absorption spectra of 4-aryl-3-thiosemicarbazides & 5-arylamino-1,2,3,4-thiatriazoles have been studied and structural assignments of importance to these systems made or suggested. Important conclusions drawn from the spectral data are : there in no suggestion of any thiol-thione tautomerism for the solid 4-aryl-3-thiosemicarbazides and that the thione structure predominates for these substances and that the diazotization products of the 4-arylthiosemicrabazides yield, 1,2,34-thiatriazole rather than the isomeric open chain thiocarbamylazides. The C=S, -N-C=S and cyclic -N-N=N- configurational assignments are discussed compounds 5-p-tolyl and 5-o-anisyl amino-1,2,3,4-thiatriazoles have shown antifungal activity against two fungi.

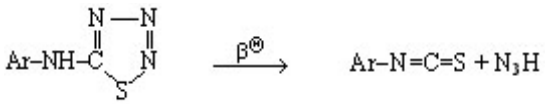
Key words: IR spectra and antifungal activity of 5-p-tolyl/ and 5-o-anisylamino 1,2,3,4-thiatraziles.

INTRODUCTION

In a previous communication Lieber, Pillai and Hites[1] reported that the reaction of 4-aryl-thiosemicarbazides[2-3] (I) with nitrous acid as well as the reaction of aryl-isothiocynates (II) with hydrazoic acid leads to the identical 5-arylamino-1,2,3,4-thiatriazoles (III).



The 5-(substituted) amino-1,2,3,4-thiatriazoles (III), or treatment with base (NaOH) gives (II) and NH_3 as well as another produce also.



EXPERIMENTAL

IR Spectroscopic Studies

The IR spectra were recorded on a Perkin-Elmer single beam spectrometer, Model 12C. with sodium chloride prism. The position of the absorption maxima are listed in Table-1 & 2 with the intensities being indicated by the following symbols : s=strong, m=medium, w=weak; vw=very weak. The compounds studied were those reported upon in the previous communication. The spectra were taken in Nujolmulls & KBr. Disc.

RESULTS AND DISCUSSION

4-substituted thiosemicarbazides

All the important absorption bands of the 4-substituted thiosemicarbazides in the region 1640-780 cm^{-1}. Summarized in Table-1. No SH band was found in these compounds in the region 2600-2500 cm^{-1}, the range in which the SH stretching vibrations are most likely to appear. Thus clearly shows that there is no thiol-thione tautomerism in these compounds in the solid state.

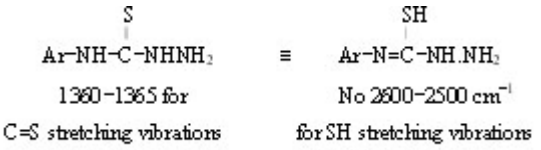
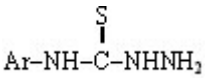


Table 1: Infrared spectra of 4-aryl-thiosemicarbazides



S. No.	Ar group	-NH (cm^{-1})	N-C=S (cm^{-1})	C=S (cm^{-1})	NH (cm^{-1})	NH_2 (cm^{-1})
1	Phenyl	1634	1522	1361	1065	960
2	o-Anisyl	1630	1520	1350	1060	955
3	p-Tolyl	1638	1525	1365	1070	970
4	1-Pyridyl	1530	1520	1360	1060	955
5	Cyclohexyl	1640	1527	1368	1068	965
6	α-Naphthyl	1632	1520	1300	6062	955
7	p-Hydroxyphenyl	1630	1520	1360	1068	965
8	p-chlorophenyl	1632	1520	1300	1058	958

Hence IR confirms the presence of C=S instead of C-SH groups.

A similar conclusion has been drown by Bogomolov & Co-workers. All the compounds studied showed N-H stretching models of vibrations. In general, the important infrared absorption frequencies of the 4-substituted thiosemicarbazides can be summarized in Table-1. The bonds due to hydrazino, $-NHNH_2$, Portion of the structure have been assigned on the basis of studies presented by Randall & Lieber. The 4-aryl-thiosemicarbazides show weak absorption at 1634 cm^{-1}. In addition to the absorption bonds discussed above, bands due to other functional group and substituted aromatic rings were also observed.

5-(substituted) amino-1,2,3,4-thiatriazoles

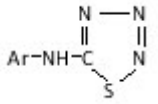
The most significant observation arising out of this study of the infrared absorption spectra of a series of eight 5-arylmaino-1,2,3,4-thiatrizoles, summarized in Table-2. In spectra there is absence

of an absorption band in the region 2170-2080 cm^{-1}, this rules out the presence of Azido group, supporting structure No. (III).

In addition to above other common absorbance are as below-

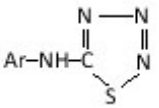
1260 cm^{-1} : cyclic -N-N=N- stretching vibrations.
1195, 1140, 1060 cm^{-1} : Aromatic C-H planar bending vibrations.
985, 955, 870 cm-1 : Aromatic C-H out of Plane bending vibrations.
800 cm^{-1} : C-Cl stretching vibrations.

Table 2: 5-Arylamino-1,2,3,4-thiatriazoles



S. No.	Ar group	-NH (cm^{-1})	C=N (cm^{-1})	Aromatic C=C (cm^{-1})	N=N (cm^{-1})	C-S (cm^{-1})	N-C-S (cm^{-1})	Aromatic C-H
1	Phenyl	3380	1595	1600, 1495	1580	1375, 750	1495, 1460	3130, 3050
2	o-Anisyl	3350	1575	1608, 1490	1585	1370, 700	1490, 1460	3100, 3010
3	p-Tolyl	3340	1540	1600, 1490	1575	1370, 740	1490, 1465	3135, 3040
4	1-Pyridyl	3270	1570	1610, 1480	1570	1370, 745	1490, 1450	3120, 3040
5	p-Hydroxyphenyl	3380	1585, 1580	1605, 1485	1575	1370, 740	1480, 1455	3170. 3040
6	p-chlorophenyl	3290	1535	1600, 1495	1570	1380	1480	3120, 3040

Table-3: Antifungal screening of 5-arylamino-1,2,3,4-thiatriazoles



S. No.	Aryl Group	Average Inhibition (5)					
		Aspergillous Niger (ppm)			*Fusarium oxyporium* (ppm)		
		1000	100	10	1000	100	10
1	Phenyl	65	36	15	65	32	14
2	p-Tolyl	82	59	36	81	58	36
3	1-Pyridyl	68	40	30	67	40	30
4	Cyclohexyl	57	55	36	59	56	35
5	o-anisyl	84	50	38	86	50	36
Dithane M-45		100	81	68	100	80	68

Antifungal Activity

Test fungi *Aspergillous niger* and *Fusarium oxyporium* were obtained from the IARI, New Delhi and maintained on Agar compounds (1 to 5) were screened invitro by Agar Plate Technique[6] at different concentration (1000, 100 & 10 ppm), Dithane M-45, a commercial fungicide was also tested under similar condition for comparison.

Results of fungicidal activity were summarized in Table-3. It is evident from the data the most active were 5-p-tolyl and 5-o-anisyl amino –1,2,3,4-thiatriazoles.

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