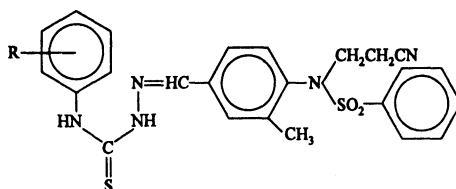


Synthesis and Characterization of 1-(1-Methyl-4-N-cyanoethyl-N-benzene sulphonyl amino benzylidene) 4-Aryl thiosemicarbazone

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The synthesis of 1-(1-methyl-4-N-cyanoethyl-N-benzene sulphonyl amino benzylidene) 4-Aryl thiosemicarbazones were carried out by refluxing 4-aryl thiosemicarbazide and 1-methyl-4-N-cyanoethyl-N-benzenesulphonyl benzaldehyde in stoichiometric amounts dissolved in aqueous ethanol in presence of few drops of acetic acid.

Key Words: Synthesis, Substituted 4-aryl thiosemicarbazone.



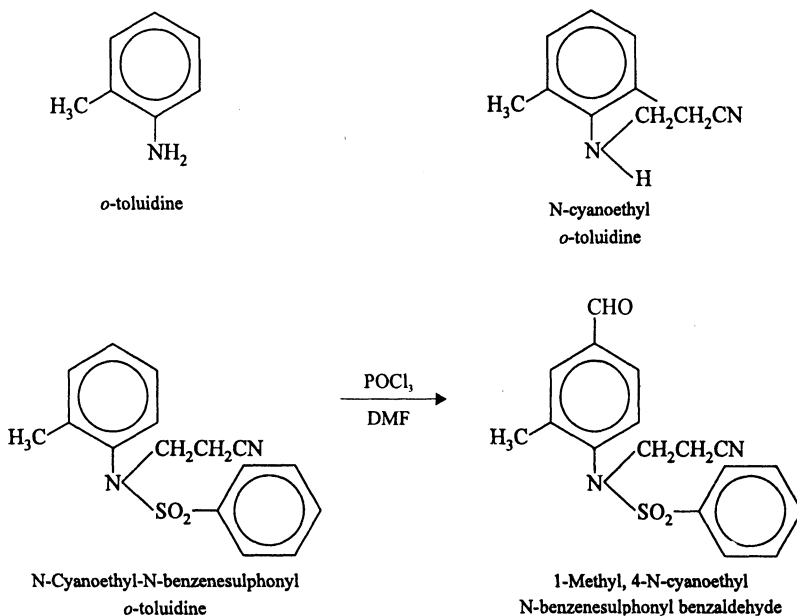
1-(1-Methyl-4-N-cyanoethyl-N-benzenesulphonyl amino benzylidene) 4-aryl thiosemicarbazone

INTRODUCTION

Thiosemicarbazones are well known antifungal and antitubercular drugs¹. They have been frequently employed for determination of inorganic ions²⁻⁴. Looking to the usefulness and importance of thiosemicarbazones, it was considered worthwhile to synthesize 4-aryl thiosemicarbazones of 1-methyl-4-N-cyanoethyl-N-benzenesulphonyl benzaldehyde with the view that these products might give better results in medicinal as well as in analytical chemistry.

1-Methyl-4-N-cyanoethyl-N-benzenesulphonyl benzaldehyde required in the synthesis was prepared in three steps. Monocyanoethylation of *o*-toluidine was carried out as reported in the literature⁵. The product 3-(*o*-toluidine) propionitrile was converted into N-cyanoethyl-N-benzenesulphonyl *o*-toluidine and formylation was carried out in DMF and POCl₃ (yield *ca.* 42%)⁶. The reaction sequence is shown in **Scheme-1**.

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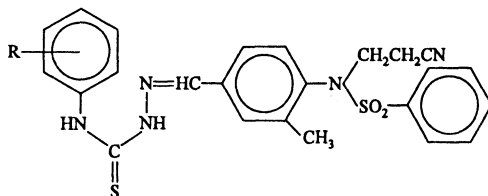


4-Aryl thiosemicarbazides needed for the condensation were synthesised according to the method reported in literature⁷.

EXPERIMENTAL

All the chemicals used were of extra pure grade. Melting points were taken in open capillary and are uncorrected. The products were routinely checked for their purity by TLC on silica gel. Compounds gave satisfactory C, H, N and S analysis. IR spectra were taken as KBr pellets on Shimadzu 8201 PC FTIR.

To a hot solution of aldehyde (0.001 mole, 0.327 g) in 60% ethanol containing 5–6 drops of glacial acetic acid was added 60% ethanolic solution of 0.001 mole of 4-aryl thiosemicarbazide. The contents were heated on a steam bath for 2 h and allowed to stand overnight. The solid which separates out was collected under suction, washed with a little 60% ethanol and recrystallized from glacial acetic acid, yield; ranges from 56 to 72%.

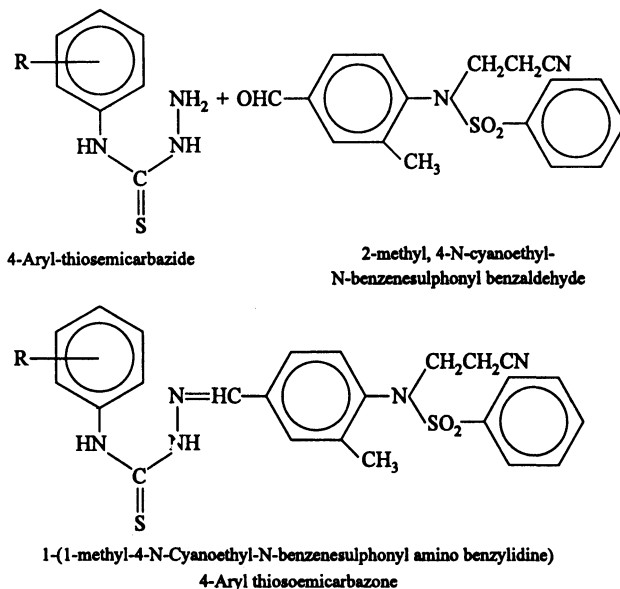


- | | | |
|---------------------------|--|---|
| 1 (a) R = H | 1 (b) R = CH ₃ (<i>o</i>) | 1 (c) R = CH ₃ (<i>p</i>) |
| 1 (d) R = Cl (<i>p</i>) | 1 (e) R = (OCH ₃) (<i>o</i>) | 1 (f) R = OCH ₃ (<i>p</i>) |

The characterisation data of the compounds **I(a–f)** are given in Table–1.

TABLE-1
PHYSICO-CHEMICAL AND IR (cm^{-1}) DATA OF SUBSTITUTED THIOSEMICARBAZONES

Compd.	R	m.p. ($^{\circ}\text{C}$)	Yield (%)	m.f.	% Found (Calcd.)			Colour	Solvent of recrystallization	IR frequencies (cm^{-1})
					C	H	N			
1a	H	132	72	$\text{C}_{24}\text{H}_{23}\text{N}_5\text{O}_2\text{S}_2$	60.1 (60.3)	4.70 (4.80)	14.4 (14.6)	Pale yellow shining crystals	Acetic acid	3431 ν (-NH), 2923, 2858 ν (CH_2), 2262 ν (CN), 1315 ν (ArC-N), 1164 ν (S=O, Sulphonamide), 1087 ν (C=S)
1b	<i>o</i> -CH ₃	131	57	$\text{C}_{25}\text{H}_{25}\text{N}_5\text{O}_2\text{S}_2$	60.9 (60.0)	5.10 (5.09)	14.0 (14.2)	Off white shining crystals	Acetic acid	3431 ν (NH), 2925, 2858 ν (CH_2), 2262 ν (CN), 1313 ν (ArC-N), 1166 ν (S=O, Sulphonamide), 1085 ν (C=S)
1c	<i>p</i> -CH ₃	126	67	$\text{C}_{25}\text{H}_{25}\text{N}_5\text{O}_2\text{S}_2$	60.8 (61.0)	4.99 (5.09)	14.1 (14.2)	Pale yellow shining crystals	Acetic acid	3431 ν (NH) 2925, 2858 ν (CH_2), 2262 ν (CN), 1313 ν (ArC-N), 1164 ν (S=O, Sulphonamide), 1085 ν (C=S)
1d	<i>p</i> -Cl	124	56	$\text{C}_{24}\text{H}_{22}\text{N}_5\text{O}_2\text{S}_2\text{Cl}$	56.5 (56.3)	4.60 (4.30)	13.3 (13.7)	Pale yellow shining crystals	Acetic acid	3431 ν (NH), 2925, 2856 ν (CH_2), 2262 ν (CN), 1315 ν (Ar C-N), 1166 ν (S=O, Sulphonamide), 1087 ν (C=S), 723, 760 ν (Ar C-Cl)
1e	<i>o</i> -OCH ₃	129	68	$\text{C}_{25}\text{H}_{25}\text{N}_5\text{O}_3\text{S}_2$	59.0 (59.1)	4.80 (4.90)	13.6 (13.8)	Pale yellow shining crystals	Acetic acid	3431 ν (NH), 2823, 2858 ν (CH_2), 2262 ν (CN), 1313 ν (Ar C-N), 1166 ν (S=O, Sulphonamide), 1085 ν (C=S)
1f	<i>p</i> -OCH ₃	131	70	$\text{C}_{25}\text{H}_{25}\text{N}_5\text{O}_3\text{S}_2$	59.2 (59.1)	4.70 (4.90)	13.7 (13.8)	Pale yellow shining crystals	Acetic acid	3431 ν (NH), 2923, 2958 ν (CH_2), 2262 ν (CN), 1315 ν (ArC-N), 1166 ν (S=O, Sulphonamide), 1087 ν (C=S)



All the six thiosemicarbazones are off white to pale yellow shining, crystalline in appearance. The mode of condensation is as follows.

ACKNOWLEDGEMENT

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