

Synthesis of Schiff's Bases and their Reduction

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1-(2'-methylamino-4'-substituted thiazole)-2-hydroxy-naphthalenes have been obtained by the reduction of corresponding Schiff's bases with sodium borohydride and lithium aluminium hydride. The Schiff's bases have been obtained by the condensation of 2-hydroxy-1-naphthaldehyde and corresponding 2-aminothiazoles.

In a plan Fig. 1 to synthesize heterocyclic steroids (I) a few Schiff's bases (IV) have been obtained in good yields by the condensation of 2-hydroxy-1-naphthaldehyde (II) and various 2-aminothiazoles (III) by refluxing in anhydrous alcohol Table 1.

In ir IV have characteristic absorption bands, 1200cm^{-1} (phenolic OH) and 1450cm^{-1} (aromatic).

These Schiff's bases or imines (IV) are smoothly reduced with sodium borohydride to corresponding

TABLE 1—PHYSICAL CONSTANTS AND ANALYTICAL DATA OF SCHIFF'S BASES(IV)

Compound	M.p. °C	Yield %	Solvent of crystallization	Formula	Analytical data %	
					Found	Required
IVa	161	70	EtOH/THF*	$\text{C}_{14}\text{H}_{10}\text{N}_2\text{SO}$	N 11.22	11.02
IVb	157	70	EtOH/THF	$\text{C}_{20}\text{H}_{14}\text{N}_2\text{SO}$	C 72.72 H 4.35 N 8.30 S 10.00	72.72 4.25 8.48 9.70
IVc	165	50	EtOH	$\text{C}_{21}\text{H}_{16}\text{N}_2\text{SO}$	C 73.15 H 4.64	73.25 4.65

* EtOH=ethanol, THF=tetrahydrofuran.

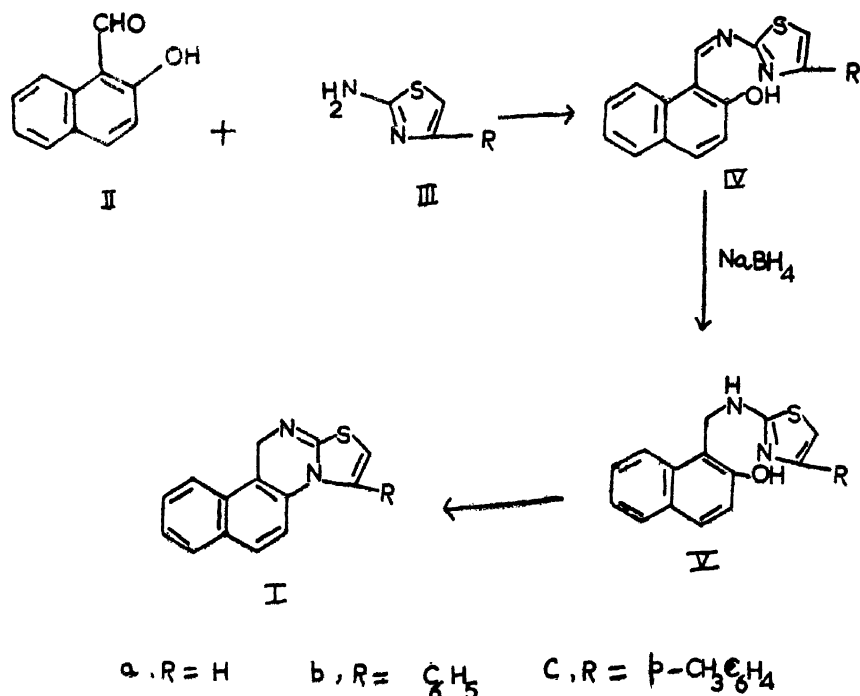


Fig. 1

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TABLE 2—PHYSICAL CONSTANTS AND ANALYTICAL DATA OF 1-(2-METHYLAMINO-4'-SUBSTITUTED THIAZOLE)-2-HYDROXY-NAPHTHALENES (V).

Compound	M.p. °C	Yield %	Solvent of crystallization	Formula	Analytical data %	
					Found	Required
Va	158	70	Ethanol	C ₁₄ H ₁₂ N ₂ SO	C 65.68 H 4.79	65.52 4.68
Vb	168	60	Ethanol	C ₂₀ H ₁₆ N ₂ SO	C 72.20 H 4.93 N 8.13 S 9.80	72.29 4.82 8.43 9.63
Vc	179	60	Acetone	C ₂₁ H ₁₈ N ₂ SO	N 8.21	8.09

1-(2'-methylamino-4'-substituted thiazole)-2-hydroxy-naphthalenes (V) Table 2. The i. r. spectra indicate an additional broad band around 3300 cm⁻¹ (>NH).

The reduction of imines (IV) with lithium aluminium hydride has not been very comfortable, often the yields are accompanied by tarry materials.

Experimental

All the melting points were taken in sulphuric acid bath using soft glass capillary tubes and are uncorrected. Microanalyses were done by C.S.I.R.O. Microanalytical Service, Australia.

2-Hydroxy-1-naphthaldehyde (II) : 2-Hydroxy-1-naphthaldehyde was obtained from β -naphthol using the method described in Vogel¹.

2-Amino-4-phenylthiazole² : Phenacyl bromide (19.9 g, 0.1 mole) and thiourea (7.6 g, 0.1 mole) were refluxed in ethanol (100 ml) for 2 hr. The solvent was removed under reduced pressure and the solid so obtained was dissolved in water, basified with aqueous sodium hydroxide and filtered. The residual solid was washed with water, dried and crystallized from benzene. M. p. 151°, yield 12.30 g (70%).

2-Amino-4-tolylthiazole : It was obtained from thiourea and ω -bromo-*p*-methyl-acetophenone. The product is crystallizable from ethanol. M. p. 136°, yield 60%.

Schiff's base (IV) : 2-Aminothiazole (III) (5 g, 0.05 mole) and 2-hydroxy-1-naphthaldehyde (8.6 g, 0.05 mole) were dissolved in minimum quantity of absolute alcohol (100 ml). The reaction mixture was refluxed for about 1 hr. The solvent was removed under reduced pressure and crystallized the residue from ethanol. The yields, m. p., etc. are given in Table 1.

Reduction of IV : The Schiff's base (IV) (12.70 g, 0.05 mole) was dissolved in ethanol (500 ml) and to it was added sodium borohydride (4 g, 0.1 mole) in small instalments. The reaction mixture was allowed to stand overnight. The solvent was removed under reduced pressure and the residue washed with water and crystallized from ethanol. The yields, m. p., etc. are given in Table 2.

Acknowledgement

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References

1. A. I. VOGEL, "A Textbook of Practical Organic Chemistry", Longmans, London, 1961, p. 704.
2. TRAUMANN, *Liebigs Ann. Chem.*, 1888, 249, 38.