

Zeitschrift: Archives des sciences [1948-1980]
Herausgeber: Société de Physique et d'Histoire Naturelle de Genève
Band: 25 (1972)
Heft: 3

Artikel: Reduction of 2-Chloro-4-methylquinoline
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DOI: <https://doi.org/10.5169/seals-739376>

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REDUCTION OF 2-CHLORO-4-METHYLQUINOLINE

BY

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ABSTRACT

From the reduction of 2-chloro-4-methylquinoline (*1*) three compounds (*2~4*) were obtained, besides 4-methylquinoline. The structures were assigned on the basis of elemental analysis and spectral studies.

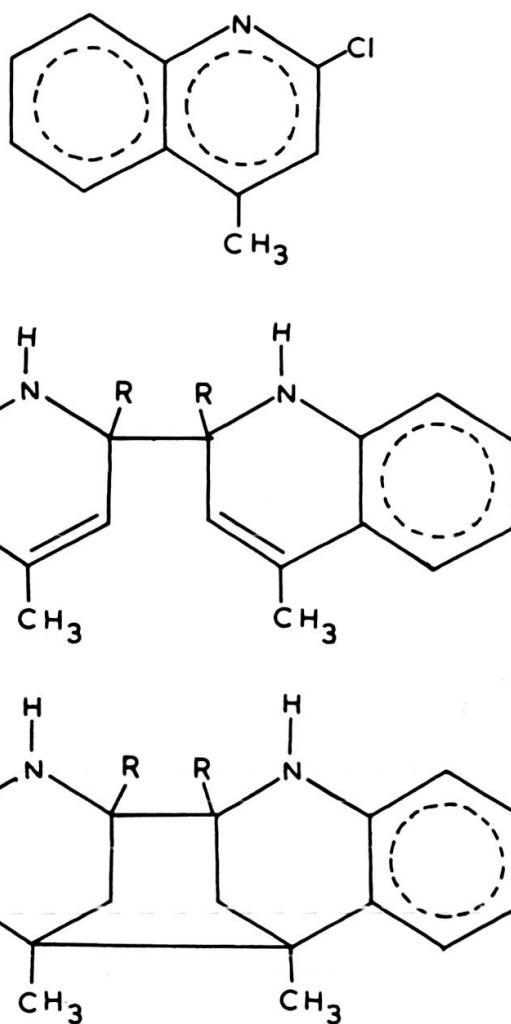
DISCUSSION

The reduction of 2-chloro-4-methylquinoline ¹ with Sn and HCl indicated the presence of six compounds on TLC examination and three products (*2~4*) besides 4-methyl-quinoline (major product) could only be separated in small amounts.

NMR spectral studies of *2* ($C_{20}H_{18}N_2Cl_2$) revealed the presence of a singlet for 6H at δ 2.55 and a singlet for 2H at δ 6.15, accounting for the presence of two methyl groups and two olefinic protons. The aromatic protons had the chemical shift in the range of δ 7.1 ~ 7.8 for 8H and for 2H attached to nitrogen at δ 10.2. However, *3* ($C_{20}H_{20}N_2$) showed the presence of a singlet for 6H of two methyl groups at δ 2.5 for two olefinic protons (as doublet) at δ 6.70 and for eight aromatic protons at δ 7.1 ~ 7.8, for 2H attached to nitrogen at δ 9.5 and for 2H as N-CH at δ 3.05 (doublet). The NMR spectra would confirm the structures as (2-2'-bis-(2-chloro-4-methyl-1H-1,2-dihydro-quinolyl) for *2* and 2,2'-bis-(4-methyl-1H-1,2-dihydro-quinolyl) for *3*. Further it could be supported from the fact that protons at nitrogen in *2* shifted to the lower field as against one in *3* along with the presence of N-CH proton in *3* and its absence in *2* thus confirming that *2* and *2'* positions in *2* were occupied by chlorine. The possibility of *2* as *3* HCl was also ruled out as chlorine was not in ionic form.

In NMR spectrum of *4* ($C_{20}H_{20}N_2Cl_2$) the signal for two methyl groups was observed as singlet at δ 1.3 (at comparatively much higher field). There were only

eight aromatic protons at δ 7.1 \sim 7.8. There were indications of 4H at δ 1.52 \sim 2.10 indicating the presence of two methylene groups. There was no signal for the olefinic proton at δ 6.4 \sim 6.7. Mass spectra of 4 gave the m/e peaks at 288, 144 and base peak at 143. On the basis of these spectral evidences, the structure to 4 was assigned as 'tricyclo (5,3,1,1^{2,6})-3,10-diaza-1,2-dichloro-6,7-dimethyl-4,5 : 8,9-dibenzodecane.'



Formation of 2, 3 and 4 along with the lepidine indicated that the reduction at first stage could involve the condensation of 2-chlorolepidine molecule at position 2 resulting in the formation of 2 which on further reduction would either cyclise at position 4 and 4' resulting in the formation of 4^{2,3} or the chlorine atom would be replaced with hydrogen.

The reduction of 1 through 3 should also yield 5 but it could not be isolated.

Table

No.	Molecular formula	Chemical Shifts (δ) of protons					
		Aromatic protons	-NH protons	=CH protons	-N-C-H protons	-CH ₂ - protons	-C-CH ₃ protons
<u>2</u> (CDCl ₃)	C ₂₀ H ₁₈ N ₂ Cl ₂	7.1-7.8 (8H)	10.2 (2H)	6.15 (s) (2H)	-	-	2.55 (s) (6H)
<u>3</u> (CDCl ₃)	C ₂₀ H ₂₀ N ₂	7.1-7.8 (8H)	9.5 (2H)	6.7 (d) (2H)	3.05 (d) (2H)	-	2.50 (s) (6H)
<u>4</u> (TFA)	C ₂₀ H ₂₀ N ₂ Cl ₂	7.1-7.8 (8H)	8.8 (2H)	-	-	1.5-2.1 (4H)	1.30 (s) (6H)

EXPERIMENTAL

Melting points are uncorrected. Microanalyses were done at Central Drug Research Institute, Lucknow. NMR spectra are from NIH, Bethesda, Md. U.S.A. or CDRI and are recorded in CDCl₃ (of 2 and 3) and in T.F.A. (of 4) on a Varian A-60D model using TMS as internal reference standard. Mass spectrum of 4 was also from NIH.

REDUCTION OF 2-CHLORO-4-METHYLQUINOLINE

2-Chloro-4-methylquinoline (17.8 g, 0.1 M) was reduced with conc. HCl (120 ml), H₂O (160 ml) and Sn (16 g) on the lines followed earlier¹. After decomposition of the complex the reaction mixture was extracted with chloroform. The chloroform extracts were dried over anhydrous Na₂SO₄ and concentrated to give a white product, insoluble in ether. Compounds 2 (mp 238°) and 3 (mp 227°) were separated from this white product on repeated crystallisation with acetone.

Ether soluble portion afforded the crude product which was distilled under reduced pressure [(0.5 mm) 80-85°]. At first a solid sublimed followed with the lepidine. The solid (4) was crystallised from acetone.

ANALYSIS

for $C_{20}H_{18}N_2Cl_2$ (2)

Calcd.: C, 67.22; H, 5.04; N, 7.84%

Found: C, 67.48; H, 5.16; N, 7.56%

for $C_{20}H_{20}N_2$ (3)

Calcd.: C, 83.33; H, 6.94; N, 9.71%

Found: C, 83.46; H, 6.72; N, 9.88%

for $C_{20}H_{20}N_2Cl_2$ (4) 246° C

Calcd.: C, 66.85; H, 5.57; N, 7.69%

Found: C, 66.33; H, 5.76; N, 7.30%

ACKNOWLEDGEMENT

The authors thank the Head of the Chemistry Department for providing the normal facilities and the authors thank UGC (DK) and CSIR (AS) for providing the scholarship and financial assistance.

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