Reduction of 2-chloro-6-methoxy-4-methylquinoline. Part II

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REDUCTION OF 2-CHLORO-6-METHOXY-4-METHYLQUINOLINE

PART-II

BY

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In continuation of our earlier communication [1] the present study deals with the reduction of 2-chloro-6-methoxy-4-methylquinoline with tin and hydrochloric acid when alongwith 6-methoxyquinoline (2) another compound identified as 2,2'-bis-6-methoxy-4-methylquinoline was also isolated (3).

2-Chloro-6-methoxy-4-methyl quinoline [1] was reduced with tin and HCl when 6-methoxy-4-methylquinoline [2] as a major component was obtained. Besides this, another compound [3] in minor quantity was also obtained and on the basis of elemental analysis an empirical formula $C_{11}H_{10}ON$ was suggested to 3. No chlorine was found to be present.

Molecular ion peak in mass spectrum was found at m/e 344, thus suggesting the molecular formula to 3 as $C_{22}H_{20}O_2N_2$.

Nmr spectra (in $CDCl_3$ using TMS as internal reference standard) revealed the presence of a singlet at δ 2.55 for six protons and another singlet at δ 3.90 also for six protons. Eight aromatic protons were found to be present in the range δ 7.05-7.95. The singlet at δ 2.55 would account for the presence of two methyl groups and a singlet at δ 3.9 would account for the presence of two methoxy groups. The absence of the chlorine in the molecule revealed the involvement of position-2 of the quinoline in the reaction indicating the compound 3 as a bis molecule.

The mass spectrum of 3 gave a base peak at m/e 172 besides the molecular ion peak indicating the cleavage of the molecule at positions 2,2' giving an ion 4.

From all these observations the structure to this compound (3) was assigned as 2,2'-bis-6-methoxy-4-methylquinoline.

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EXPERIMENTAL

2-Chloro-6-methoxy-4-methylquinoline (41.5 g,0.2M) was reduced with Conc. HCl (300 ml), H_2O (300 ml) and tin (40.0g) on the lines followed earlier [2]. The reaction mixture treated in usual manner [1] gave a crude solid product. The tlc examination of this crude product revealed the presence of two components. These components were separated by repeated crystallisation with petroleum ether (40-60°), one which was more soluble was identified as 6-methoxy-4-methylquinoline (18.6 g. 48%, mp. 51°)[3] and another which was less soluble was identified as 2,2'-bis-6-methoxy-4-methylquinoline (3). (White needles, mp. 135°, 7.71 g. 10%). Its purity was ascertained through tlc examination over Silica Gel G plates, using ethyl alcohol, ethyl acetate, formic acid (6:2:2) as mobile phase (R_f values: 2, .78; 3, .69).

ANALYSIS

Calcd for	$C_{22}H_{20}O_2N_2$ (3)
	C, 76.74%; H, 5.81%; N, 8.14%
Found	C, 76.38%; H, 5.64%; N, 8.38%.

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