

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

Autor(en): **Pandey, R. K. / Surana, Asha / Joshi, B. C.**

Objekttyp: **Article**

Zeitschrift: **Archives des sciences [1948-1980]**

Band (Jahr): **29 (1976)**

Heft 2

PDF erstellt am: **25.05.2024**

Persistenter Link: <https://doi.org/10.5169/seals-739680>

Nutzungsbedingungen

Die ETH-Bibliothek ist Anbieterin der digitalisierten Zeitschriften. Sie besitzt keine Urheberrechte an den Inhalten der Zeitschriften. Die Rechte liegen in der Regel bei den Herausgebern.

Die auf der Plattform e-periodica veröffentlichten Dokumente stehen für nicht-kommerzielle Zwecke in Lehre und Forschung sowie für die private Nutzung frei zur Verfügung. Einzelne Dateien oder Ausdrucke aus diesem Angebot können zusammen mit diesen Nutzungsbedingungen und den korrekten Herkunftsbezeichnungen weitergegeben werden.

Das Veröffentlichen von Bildern in Print- und Online-Publikationen ist nur mit vorheriger Genehmigung der Rechteinhaber erlaubt. Die systematische Speicherung von Teilen des elektronischen Angebots auf anderen Servern bedarf ebenfalls des schriftlichen Einverständnisses der Rechteinhaber.

Haftungsausschluss

Alle Angaben erfolgen ohne Gewähr für Vollständigkeit oder Richtigkeit. Es wird keine Haftung übernommen für Schäden durch die Verwendung von Informationen aus diesem Online-Angebot oder durch das Fehlen von Informationen. Dies gilt auch für Inhalte Dritter, die über dieses Angebot zugänglich sind.

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

PART-II

BY

R. K. PANDEY, ASHA SURANA and B. C. JOSHI¹

In continuation of our earlier communication¹ 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.

¹ Chemical Laboratories, University of Rajasthan, Jaipur-302004, India.

EXPERIMENTAL

2-Chloro-6-methoxy-4-methylquinoline (41.5 g, 0.2M) was reduced with Conc. HCl (300 ml), H₂O (300 ml) and tin (40.0g) on the lines followed earlier². The reaction mixture treated in usual manner¹ 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 ₂ N ₂ (3) |
| | C, 76.74%; H, 5.81%; N, 8.14% |
| Found | C, 76.38%; H, 5.64%; N, 8.38%. |

ACKNOWLEDGEMENT

The authors thank the Head, Chemistry Department for providing the necessary facilities and Defence Research and Development Orgn., Government of India for financial assistance.

REFERENCES

- [1] D. KISHORE, Asha SURANA and Bhuwan C. JOSHI, *Archives des Sciences*, Vol. 26, (1973).
- [2] G. I. MIKHALOV and J. GEN, Chem. USSR. 6, 511-15, (1936). Chem. Abstr. 30, 63725, 3446 (1936), Russ. Pat. 39164.
- [3] K. N. CAMPBELL, R. STAUT TIPSON, R. C. ELDERFIELD, B. K. CAMPBELL, Mary A. CLAPP, W. J. GENSHER, Dwight MARRISON and W. J. MARAN. J. Org. Chem. 11, 803-11 (1946).