

A GREEN MICROWAVE ASSISTED SYNTHESIS OF NEW (ANTHRACENE-9-YL) METHYLAMINES AS AN ENVIRONMENTALLY FRIENDLY ALTERNATIVES

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ABSTRACT

Synthesis of polynuclear aromatic hydrocarbons and its derivatives was carried under microwave irradiations. The title compound, (anthracene-9-yl) methylamine, was synthesized in higher yield by reaction of 9-chloromethylantracene with pyrrolidine and hexamethyleneimine. The structure of this new compound was confirmed by elemental analysis, IR, Mass, ¹H NMR spectral data.

Keywords: 9-Chloromethyl Anthracene, Pyrrolidine, Microwave Irradiation, Mass, NMR.

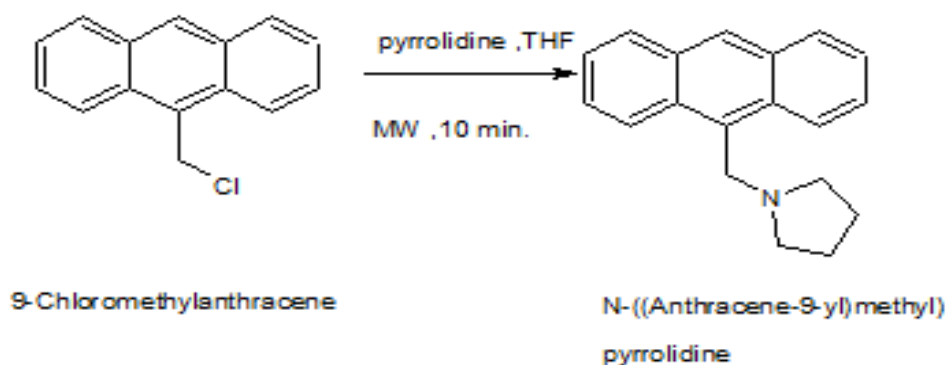
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INTRODUCTION

A considerable amount of research has been devoted to the synthesis of (anthracene-9-yl) methylamines used in many synthetic routes¹. In 1986, Gedye et al.^{2,3} and Giguere et.al.^{4,6} demonstrated that a wide variety of organic reaction can be conducted very rapidly using microwave irradiation. Since then, several other groups have described accelerated organic reaction⁷⁻⁹. Additionally some recently reported example include one pot reaction¹⁰ and particular organic reactions such as Suzuki couplings¹¹, claisen rearrangement¹², but no study of the microwave assisted synthesis of (anthracene-9-yl) methylamines has been reported. We wish to report herein a facile high yielding synthesis of this compound in a short time using a domestic microwave oven.

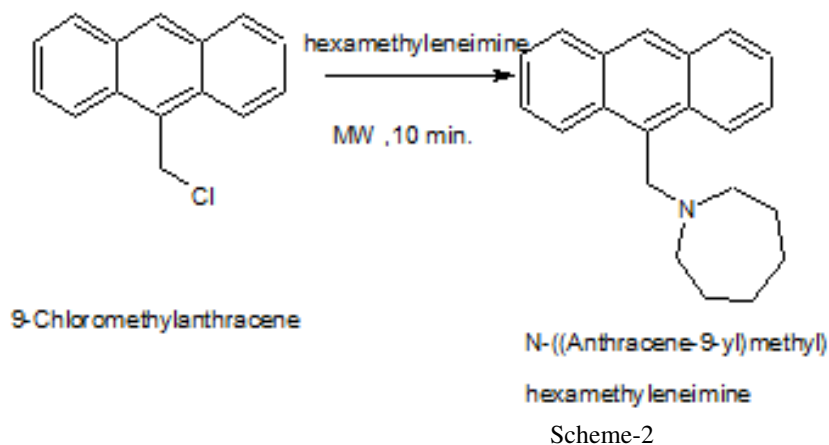
EXPERIMENTAL

In the reaction mixture 9-chloromethylantracene (0.01 mole), THF (10 ml) and pyrrolidine (0.01 mole) taken in a 250 ml borosil flask under constant stirring. Then the reaction mixture was irradiated in a microwave oven at 180°C for 10 min. with short interval of 30 sec.-1 min. . The completion of the reaction monitored by TLC examination, then reaction mixture was poured in to water and extracted with diethyl ether to remove pyrrolidine hydrochloride formed as a side product. The organic layer was separated, washed with water and dried over anhydrous Na₂SO₄. Removal of the solvent yielded the product (Scheme-1).



Scheme-1

In the reaction mixture 9-chloromethylanthracene (0.01 mole), THF (10 ml) , hexamethyleneimine (0.01 mole) taken in a 250 ml borosil flask under constant stirring .Then the reaction mixture was irradiated in a microwave oven at 180⁰C for 10 min. with short interval of 30 sec.-1 min. . The completion of the reaction monitored by TLC examination , then reaction mixture was poured in to water and extracted with diethyl ether to remove hexamethyleneimine hydrochlorides formed as a side product .The organic layer was separated , washed with water and dried over anhydrous Na₂SO₄ .Removal of the solvent yielded the product(Scheme-2).



RESULT AND DISCUSSION

The reaction of substituted anthracenes with amine afforded N-((Anthracene-9-yl) methyl) pyrrolidine and N-((Anthracene-9-yl) methyl) hexamethyleneimine by MWI. Identify of newly prepared (anthracene-9-yl) methylamines was established on the basis of their elemental analysis and spectral data.

N-((Anthracene-9-yl) methyl) pyrrolidine (1)

¹HNMR 400MHz (CDCl₃): δ1.61-1.89(m, 4H), δ2.82-3.07 (m, 4H), δ4.62(s, 2H), δ7.51-8.54 (m, 9H, aromatic); m/z 261 (M⁺) Elemental analysis C (87.25%), H (7.33%), N (5.40%)

N-((Anthracene-9-yl) methyl) hexamethyleneimine (2)

¹HNMR 400MHz (CDCl₃): δ1.77-1.80 (m, 6H), δ2.70 (m, 6H), δ4.62 (s, 2H), δ7.46-8.58 (m, 9H, aromatic); m/z 289 (M⁺) Elemental analysis C (87.16%), H (8.01%), N (4.83%)

Table-1: Physical data of compound 1 & 2

Compound	Molecular Formula	MP (°C)	Yield (%)	Reaction Time (min.)
1	C ₁₉ H ₁₉ N(261)	111	85	10
2	C ₂₁ H ₂₃ N(289)	105	86	10

CONCLUSION

All the reactions were carried out microwave irradiation method under solvent condition which leads to considerable time saving better yields and environmentally benign procedure.

ACKNOWLEDGEMENTS

The authors are grateful to Dept. of Chemistry, B.N.P.G. College for providing the experimental facilities and CDR I Lucknow for spectral analysis.

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[RJC-1156/2014]