

Green and Energy Efficient Synthesis of Pyrrole Derivatives

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Abstract— Exposure of microwave irradiation (MW) to a mixture of hexane-2, 5-dione with primary amines has resulted in to formation of substituted pyrroles in less than 100 seconds.

Keywords— MW; Primary amines; Hexane-2, 5-dione; Green chemistry

1. INTRODUCTION

Microwave assisted synthesis is a rapidly growing area in the field of synthetic organic chemistry. Microwave constitutes a nonclassical heating technique which can be known as Bunsen burner of the present time. Recently, it is growing as one of the most popular method of chemical synthesis which can reduce the reaction time dramatically as well as increase the yield and purity of the product. The use of microwave offers an excellent option for “Green Chemistry Scientists” as it reduces the chemical wastes, reaction time and energy consumption. In recent years, it has regularly drawn the attention of organic chemists. This non conventional heating technique has promoted number of reactions previously not observed under conventional method of chemical synthesis.

The development of environmentally benign synthetic method has been a primary objective of the synthetic organic chemist. The pressure of ever increasing demand of the substance of society has led to develop an alternative method of synthesizing substances. Thus, microwave chemistry has emerged as a discipline that has offered solution to all aspect of synthetic chemistry to an appreciable extent.

The field of heterocycles synthesis has been markedly affected by the application of microwave. Microwave assisted synthesis of indoles [1, 2], β -lactams [3], thiophenes [4], furans [4], dihydropyridines [5, 6], and quinazolines [7] and 2-cyanobenzothiazoles [7, 8] has been reported in recent years. Numbers of chemical reactions [9], Trost’s γ -addition, Heck reaction, Cannizzaro reaction, Aza-Michael reaction, Prins reaction etc. have been successfully carried out under microwave irradiation. In continuation of developments regarding microwave assisted synthesis of organic substances, we wish to report synthesis of some pyrrole derivatives.

2. EXPERIMENTAL PROCEDURE

The melting points of the synthesized compounds were recorded by melting point apparatus and were uncorrected. The structural confirmation of the synthesized compounds was done by scanning their ¹H NMR and ¹³C NMR. The Bruker DRX-100 spectrometer (chemical shift δ ppm) was used to record ¹H NMR and ¹³C NMR of the synthesized compounds. The reactions were carried out by exposure of microwave irradiation (MW) using domestic microwave oven (LG MC2146BRT; 800 watts with 2450 MHz frequency).The schematic route of the synthesis of the studied compounds is given as Fig. 1.

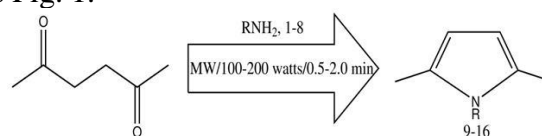


Fig. 1 Synthetic route for the synthesis of studied compounds

The structures of synthesized pyrrole are given below as:

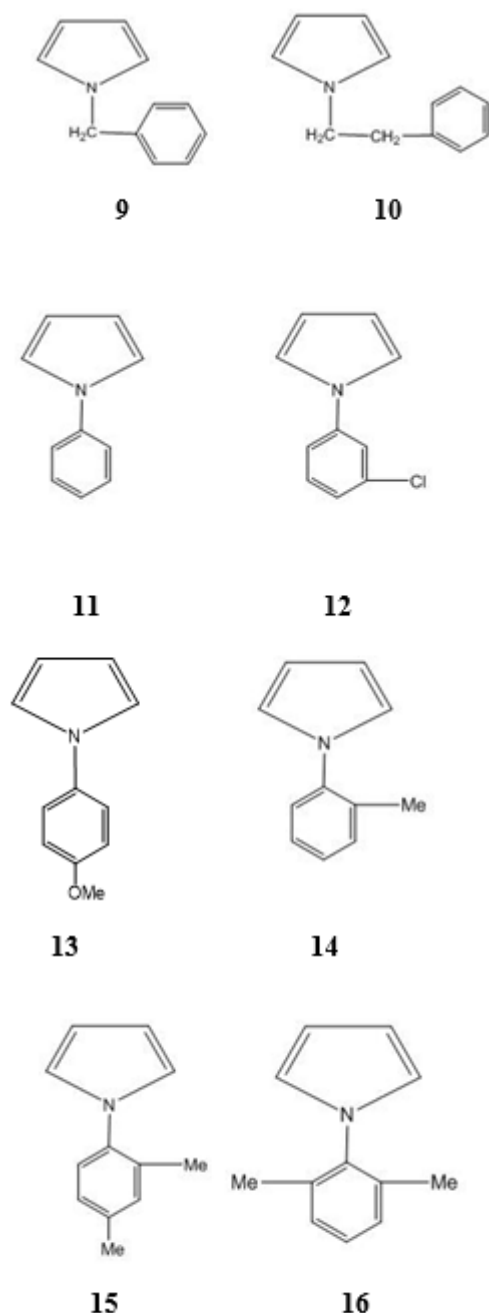


Fig. 2 Structure of synthesized pyrrole derivatives

The time of synthesis, power used and spectral data of synthesized pyrrole derivatives are given below as:

1-benzyl-H-pyrrole (9): time of synthesis = 30 sec, power used = 500 watt, yield = 78%, M.P. = 118 °C

¹H NMR: 7.2-7.33 (multiplets; benzene ring), 2.16 (Me), 5.82 (dd; pyrrole ring NH), 5.88 (N-CH₂-)

¹³C NMR: (Benzene ring; 125.7, 127.6, 128.6 & 137.3); 49 (N-CH₂-); 13.1 (Me); 105.6 (pyrrole ring)

1-Phenylethyl-1H-pyrrole (10): time of synthesis = 40 sec, power used = 500 watt, yield = 80%, M. P. = 126 °C

¹H NMR: 7.19-7.23 (multiplets; Benzene ring), 2.82 (CH₂), 4.18 (CH₂-N), 2.19 (Me), 5.78 (Pyrrole ring)

¹³C NMR: Benzene ring (125.9, 127.7, 128.6, 136.5); 37.7 & 48.5 (CH₂); 13.1 (Me); 105.6 & 127.6 (Pyrrole ring)

1-Phenyl-1H-pyrrole (11):

Time of synthesis = 35 sec, power used = 500 watt, yield = 77%, M.P. 120 °C

¹H NMR: 7.21, 7.38 (dd) (Benzene ring); 2.43 (Me); 2.01, 5.84 (Pyrrole ring)

¹³C NMR: Benzene ring (122.7, 129.6, 135.2, 135.6), 21.3 (Me), 12.7 (Me attached to pyrrole ring), 105.9 & 128.2 (pyrrole ring) 1-(3-chlorophenyl)-1

1-(3-Chlorophenyl)-1H-pyrrole (12):

Time of synthesis = 60 sec, power used = 500 watt, yield = 85%, M.P. 132 °C

¹H NMR: 7.34, 7.38, 7.43, 7.93 (multiplet); Benzene ring, 2.01 (Me), 5.84 (pyrrole ring)

¹³C NMR: Benzene ring (117.1, 119.4, 125.6, 131.9, 143.3); 12.7 (Me), 105.9, 128.2 (Pyrrole ring)

1-(4-Methoxyphenyl)-1H-pyrrole (13):

Time of synthesis = 60 sec, power used = 500 watt, yield = 81%, M.P. 122 °C

¹H NMR: 7.10, 7.83 (Benzene ring), 3.81 (OCH₃), 2.01, 5.84 (Pyrrole ring)

¹³C NMR: Benzene ring (114.9, 126.5, 130.9, 157.4); 55.8 (OCH₃); 12.7 (Me); 105.9, 128.2 (Pyrrole ring)

1-(o-tolyl)-1H-pyrrole (14):

Time of synthesis = 60 sec, power used = 500 watt, yield = 84%, M.P. 124 °C

¹H NMR: 7.22, 7.53, 7.60 (Benzene ring); 1.92 (Me); 1.87, 5.83 (Pyrrole ring)

¹³C NMR: Benzene ring (121.2, 125.8, 126.3, 128.1, 131.4, 141.5); 12.7 (Me); Pyrrole ring (105.9, 128.2)

1-(2, 4-dimethylphenyl)-1H-pyrrole (15):

Time of synthesis = 90 sec, power used = 500 watt, yield = 81%, M.P. 141 °C

¹H NMR: 7.21, 7.32, 7.63 (Benzene ring), 1.92 & 2.31 (Me attached to Benzene ring); 5.83 (Pyrrole ring); 1.87 (Me attached to Pyrrole ring)

¹³C NMR: Benzene ring (130.5, 131.4, 135.4, 137.3, 138.5); 21.6 & 25.1 (Me attached to Benzene ring); Pyrrole ring (105.9, 128.2); CH₃ attached to pyrrole ring (12.7)

1-(2, 6-dimethylphenyl)-1H-pyrrole (16):

Time of synthesis = 90 sec, power used = 500 watt, yield = 81%, M.P. 141 °C

¹H NMR: 7.4, 7.43 (Benzene ring); 1.92 (Me attached to Benzene ring); 1.91 (Me attached to Pyrrole ring); 5.83 (Pyrrole ring)

¹³C NMR: Benzene ring (125.3, 125.7, 128.4, 141.8); CH₃ attached to benzene ring (17.3); 105.9 & 128.2 (Pyrrole ring); 12.7 (CH₃ attached to Pyrrole ring).

3. RESULTS & DISCUSSION

Thus, the exposure of MW irradiation offers fast, clean, energy efficient and green method of synthesis of pyrrole derivatives.

4. CONCLUSIONS

- (i) Exposure of MW irradiation offers fast and clean synthesis of pyrrole derivatives.
- (ii) MW exposure saves markedly time of reaction.
- (iii) MW exposure offers energy efficient production of pyrrole derivatives.
- (iv) MW assisted method of synthesis increased the yield of the products compared to conventional method of synthesis.

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