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Comparism Of Thermal And Microwave Methods In Structural Modification Of Lumefantrine

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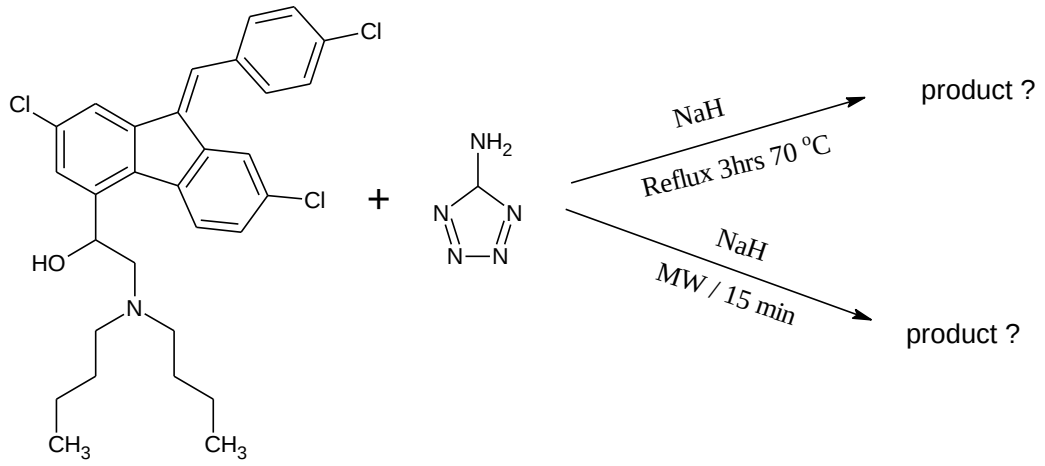
Abstract

The three chlorine atoms on lumefantrine were replaced with 5-aminotetrazole using both thermal method (TM) and microwave method (MW). The products were characterized with GC-MS spectral analyses. The usual isotopic mass percentages of 35(75%) and 37(25%) of chlorine abundance in nature were absent in the product's fragmentation peaks. Gas Chromatogram of the two methods showed that the percentage yield of the microwave method (MW) is about double that of the thermal method (TM).



Reaction Scheme

Key Words: Gas Chromatogram, Hyphenated Gas Chromatography and Mass Spectroscopy (GC-MS), Thermal Method (TM), Microwave Method (MW), Isotopic Mass.

Experimental

Lumefantrine (2 g, 0.0038M) and 5-aminotetrazole (0.999 g, 0.0117M) were separately weighed and transferred into RB-flask containing 50mls of toluene. Into this was carefully added sodium hydride (0.273 g, 0.0114M) and refluxed (TM) for 3hrs at 70°C. In the microwave method (MW), Lumefantrine (2 g, 0.0038M),

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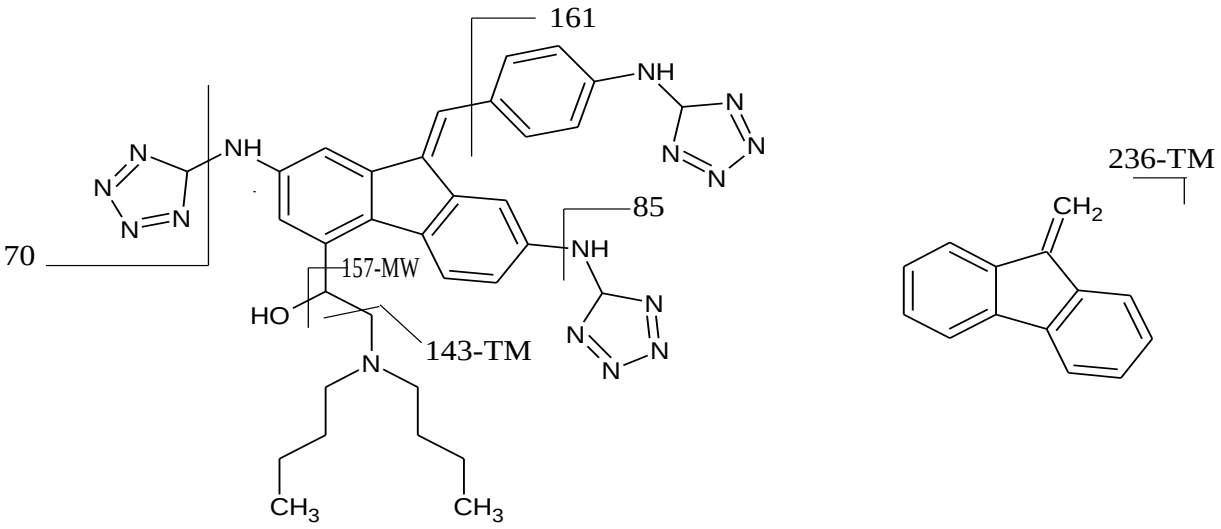
5-aminotetrazole (0.999 g, 0.0117M) and sodium hydride (0.273 g, 0.0114M, as 60% dispersion in paraffin oil) were added to 100 ml beaker. The beaker was placed in a household microwave oven for 15 minutes with periodic stirring at intervals of 5 minutes. The melting point of lumefantrine (125-128°C) (Literature value; 128°C)[3], and products from both thermal(TM) and microwave(MW) gave 155-160°C using Griffin Melting Point Apparatus. The R_f-values for thermal(TM) and microwave (MW) methods are 0.40 and 0.46 respectively using the same solvent system (Hexane, Ethyl acetate and glacial acetic acid. 8:2:1). The percentage peak area of the GC-MS chromatogram for microwave method is 11.91% while that of thermal method is 5.42%. The fragmentation pattern shown below is confirmed by MS spectral patterns in Figures 1 and 2 for the modified lumefantrine with IUPAC name: (9E)-5-[1-(dibutylamino) propan-2-yl]-9-[4-(1H-pentazol-1-ylamino) benzylidene]-N, N'-di(tetrazolidin-5-yl)-9,9a-dihydro-4aH-fluorene-2,7-diamine.



Fragmentation pattern of modified lumefantrine with 5-amino tetrazole

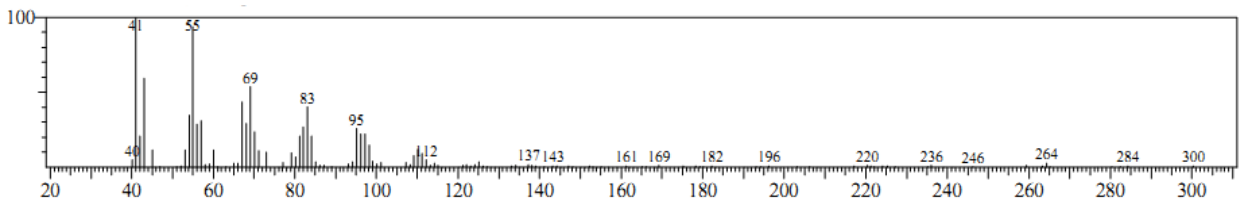


Figure 1: Mass Spectra for Thermal Method (TM)

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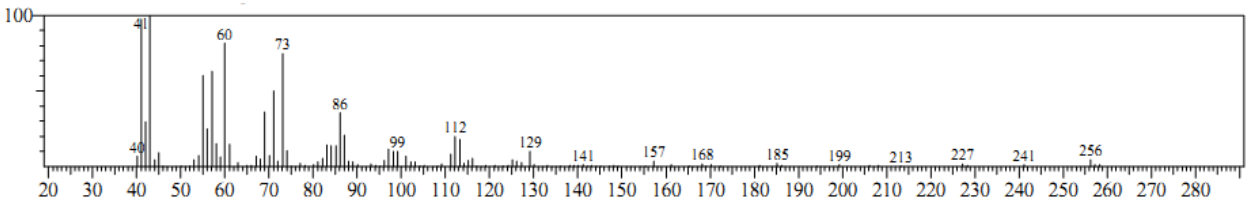

Figure 2: Mass Spectra for Microwave Method (MW)

Discussion and Conclusion

The peak area of a compound is directly proportional to its concentration based on equation (1)[2]: Concentration = (Response Factor) x (Peak Area) … … … … (1). The results obtained show that the percentage peak area was higher with microwave method (11.91%) than that for thermal method (5.42%). This is an indication that the percentage yield of the microwave method is about double that of the thermal method[1].

Acknowledgments

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