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Spectroscopic analysis on Synthesis 1-Methyl-3-(2'-Phenylethyl)-1H,3H-Quinazoline-2,4-dione

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ABSTRACT: *Carbomethoxy-methylanthranilic acid was synthesized from methylanthranilic acid with methylchloroformate in presence of potassium carbonate. Cyclisationreaction of N-carbomethoxymethylanthranilic acid was performed by heating to get N-methylisatoic anhydride. 2-metholamino-N-(2'-phenylethyl) benzamidewas formed from Isatoic anhydride and phenylethyl amine. High yield of 1-methyl-3-(2'- phenylethyl)-1H,3H-quinazoline-2,4-dione alkaloids were formed by condensation and cyclisation reaction between amide and methylchloroformate. The synthesis compound was identified with the help of its melting point, C^{13}NMR and H^{1}NMR spectra.*

KEYWORD: *C^{13}NMR, H^{1}NMR, Carbomethoxy-methylanthranilic acid,quinazoline, alkaloid*

I. INTRODUCTION

Synthetic and natural quinazoline alkaloids are known to have a variety of biological responses. Fifty quinazoline derivatives with varieties of biological activities are used for clinical purposed. Leaves and root of *AdhatodavasicaNees*, which contain some quinazolines alkaloids, are used for treatments for bronchitis, asthma, diarrhea and dysentery. These parts are also used for antiseptic, antiperiodic and anthelminthic properties [1]. Various types of quinazolinealkaloids and their chemotaxonic efforts are reported in the Rutaceae family. 4-quinazolines alkaloids have been isolated from seeds husks of *Zanthoxylumarborescens* [2]. Anti-Inflammatory activity has been reported in various quinazolines. Substituted at position-3 of the quinazolines nucleus have marked influenced on the activity [3]. In the present work quinazoline alkaloids are synthesized and evaluated their spectroscopy analysis.

II. METHODOLOGY

Formation of N-Carbomethoxy-methylanthranilic acid[4]

About 1.51g of N-methylanthranilic acid and 0.70g ofPotassium carbonate were dissolved in water. After addition of 4-5 ml methylchloroformate, the mixture was shook vigorously and warmed up to 90^0c. Then oil was separated out and white crystal was formed after cooling. The product was recrystallised with hot water. Its Melting point was 137^0C and the yield was 75.4%.

Formation of N-methylisatoic anhydride[4]

1.5g of N-methyl Carbomethoxyanthranilic acid was heated up to 220^0C for half an hour inside the oven. Then after cooling this reddish oil liquid was converted into crystals which were recrystallised with ethanol. The yellow small needle type crystals having melting point of 177^0C and the yield was 52%.

Formation of 2-metholamino-N-(2'-phenylethyl) benzamide[2]

About 0.689g of isatoic anhydride was dissolved in 0.522ml of phenylethylamine and then 10ml of dioxin was added to it. The mixture was heated up to 100^0C for 10 min. Then excessive amount of water was added to get oil which was crystallized by scratching and cooling. The crystals were washed with 5% K_2CO_3 and water, and then recrystallised with methanol. Its melting point was 106^0Candthe yield was 70%.

Formation of 1-methyl-3 (2'-phenylethyl)-1H,3H-quinazoline-2,4-dione[2]

The amide was dissolved in dioxin and aqueous solution of Na_2CO_3 and then methylchloformate were added to it. The solution was stirred at room temperature for 20 minutes. Excessive amount of water were added then white crystals were appeared. The crystals were washed with dilute HCl and 5% Na_2CO_3 and recrystallised with MeOH. Its melting point was 101^0C and yield 88%.

NMR spectroscopy investigation of Quinazoline

Spectra were recorded on a Brucker DPX 300 FT spectrometer, operating at 293k. Data sets were processed using standard Bruker software.

H^1: 300, 13 MHz, internal standard TMS (tetramethylsilane).

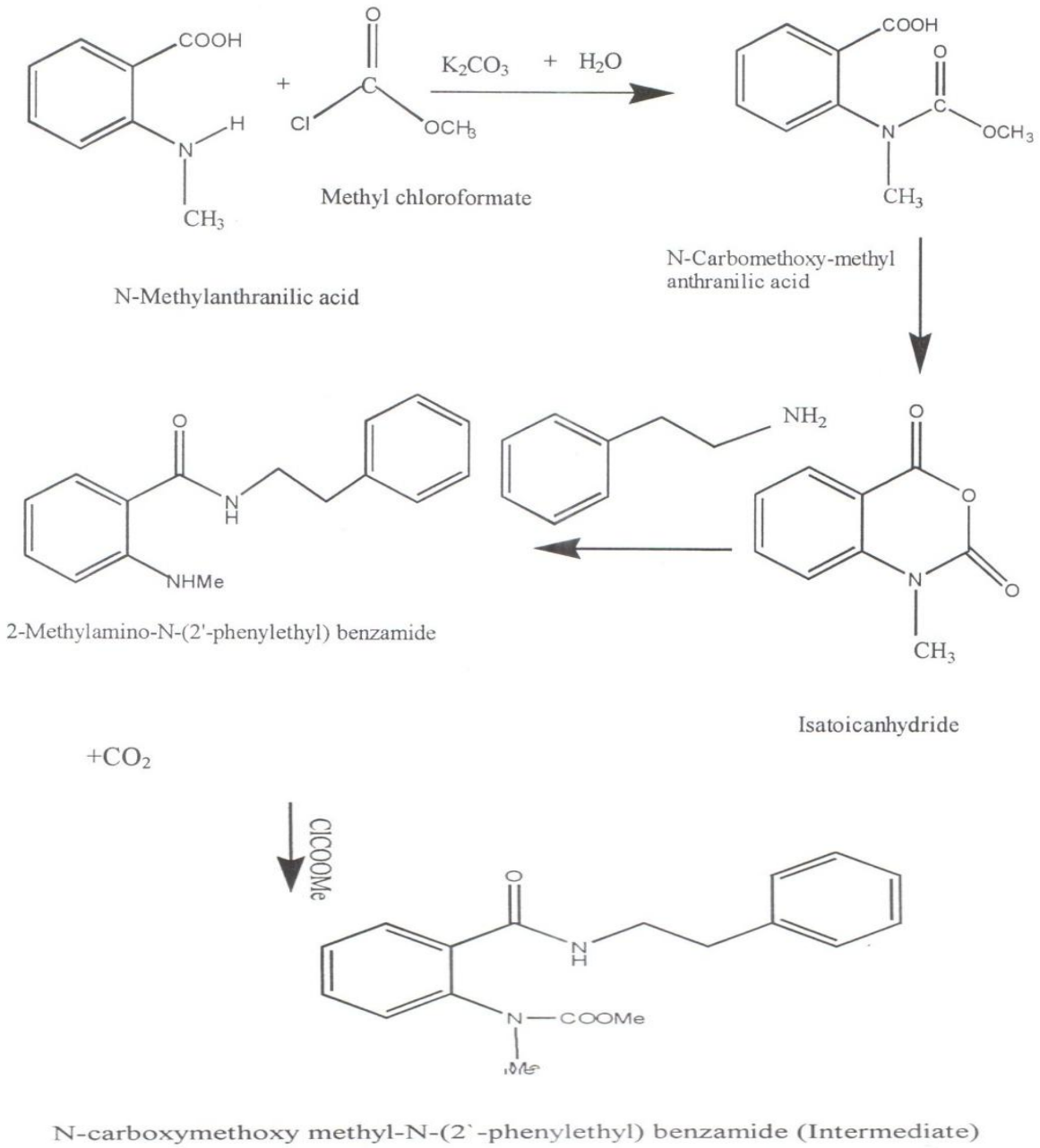
C^{13}: calibrated to CDCl3 triplett, (p=77, 0 ppm).

Melting points

Melting points was performed on electronic melting point apparatus.

III. RESULTS AND DISCUSSION

N-Carbomethoxy-methylanthranilic acid was sky blue colour with single spot on TLC in 254 nm ultraviolet light and Rf value was 0.32 on 10% methanol in chloroform. Formation of N-methylisatoic anhydride was performed by a cyclisation reaction of N-carbomethoxymethylanthranilic acid by heating. It was blue fluorescence in 366 nm ultraviolet light and Rf value was 0.79 on mobile phase of 10% methanol in chloroform. The crystals of amide had melting point sharply 106^0C and had sky blue fluorescence 366nm of ultraviolet light. Condensation and cyclisation reactions were occurred between amide and methylchloroformate, and gave 1-methyl-3-(2'- phenylethyl)-1H,3H-quinazoline-2,4-dione alkaloids was formed. It has dark blue fluorescence on 254nm ultraviolet light and Rf value was 0.60 on mobile phase of 10% methanol inchloroform. The yield was 88%.The synthetic route to 1-methyl-3-(2'- phenylethyl)-1H,3H-quinazoline-2,4-dione is shown in figure 1.



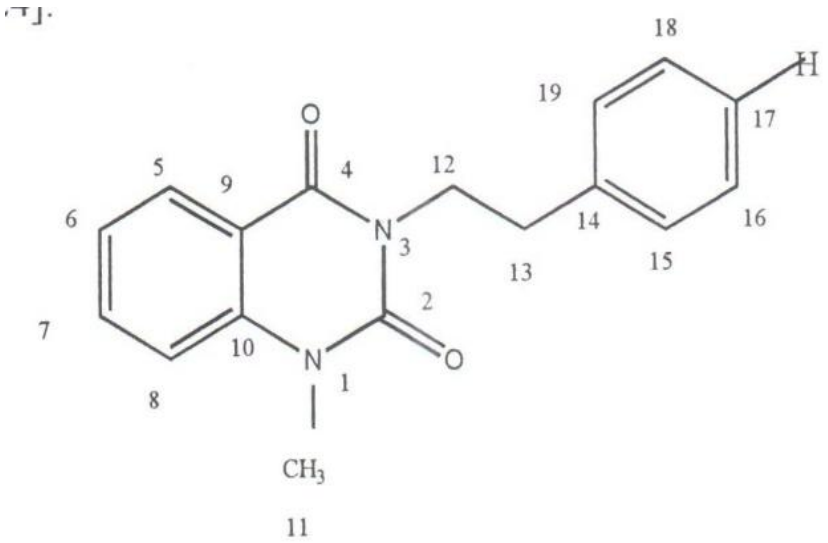


Fig. 1: synthetic route to 1-methyl-3-(2'- phenylethyl)-1H,3H-quinazoline-2,4-dione

NMR spectroscopy investigation of Quinazoline alkaloids.

The obtained experimental data of ^{1}H-NMR spectrum are as follows: ^{1}H-NMR($CDCl_3$):8.36-8.33(*m,* 1H,h-5), 7.79-7.77(*m*,1H,H-8), 7.47-7.30(*m*,7H, H-15, H-16, H-17.H-18,H-19,H-6,H-7),4.46-4.39(*m,* 2H, -N-CH_2-), 3.72(m,3H,N-CH_3) and 3.12-3.07(*m*,2H,Ar-CH_3). The spectrum of fig 2 shows two multipletes of 3.10 and 4.32ppm, representing the ethylene group of the side chain. The N-methyl group could be found at 3.72ppm. The results are total agreement with published data {2}.

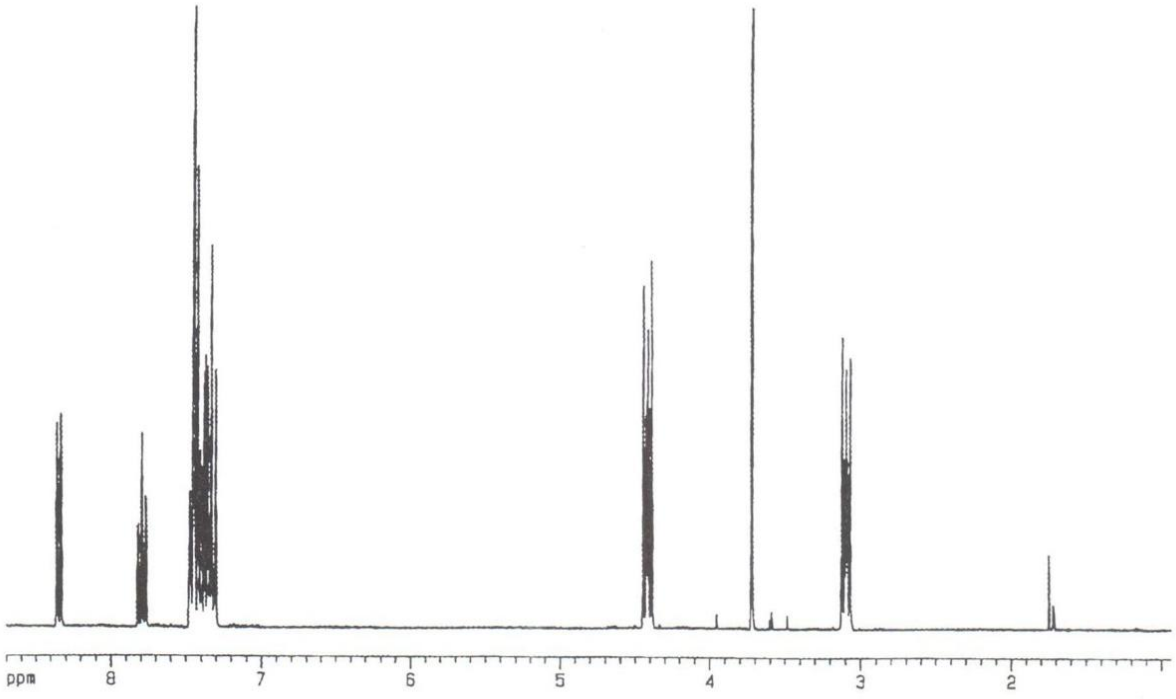


Fig. 2:[1] HNMR spectrum of Quinazoline alkaloids.

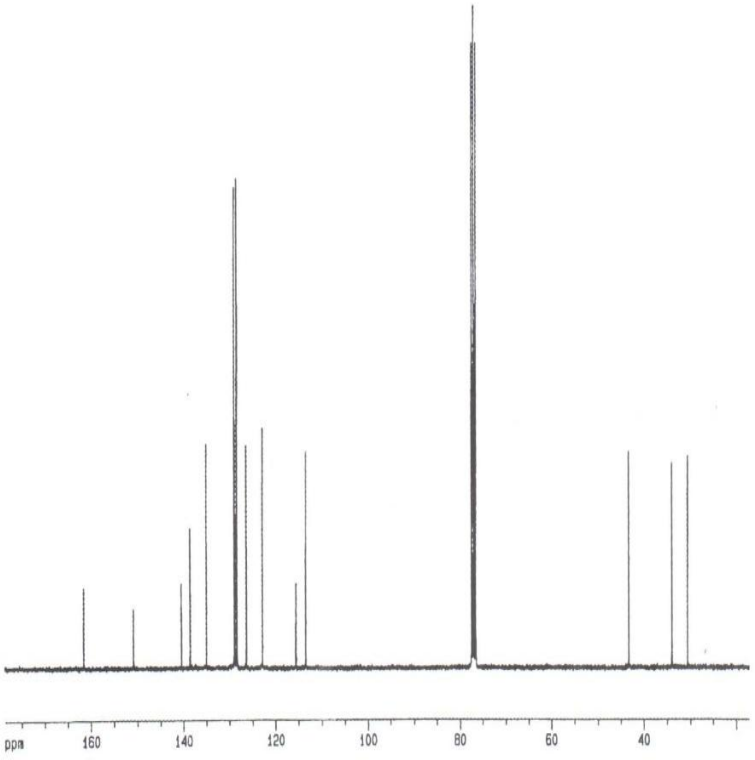


Fig. 3:[13] C-NMR spectrum of Quinaziline alkaloids.

The obtained experimental data of ^{13}C-NMR spectrum are as follows:-^{13}C-NMR ($CDCl_3$): 161. 57(C-4), 150.85 (C-10), 140.49 (C-2), 138.61 (C-14), 135.03 (C-7), 128.97 (C-15 and 19).128.87 (C-5), 128.46 (C16 and 18), 126.42 (C-17), 122.92 (C-6), 115.57 (C-9), 113.47 (C-8), 43.30 (C-13), 34.01 (c-12) and 30.64 (C-11). The results obtained from ^{13}C-NMR spectrum in figure 3 are total agreement with the published data {2}.

IV. CONCLUSION

Spectroscopic analysis method was used to identify the product. The synthesis process is a useful method for the preparation of quinazoline alkaloids with highly yield as well as easily accessible starting materials.

V. ACKNOWLEDGMENTS

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