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An Electron Bombardment Photodisintegration Spectrum Analysis of 3-Amino 6-methoxyl-2-MethylQuinazolin-4-(3H)-one Derivative

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Abstract: *Background:* The synthesis of new heterocyclic derivatives has attracted considerable attention. The explosive growth of heterocyclic chemistry is emphasized by the large number of research publications, monographs, and reviews. The heterocyclic organic compounds are extensively disseminated in natural and synthetic medicinal chemistry and are vital for human life. Looking at the previous studies on quinazolinones derivatives, only limited information is available on their mass spectral along with the preparation of novel quinazolin-4-(3H)-one derivatives. *Objective:* Aspiration of this investigation, was to synthesize a new 3-Amino-2-Methyl-6-methoxy-quinazolin-4-one was synthesized via the reaction between 2-Methyl-6-methoxy-benzo-1,3-oxazin-4-one and hydrazine hydrate and investigate their electron impact (EI) mass spectral disintegration. *Method:* The consolidation of 2-amino-methyl-5methoxybenzoate with acetic anhydride yielded the cyclic compound 2- methyl-4, 5-disubstituted-1, 3-benzo-oxazine-4-one which then produce a novel 2,3-disubstituted quinazolin-4 ones via the reaction with hydrazine hydrate. The compounds synthesized were enormously confirmed by means of Infrared, Nuclear Magnetic Resonance (1H and 13C), Gas Chromatography Mass Spectrophotometer and Elemental analysis. Discussion: The molecular ion of m/z 205 splints to give m/z 190 by loss of –NH group. The ion of m/z 190 was broken to give m/z 177 by losing CH group. This fragmented to m/z 162 by loss of –CH_3 group and then m/z 136 by loss of CN group. The loss of O gave m/z 120 which fragment to give m/z 93 by loss of –HCN and finally gave m/z 65 by loss of CO group. *Conclusion:* The electron impact ionization mass spectra of compound 2 show a weak molecular ion peak and a base peak ofm/z 205 resulting from a break up fragmentation. Compound 2 give a characteristic fragmentation pattern. From the examination of the mass spectra of compound 2, it was found that the molecular ion had fragmented to the m/z 190. The final disintegration led to ion of m/z 93 and ion of mass m/z 65, respectively.

Keywords: Mass Spectroscopy, Synthesis, Quinazoline-4-One, 2-Methyl 6-subtituted 1, 3-Benzo-Oxazine-4-One, 3-Amino-2-Methyl-6-methoxy-quinazolin-4-one, Nucleophile, Electron Impact Ionization Mass Spectra

1. Introduction

Compounds having all three activities, analgesic, anti─inflammatory, and anticonvulsant activities are not common. Quinazolines and encapsulated quinazolines displayed potent antimicrobial [1] and central nervous system (CNS) activities like analgesic, [2] anti-inflammatory [3] and anticonvulsant [4] activities. In view of these truth and to develop our earlier reported 2-phenyl-3-substituted quinazolines, [5] 2,3-disubstituted quinazolines, [6] 2-methyl-3-substituted quinazolin-4- (3H)-ones [7, 8], that showed good analgesic and anti-inflammatory activities.

The chemistry of heterocyclic compounds has been an fascinating field of study for a long time. The synthesis of new quinazolinone derivatives and exploration of their chemical and biological attitude have gained more importance in recent decades for biological, medicinal and agricultural reasons. Quinazolinones represents a significant class of heterocyclic compounds. Their derivatives possess a broad scope of biological activity in both agrochemicals and pharmaceuticals such as insecticidal, herbicidal, antibacterial, antifungal, analgesic, anti-inflammatory, antimalarial, antiviral, anti-HBV, antianexiety, anticancer, anti-HIV, antitubercular and anticonvulsant [8-22].

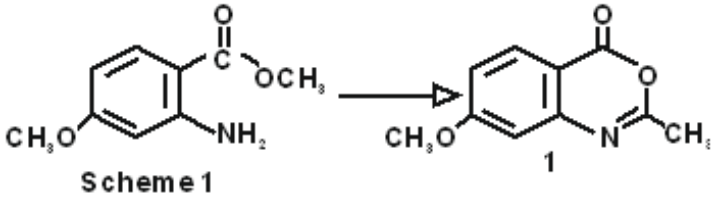
Molecular modeling studies on the interaction of one of the derivatives, 7-chloro-3- (4-chlorophenylsulfonyl) quinazoline-2,4(1H, 3H)-dione [23], with the mobile site of human heart chymase shows good feasibility and interaction. [24] The main synthetic scheme to quinazolinone compounds include the consolidation of anthranilamide (2-aminobenzamide), (3) with structurally diverse acid anhydrides, aldehydes or ketones in the presence of different catalysts [25, 26]. Cycloaddition of anthranilic acid derivatives with amines, imines, iminohalides have also been communicated [27, 28] There have been decription of microwave-supported synthesis of quinazolinones from anthranilic acid derivatives and from isatoic anhydride [29-31].

In the current research we aimed to synthesize this new 2, 3- quinazolin-4(3H)-ones derivatives, look into the electron bombardment photodisintegration spectrum analysis and their fragmentation pattern.

2. Materials and Methods

2.1. General Experimental Procedure

All reagents and solvents were purchased from sigma-Aldrich chemical supplier in Germany. A Kofler hot stage apparatus was used for the determination of melting points and are uncorrected. A Buck scientific IR M500 instrument was used in recording the IR spectra. The ^{1}H and ^{13}C NMR spectra were recorded in DMSO-*d*6 at 400MH_z with HAZ VOLATILE V2.M. Chemical shifts are reported in ppm relative to tetramethylsilane period. Gas chromatography mass (GC/MS) spectra were obtained on a Finingan MAT 44S mass spectrometer operating at electron impact energy of 70eV. Elemental analysis data agreed with the calculated values. Analytical thin layer Chromatography (TLC) was used to monitor the reactions.



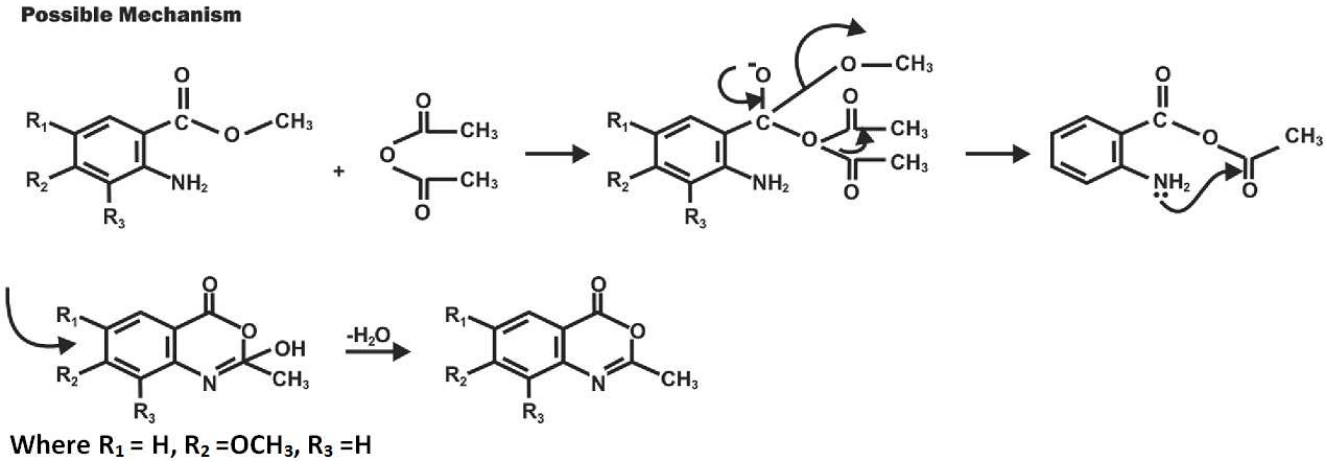
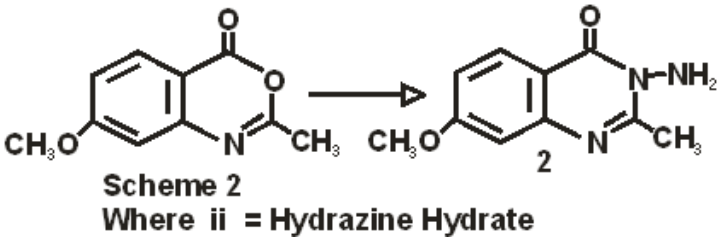


Figure 1. *Possible Mechanism For Synthesis of Compound 1.*



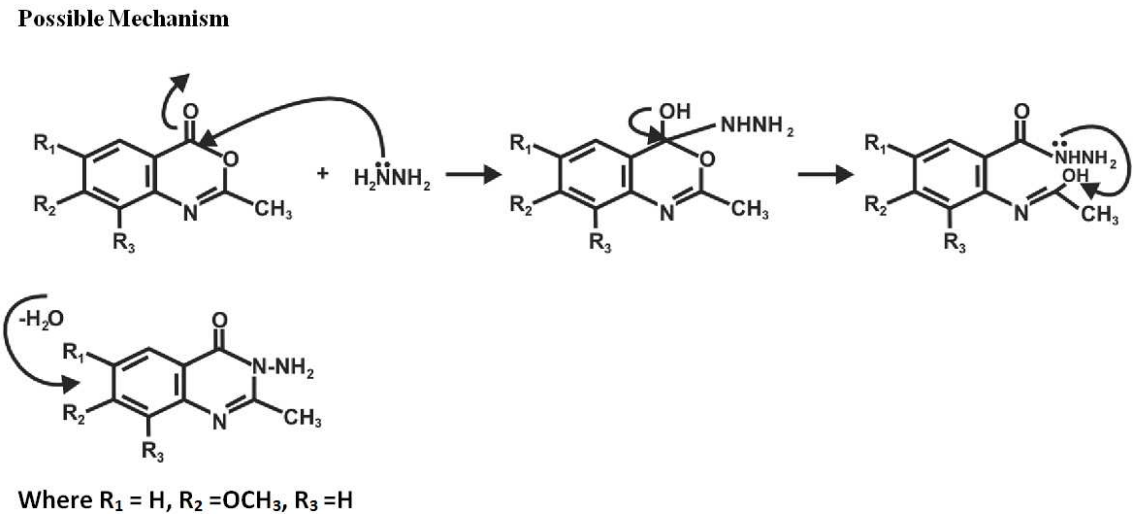


Figure 2. *Possible Mechanism For Synthesis of Compound 2.*

2.2. Synthesis of 6-methoxyl-2-methyl-4H-benzo [D] [1, 3]-oxazin-4-one (1)

This involved the consolidation of Methyl-2-amino-5-methoxyl-benzoate 2.11g (0.01mol) and 1.02g (0.01mol) acetic anhydride in 30ml ethanol medium were reacted. The reaction was heated under reflux with stirring using a magnetic stirrer until the reaction mixture showed no trace of starting material when the TLC was developed (2 hours).

At the end of the reaction, work up was done. Ethanol was removed in vacuum and the crude mixture was poured into 50ml of ice water on a cold water bath. The mixture was stirred for 30 minutes filtered and extracted into ethyl acetate and allowed to evaporate at room temperature to give solid products which were recrystallized from hexane or dichloromethane-hexane mixture. Yield was 1.99g (93%), mp: 140-145°C.

2.3. Synthesis of 3-amino-6-methoxy-methyl Quinazolin-4 (3H)-one (2)

The contraction of equimolar amounts of 2-methyl-5-methoxyl-4H-benzo [D] [1, 3] –oxazine-4-one (1.06g, 0.005mol) and hydrazine hydrate (0.93g, 0.01mol) were added to 30ml boiling ethanol with stirring using a magnetic stirrer until the reaction mixture showed no trace of starting material when the TLC was developed (3 hours).

At the end of the reaction, the reaction mixture was crystallized in vacuum under reduced pressure using rotary evaporator. The white precipitate formed was then filtered, washed three times with 20ml of distilled water [20ml x 3]. The white crystals were dried and recrystallized from dimethylformamide (DMF) to give pure 3-amino-2-methylquinazolin-4 (3H) –one. Yield was 0.98g (90%) mp: 95-98°C.

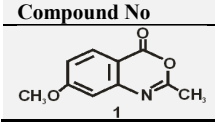
3. Result

The initiation of 2-Amino substituent is a good strategy to improve the chemical balance of benzoxazinone. Because to the pharmacological properties of 4(3H)-quinazolinone derivatives, 2,3-disubstituted derivatives of quinazoline-4-one was synthesized via the synergy of the benzoxazinone derivative with nitrogen nucleophile with the aim of achieving a more pricise information about the course of the reaction and some vital pharmaceutical compounds. The reaction of 4, 5-disubstituted derivatives of methylanthranilate and acetic anhydride yielded the cyclic compound 2-methyl-6, 7-dimethoxyl-benzo-1, 3-oxazin-4-one. The reaction of this compound with hydrazine hydrate produced the novel 2, 3-disubstituted quinazoline-4-one.

Table 1. *Characterization and Physical data of Synthesized Compounds.*

Compound No	Solvent	Formula M. wt	Analysis% Calc/Found	
			C	H
1	Ethanol	$C_{10}H_{11}N_3O_2$ (191.102)	45.30 45.25	4.87 4.88
2	Ethanol	$C_{11}H_{13}N_3O_3$ (205.109)	46.22 46.27	4.31 4.35

Table 2. *^{13}C-NMR of Synthesized Compounds.*

Compound No	δ (ppm) Carbon atom number
 1	168.16(C-2), 155.70(C-5), 149.34(C-8) 140.27 (C-1), 113.26 (C-6), 100.45 (C-4) 100.03 (C-3), 100.01 (C-7), 56.43 (C-10), 51.63 (C-11), 16.85 (C-9)

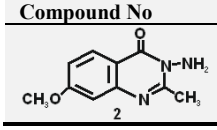
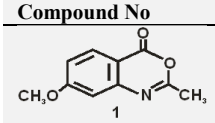
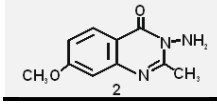
Compound No	δ (ppm) Carbon atom number
 2	160.26 (C-2), 155.30 (C-5), 153.48 (C-1) 148.05 (C-8), 142.66 (C-6), 113.36 (C-1) 107.15 (C-3), 104.55 (C-7), 56.71 (C-10) 56.32 (C-11), 22.47 (C-9)

Table 3. *^{1}H-NMR of Synthesized Compounds.*

Compound No	δ (ppm) Carbon atom number
 1	7.15 (s, 1H), 6.39 (s, 1H), 3.74 (s, 3H), 3.58 (s, 3H)
 2	7.40 (s, 1H), 7.11 (s, 1H), 5.83 (s, 2H), 3.92 (s, 3H), 2.56 (s, 3H)

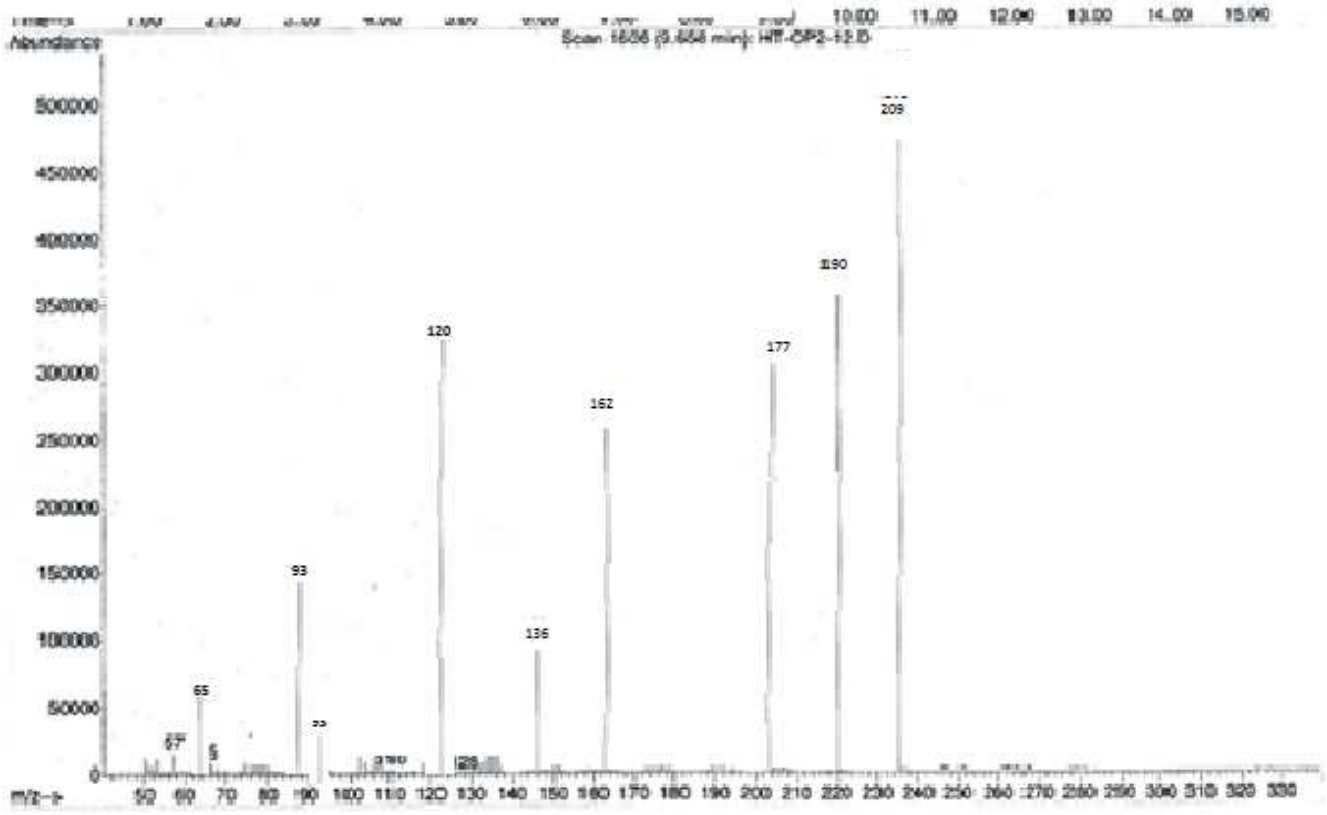


Figure 3. *70ev mass spectrum of compounds 2.*

Table 4. *EI Mass Spectra (70ev) of Compound, 2 m/z (relative intensity, %).*

Compound	M+	M-	m/z	Other Ions
2	$[C_{10}H_{11}N_3O_2]^{+\cdot}$ 205 (16)	NH	$[C_{10}H_{10}N_2O_2]^{+}$ 190 (12)	191 (8), 136 (10), 102 (3), 93 (5), 78 (3), 57 (3)
		CH	$[C_9H_9N_2O_2]^{+}$ 177 (10)	
		CH_3	$[C_8H_6N_2O_2]^{+}$ 162 (14)	
		CN	$[C_7H_6NO_2]^{+}$ 136(10)	
		O	$[C_7H_6NO]^{+}$ 120 (4)	
		HCN	$[C_6H_5O]^{+}$ 93(8)	
		CO	$[C_5H_5]$+ 65(2)	

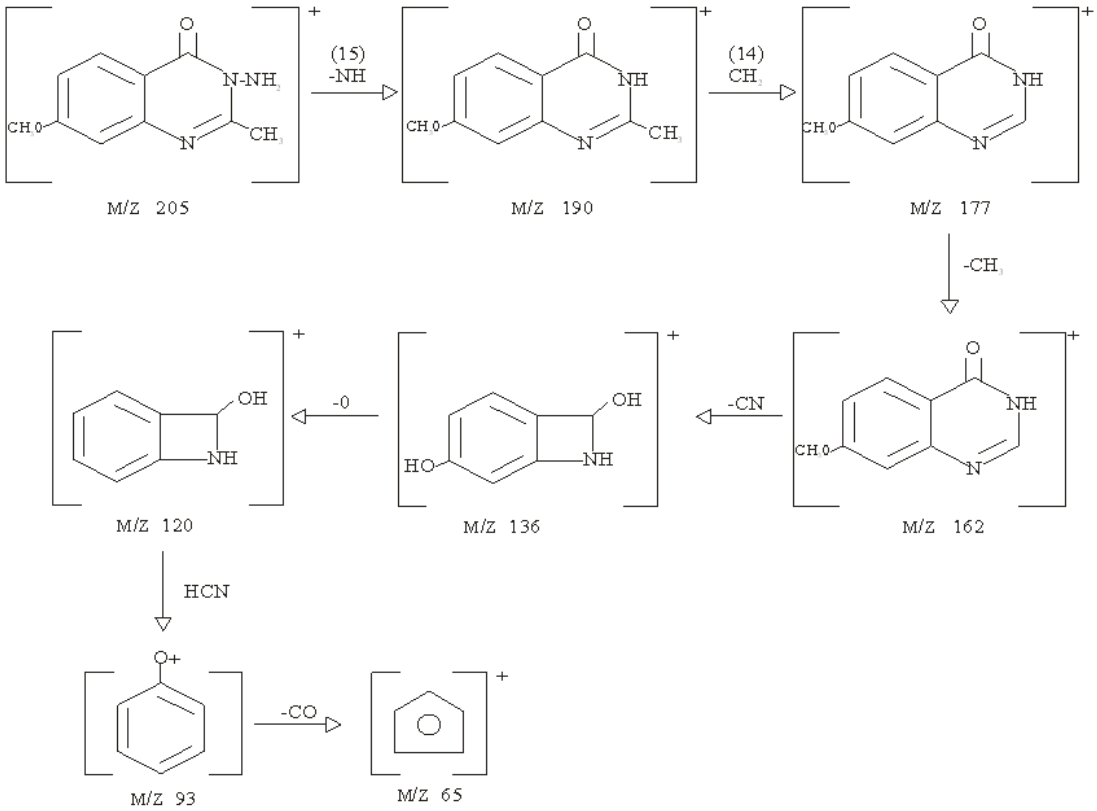


Figure 4. *Main Photodissociation Pathway of Compound 2.*

3.1. Characterization of 6-methoxl-2-methyl-4H–benzo[d] [1,3]–oxazine–4–one (1)

^{1}H NMR (400MHz, DMSO) δ 7.16 (s, 1H), 6.40 (s, 1H), 3.78 (s, 6H), 3.68 (s, 3H), ^{13}(NMR (400MHz, DMSO) δ 168.28, 155.80, 149.23, 140.28, 113.37, 100.56, 100.05, 56.94, 56.94, 56.13, 51.93, 16.95: IR (KBr, cm^{-1}) 3381, 3203, 3135, (NHz), 3018 (CH aromatic), 2951, 2871, 2718 (CH aliphatic), 1662 (C=O). Anal. Cal 1159 (C-O) for $C_{11}H_{11}NO_4$; C 62.20; H 5.18. Found: C 62.10, H 4.98.

3.2. Characterization of 6-methoxyl-2-methyl-4H–benzo[d] [1,3]–oxazine–4–one (2)

^{1}H NMR (400 MHz, DMSO) δ 7.41 (s, 1H), 7.10 (s, 1H), 5.80 (s, 2H), 3.90 (s, 6H), 2.58 (s, 3H), ^{13}C NMR (400MHz, DMSO) δ 160.28, 155.29, 154.57, 149.07, 143.77, 113.65, 108.24, 105.64, 56.80, 56.63, 22.58, IR (KBr, cm^{-1}) 3301 (NH_2), 1622 (C=O), Anal. Cal. for $C_{11}H_{13}N_3O_3$; C 56.11, H 5.53; Found, C 56.40, H 5.41.

4. Discussion

Structural clarifications of compounds synthesized were identified by correct elemental analysis and careful inspections of spectral data. Looking at the ^{1}H NMR spectra of the compounds synthesized, compound 1 displayed a singlet signal at: δ 3.74 accredited to methoxy group and singlet at δ 3.58 which was due to methyl group. Other singlets appeared at δ7.15 and 6.39 ascribed to aromatic protons. Also, ^{1}H NMR spectrum of compound 2 showed a symbolic signal at δ 2.56 (singlet) equivalent to methyl group and singlet at: δ 3.92 for methoxy group. Two singlets appeared at δ7.40 and 7.11 traced to aromatic protons. Another signal appeared at 5.83 which was associated to the protons of the amino group. For the IR spectra, compound 1 was identified by absence of υ NH_2 and presence of υ C-O stretch in 1100cm^{-1} region of the compound. Compound 2 was represented by absence of υ C-O and presence of υNH_2 in 3302cm^{-1} region of the compound.

The ^{13}C NMR spectrum of compound 1, revealed signals at δ16.85, 51.63 and 56.43 credited to methyl and the two methoxy groups appropriately, while the aromatic carbon atoms appeared between δ values 100.01-168.16 with the carbonyl carbon atom appearing as the highest δ value of 168.16. Similarly, compound 2 showed signals at δ22.47, 56.32 and 56.71 ascribed to methyl and the two methoxy groups consequently, while the aromatic carbon atoms appeared between δ values 104.55-160.26, with the carbonyl carbon atom appearing as the highest δ value of 160.26.

The ^{13}C nuclear magnetic resonance revealed low δ values for the aliphatic carbons. This is because the alkyl group is electron donating and hence produces a shielding effect which makes the carbon atom to resonate at low δ values. The aromatic and the carbonyl carbon atoms appeared at high δ values. This is because the aromatic ring is electron withdrawing and the aromatic carbons are highly upfield and resonate at high frequency. The electronegative effect of the oxygen atom on the carbonyl group makes the carbonyl carbon to appear at higher δ value.

Compound 2, molecular formula $C_9H_8ClN_3O$ (*m/z* 205.109 [M^+]), had NMR data similar to1, except for an additional signal at δ_H 5.80 in the ^1H NMR spectrum which was allocated to the amino protons (2H) (Table 2). Table 4 lists the m/z (relative abundance,%) values of principal fragments of the studied compound, while figure 1 illustrates the mass spectrum of the compound shows a molecular ion of m/z 205 corresponding to the molecular mass of the compound. The molecular ion of m/z 205 fragments to give m/z 190 by loss of –NH group. The ion of m/z 190 was broken to give m/z 177 by losing CH group. This fragmented to m/z 162 by loss of $–CH_3$ group and then m/z 136 by loss of CN group. The loss of O gave m/z 120 which fragment to give m/z 93 by loss of –HCN and finally gave m/z 65 by loss of CO group.

5. Conclusion

The current work displayed that the mass spectra of compound 2 has relatively small molecular ion and peaks typical of a fractionalization and reorganization processes type atomization. Compound 2 give a characteristic disintegration pattern with a very stable fragment of benzopyrazolone (m/z 205).

6. Recommendations

The Electron Impact Ionization Mass Spectra of 3-Amino 5,6-dimethoxyl-2-methyl quinazolin-4-(3H)-one [32] and 3-Amino 6-Chloro-2-methyl Quinazolin-4-(3H)-one [33] Derivatives with the characteristic fragmentation pattern have been done. For better information, I recommend that characteristic fragmentation pattern be done for compounds synthesized.

Conflicts of Interest/Competing Interests

All the authors do not have any possible conflicts of interest.

Availability of Data and Material

There are availability of data and materials.

Code Availability

There is no code available in this research.

Authors' Contributions

Dr. Osarumwense Osarodion Peter design the work, Ayedun Hassan and Arigbede John carefully assisted in the laboratory work and in the spectral analysis of the samples.

Author Declaration

The authors hereby declare that the work presented in this article is original and that any liability for claims relating to the content of this article will be borne by them.

Ethics Approval and Consent to Participate

Ethic approval, consent to participate and the procedure used was approved by the Ethic approval committee of Ondo State University of Science and Technology, Okitipupa, Ondo State, Nigeria.

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