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A green Synthesis of 2-Substituted Quinazolin-4(3H)-ones

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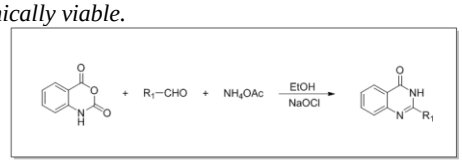
GRAPHICAL ABSTRACT

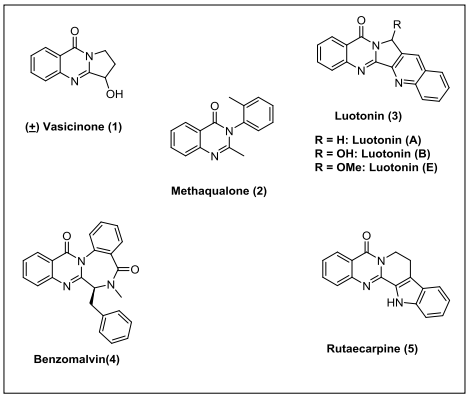
Various 2-Substituted Quinazolin-4(3H)-one's derivatives in good to excellent yield have been prepared in one–pot reaction by condensation of Isatoic anhydride, an aldehyde, and ammonium acetate using NaOCl as an oxidant in ethanol at 80-85°C, this strategy is simple and catalyst-free. The reaction is green and economically viable.



Keywords: Isatoic anhydride, NaOCl, One-pot, Economically viable, Green chemistry, 2-Substituted Quinazolin-4(3H)-ones.

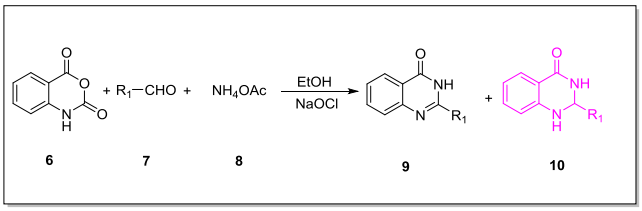
INTRODUCTION

Quinazolinones moieties are fused heterocyclic compounds that form building blocks of various natural products and synthetic analogs possessing an extensive array of biological activities [1-8]. The quinazolinone moiety is a very important pharmacophore that have many application in pharmaceutical chemistry and it show a diverse range of pharmacological activities, including anti-inflammatory, antihypertensive, anticancer, anticonvulsant, and antibacterial activities. For example, vasicinone (Figure 1) is a pyrrolo[2,1-b]quinazoline alkaloid, isolated from the aerial parts of adhatoda (Justicaadhatoda, also known as Adhatoda vasica Nees), a plant used extensively in indigenous medicine for treating colds, coughs, bronchitis, and asthma [9]. Methaqualone (2) is a hypnotic/sedative that was used for the treatment of insomnia until it was banned and listed as an addictive drug in 1985. Luotonin A (3), commonly found in the Chinese plant Peganum nigellastrum, is a human DNA topoisomerase I inhibitor and displays cytotoxicity towards the murine leukemia P388 cell line (IC50 = 1.8 µg/mL) by stabilizing the topoisomerase I–DNA complex [10,11]. Benzomalvin A (4) is an inhibitor of human neurokinin-1, isolated from a fungal culture of a Penicilliumspecies [12,13]. Rutaecarpine (5) is the major alkaloid component of the traditional Chinese herbal drug Wu-chu-yu (evodia fruit; Evodia rutaecarpa), used extensively as a remedy for headaches, cholera, and dysentery [14-17].



Because of their diverse range of pharmacological activities, 2-substituted quinazoline-4(3*H*)-ones have attracted interest from the synthesis community, several synthetic methodologies are available for the preparation of 2-aryl- and 2-alkylquinazolin-4(3*H*)-ones involves two stages: generally the condensation of 2-aminobenzamide with an aliphatic or aromatic carboxylic acids, acid chlorides, their nitriles, imitates, and orthoester derivatives have been used for 2-substituted quinazolin-4(3*H*)-ones. In the other hand direct condensation of 2-aminobenzamide with aldehyde and subsequent oxidation with a reagent such as copper (II) chloride, 2,3-dichloro-5,6-dicyano-1,4-benzoquinone, potassium permanganate, or manganese dioxide.[7] Primary alcohols and *o*-aminobenzamides by an iridium-catalyzed dehydrogenation has also been recently reported [22]. Isatoic anhydride [2*H*-3,1-benzoxazine-2,4(1*H*)-dione] has been widely used in syntheses of 2-substituted quinazolinones by condensation with amines and aldehydes in the presence of iodine, silica, sulfuric acid, Nafion-H, ionic liquids, or Lewis acids such as gallium(III) triflate. However, these reactions give the corresponding 2,3-dihydro quinazoline- 4(1*H*)-ones as major byproducts [23].

Several of these reported procedures for the preparation of 2-substituted quinazolinone derivatives were neither versatile nor compatible with differently substituted starting materials. They are associated with many practical problems. For example, the use of high temperature for polyphosphoric acid-mediated condensation the reaction led to the formation of by-product and quinazolinone in low yield. Though metal catalysts give better yields, the prohibitive cost of the catalysts make them unsuitable for industrial application. We report a simple, efficient, and high-yielding preparation of 2-substituted quinazolinones 9 by the oxidative cyclization with NaOCl reaction of isatoic anhydride (6), aldehydes 7 and Ammonium acetate (Scheme 1).



Scheme 1: Synthesis of 2-Substituted Quinazolinones derivatives

RESULTS AND DISCUSSIONS

2-substituted Quinazolinones derivatives were prepared by refluxing Isatoic anhydride, various substituted aldehydes, and ammonium acetate using NaOCl as an oxidant in ethanol to afford good to excellent yields (Scheme 1). The completion of the reaction was monitored by TLC (EtOAc in Hexane) as eluent. This methodology was applied to synthesize a variety of 2-Substituted quinazolinones derivatives and the results are summarized in Table 3**.**

Initially, we investigated various reaction conditions in the model reaction (Tables 1 and 2). First, we focused our attention on screening various solvents in an attempt to improve the yield (Table 2), solvents such as water, 1,4-Dioxane, methanol, N, N-dimethyl formamide, and ethanol were examined, but all were found to be less effective solvents except methanol and Ethanol (entries 1−6). No reaction was observed in toluene and water because of the poor solubility of isatoic anhydride (6). However, the reaction in ethanol showed a clean conversion in around 1-2 hours, and the isolated yield was improved to 85% (entry 3). From these studies, the optimal conditions were established for the selective formation of 2-aryl quinazolinone derivatives.

Entry	Solvent	Temp(°C)	Time(h)	Yield (%)	
				9	10
1	H2O	80-85	24	-	-
2	Methanol	80-85	3	50	05-Oct
3	Ethanol	80-85	2.5	85	<5
4	Toluene	80-85	10	0	0
5	DMF	80-85	10	20	05-Oct
6	1,4-Dioxane	80-85	4	50	05-Oct

Table 1: Solvent Screening for the Synthesis of quinazolinone 9 in the presence of NaOCl.

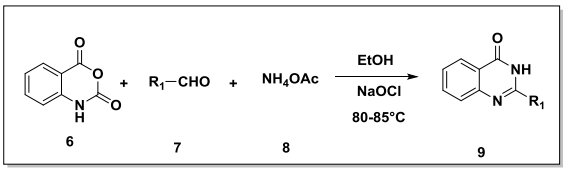
As shown in Table 2, when the reaction was conducted in the absence of an oxidant, a mixture of products was obtained, the desired quinazoline 9 was formed as the minor product (<10%), with the 2, 3-dihydro quinazoline-4(1*H*)-one derivative 10 as the major product on TLC (80%; Table 1, entry 1). we carried out reactions in the presence of various oxidants (entries 2−7). Among these, the sodium hypochlorite reaction showed an enhanced selectivity towards the desired product, giving quinazolinone 9 in 80% yields. It should be noted that 1.0-1.5 E.qt NaOCl was efficient enough to oxidize the reaction, and increasing the amount of oxidant did not improve the yield significantly, finally, we achieved an optimized condition using 1.0-1.5 mole E.qt NaOCl as the oxidant in ethanol [24-27].

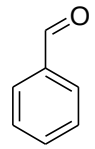
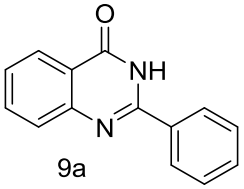
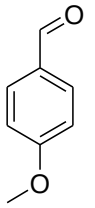
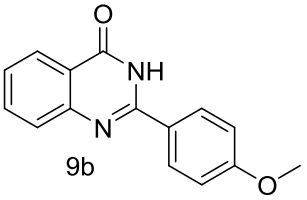
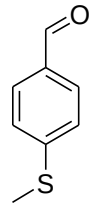
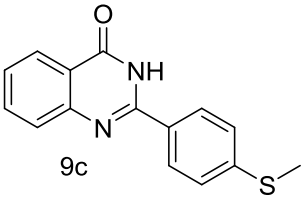
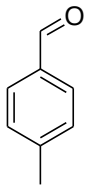
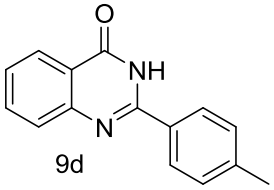
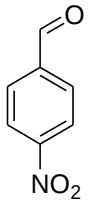
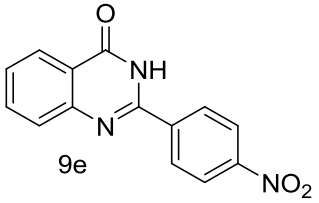
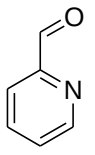
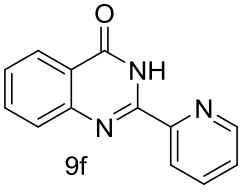
S. No	Oxidative Reagent	Eq.	Time (h)	Temp°C	Yield (%) b	
					9	10
1	No reagent	-	24	80-85	-	70
2	N-Iodosuccinimide (NIS)	1.1	24	25-30	30-40	05-Oct
3	N-Chlorosuccinimide (NCS)	1.1	12	25-30	30-40	30-35
4	N-Bromosuccinimide (NBS)	1.1	0.5	25-30	70-75	0
5	Dess Martin periodinane (DMP)	1.1	3	25-30	40-50	Oct-15
6	(Diacetoxyiodo)benzene (DIB)	1.1	3	25-30	45-50	Oct-15
7	Iodine (I2)	1.1	3	25-30	40-45	05-Oct

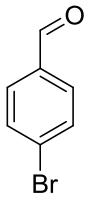
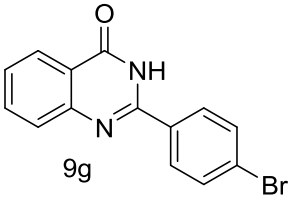
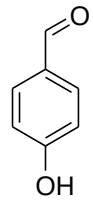
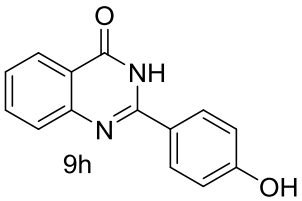
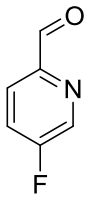
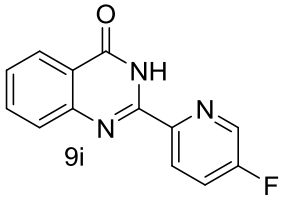
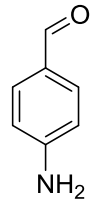
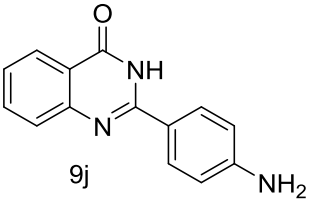
Table 2: Oxidative reagent screening for the Synthesis of quinazoline 9

The scope of the method was evaluated by using a variety of substituted aryl aldehydes 7 under the optimized conditions, and the results are shown in Table 3, the desired products 9a–j were obtained in high yields in all cases, regardless of the nature of the substituents on the aldehydes interestingly, neither electron-donating (methyl, 4-hydroxy, 4-methoxy and 4-amino) nor electron-withdrawing groups (4-Bromo or 4-nitro) on the aldehydes had any effect on the reaction profile and yield (Table 3, entries 1−8). Heteroaromatic aldehydes also gave the corresponding Quinazolinones in good yields (entries 6 and 9).

Scheme-3



Entry	Aldehyde	Product	Time (h)	Yield (%)b
1		 9a	2.5	76
2		 9b	3	82
3		 9c	2.5 h	80
4		 9d	2.5	79
5		 9e	2	75
6		 9f	2	68

7		 9g	2	71
8		 9h	2.5	69
9		 9i	2	74
10		 9j	2	73

[a]Reaction conditions: Isatoic anhydride (6) (1.0 mmol), aldehyde (7) (1.1 mmol), Ammonium acetate (8) (3.0 mmol), Sodium hypochlorite (1.5 mmol), Ethanol (10 vol) 80-85°C. [b] Yield of isolated products

Table 3: Preparation of 4-(3*H*) Quinazolinones under optimized conditions

CONCLUSION

We have developed an efficient oxidative synthesis of 2-substituted Quinazolinones from isatoic anhydride (6), ammonium acetate, and various aldehydes (**7**) in good to excellent yields with NaOCl. The reaction proceeds under mild conditions without needing an expensive catalyst, and it provides the desired Quinazolinones with high selectivity.

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SUPPLEMENTARY INFORMATION

Synthesis of 2-substituted quinazolinone derivative 9a

To a mixture of isatoic anhydride (0.1 g, 0.61 mmol), benzaldehyde (0.078 g, 0.67 mmol) and NH_4Cl(0.036 g, 1.84 mmol) in 10 mL of ethanol was added NaOCl (0.063 g, 0.91 mmol). The resulting mixture was stirred for 2-3 h at 80-85°C. The completion of the reaction was monitored by TLC (ethyl acetate in hexane). The reaction mass poured into water and the product was precipitated as a solid, the content was filtered and washed with water twice and it was dried and recrystallized from ethanol to give pure compound 9a. The same procedure was used for all the compounds.

Spectral data

Analytical data of 2-Substituted-4(3H)-Quinazolinones (9a-m)

2-Phenylquinazolin-4(3*H*)-one (9a): White solid; Yield: 84.6 %; Mp: 236-238°C; ^{1}H-NMR (DMSO-d_6, 400 *MHz*): 7.53-7.58 (m, 4H,ArH), 7.74 (d, *J*=7.6 Hz, 1H, ArH), 7.82-7.86 (m, 1H, ArH), 8.15-8.19 (m, 3H, ArH), 12.54 (s, br,1H, NH); ^{13}C-NMR (DMSO-d_6,100 *MHz*): 120.9, 125.8, 126.9, 127.4, 127.7, 128.1, 128.6, 131.4,132.7, 134.6, 152.3, 162.2; MS: *m/z*=223.1 [M +H];

2-(4-Methoxyphenyl) quinazolin-4(3*H*)-one (9b): White Solid; Yield: 90.1%; Mp: 245.5-247.2°C; ^{1}H-NMR ($CDCl_3$, 400 *MHz*): 3.91 (s, 3H, OCH_3), 7.06 (dd, *J*=6.8, 2.0 Hz, 2H, ArH), 7.45-7.49 (m, 1H, ArH), 7.76-7.81 (m, 2H, ArH), 8.06 (dd, *J* = 6.8, 2.0 Hz, 2H, ArH), 8.30 (d, *J*=7.8 Hz, 1H, ArH), 10.04 (s, br, 1H, NH); ^{13}C-NMR ($CDCl_3$+ DMSO-d_6, 100*MHz*): 54.1, 112.6, 119.7, 123.9, 124.6, 124.7, 126.1, 128.2,132.9, 148.1, 150.8, 160.8, 161.7; MS: *m/z*=253.1[M +H];

2-(4-(Methylthio)phenyl)quinazolin-4(3*H*)-one(9c): White solid; Yield: 93.2%; Mp: 237-239°C; ^{1}H-NMR (DMSO-d_6, 400 *MHz*): 2.55 (s, 3 H, SCH_3), 7.40 (d, *J*=8.4 Hz, 2H, ArH), 7.50 (t, *J* = 7.2 Hz, 1H, ArH), 7.72 (d, *J*=7.6 Hz, 1H, ArH), 7.83 (t, *J* =7.6 Hz, 1H, ArH), 8.13-8.16 (m, 3H, ArH), 12.48 (s, br, 1H,NH); ^{13}C-NMR (DMSO-d_6, 400 *MHz*): 14.1, 120.8, 125.1, 125.8, 126.4, 127.4, 128.0, 128.6, 134.6, 143.0, 148.7, 151.78, 162.2; MS: *m/z*=269.0[M +H].

2-(p-Tolyl)quinazolin-4(3*H*)-one(9d): White solid; Yield: 83.2%; Mp: 239-240°C; ^{1}H-NMR (DMSO-d_6, 400 *MHz*): 2.39 (s, 3H, CH3), 7.36 (d, *J*=7.8 Hz, 2H, ArH), 7.51 (t, *J*=7.8 Hz, 1H, ArH), 7.72 (d, *J*=7.8 Hz, 1H, ArH), 7.81-7.85 (m, 1H, ArH), 8.10 (d, *J*=7.8 Hz, 2H, ArH), 8.15 (d, *J*=7.3 Hz, 1H, ArH), 12.46 (s, br, 1H, NH); ^{13}C-NMR (DMSO-d_6, 100*MHz*): 21.0, 120.9, 125.8, 126.4, 127.4, 127.6, 129.2, 129.9, 134.5, 141.4, 148.8, 152.2, 162.2; MS: *m/z*=237.1[M +H].

2-(4-Nitrophenyl) quinazolin-4(3*H*)-one (9e): Yellow solid; Yield: 84.0%; Mp: >300 °C; ^{1}H-NMR (DMSO-d_6, 400 *MHz*): 7.59 (t, *J*=8.0 Hz,1H, ArH), 7.80 (d, *J*=8.0 Hz, 1H, ArH), 7.86-7.91 (m, 1H, ArH), 8.19 (d, *J*=7.2 Hz, 1H, ArH),8.37-8.43 (m, 4H, ArH), 12.91 (s, br, 1H, NH); ^{13}C-NMR (DMSO-d_6, 100 *MHZ*): 121.2, 123.6,125.9, 127.3, 127.8, 129.3,134.8, 138.5, 148.3, 148.9, 150.7, 162.0; MS: *m/z*=268.1[M +H].

6. 2-(Pyridin-2-yl) quinazolin-4(3*H*)-one(9f): Brown solid; Yield: 79.2%; Mp: 168-170°C; ^{1}HNMR (DMSO-*d6*, 400 *MHz*): 7.58 (t, *J*=7.6 Hz, 1H, ArH), 7.65-7.68 (m, 1H, ArH), 7.81 (d, *J*=8.0 Hz, 1H, ArH), 7.89 (t, *J*=7.6 Hz, 1H, ArH), 8.06-8.09 (m, 1H, ArH), 8.19 (d, *J*=7.6 Hz, 1H, ArH), 8.47 (d, *J*=8.0 Hz, 1H, ArH), 8.76 (d, *J*=5.2 Hz, 1H, ArH), 11.83 (s, br, NH); ^{13}C-NMR (DMSO-*d6*, 100 *MHz*): 122.0, 122.1, 126.1,126.5, 127.2, 127.7, 134.7, 137.9, 148.6, 148.9, 149.9, 160.7; MS: *m/z*=224.1[M +H].

7. 2-(4-Bromophenyl) quinazolin-4(3*H*)-one (9g):
White solid; Yield: 80.4%; Mp: 295-296°C; ^{1}H-NMR (DMSO-d_6, 400 *MHz*): 7.52-7.56 (m, 1H, ArH), 7.74-7.78 (m, 2H, ArH), 7.83-7.87 (m,

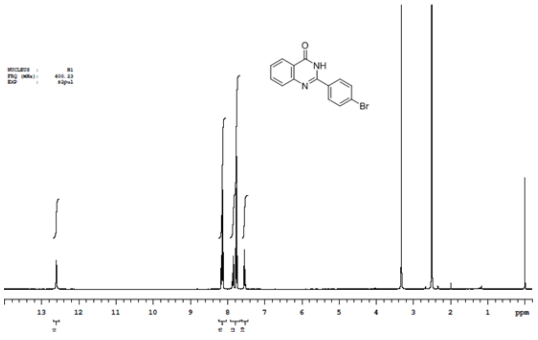
1H, ArH), 8.12-8.17 (m, 3H, ArH), 12.60 (s, br,1H, NH); ^{13}C-NMR (DMSO-*d*6, 100 *MHz*): 121.0, 125.2,125.8, 127.5, 129.8, 131.6, 131.9, 134.6, 148.5, 151.4, 162.1; MS: *m/z*=301.0 [M +H].

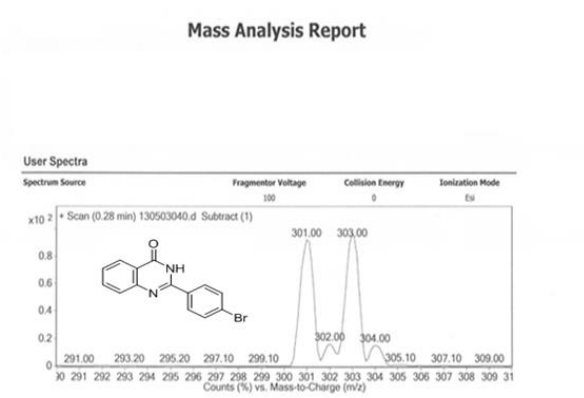
8. 2-(4-Hydroxyphenyl) quinazolin-4(3*H*)-one (9h): White solid; Yield: 80.0%; Mp:261-263°C; 1HNMR (DMSO-*d6*, 400 *MHz*): 6.89 (d, *J*=8.8 Hz,2H, ArH), 7.47 (t, *J*=8.0 Hz, 1H, ArH), 7.67 (d, *J*=7.6 Hz, 1H, ArH), 7.78-7.89 (m, 1H, ArH),8.07-8.12 (m, 3H, ArH), 10.14 (s, br, 1H, OH), 12.30 (s, br, 1H, NH); ^{13}C-NMR (DMSO-d_6,100 *MHz*): 115.3, 123.2, 125.8, 125.9, 127.2, 129.5, 134.5, 149.0, 160.5, 184.3; MS: *m/z* =239.1[M +H].

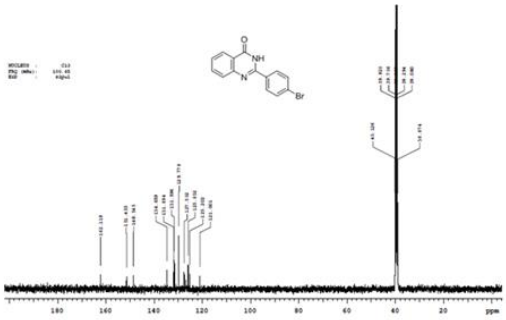
9. 2-(5-Fluoropyridin-2-yl) quinazolin-4(3*H*)-one (9i): Brown solid; Yield: 80.2%; Mp: 180-183°C; 1HNMR ($CDCl_3$, 400 *MHz*): 7.51-7.56 (m, 1H, ArH), 7.61-7.65 (m, 1H, ArH), 7.77-7.80 (m, 2H, ArH), 8.35 (d, *J*=7.4 Hz, 1H, ArH), 8.51 (d, *J*=2.9 Hz, 1H, ArH), 8.63 (dd, *J* = 8.8, 4.8 Hz, 1H, ArH), 10.76 (s, 1H, br, NH); ^{13}C-NMR ($CDCl_3$, 100 *MHz*): 122.2, 123.7(d, *J*=5.4 Hz), 124.4(d, *J*=18.4 Hz), 126.7, 127.4, 127.9, 134.6, 137.2 (d, *J*=25.2 Hz), 144.7, 147.9, 148.9, 161.1 (d, *J*=260.0 Hz), 161.3; MS: *m/z*=242.1[M +H]

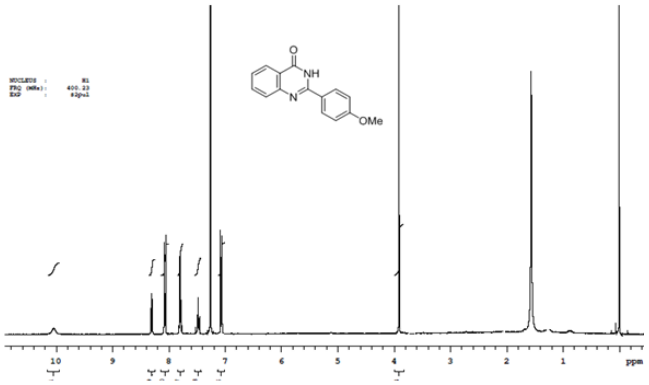
10. 2-(4-Aminophenyl) quinazolin-4(3*H*)-one (9j): Yellow solid; Yield: 78.2%; Mp: 250-254°C; ^{1}H-NMR (DMSO-*d6*, 400 *MHz*): 5.83 (s, 2H, NH_2), 6.62 (d, *J*=8.8 Hz, 2H, ArH), 7.40 (t, *J*=6.9 Hz, 1H, ArH), 7.61 (d, *J*=8.3 Hz, 1H, ArH), 7.76 (t, *J*=6.8 Hz, 1H, ArH), 7.95 (d, *J*=8.8 Hz, 2H, ArH), 8.08 (dd, *J*=6.9, 1 Hz, 1H, ArH), 12.06 (s, br, 1H, NH); ^{13}C-NMR (DMSO-d_6, 100 *MHZ*): 113.0, 125.3, 125.8, 126.9, 129.1, 134.4, 152.2, 162.4; MS: *m/z*=238.1[M +H].

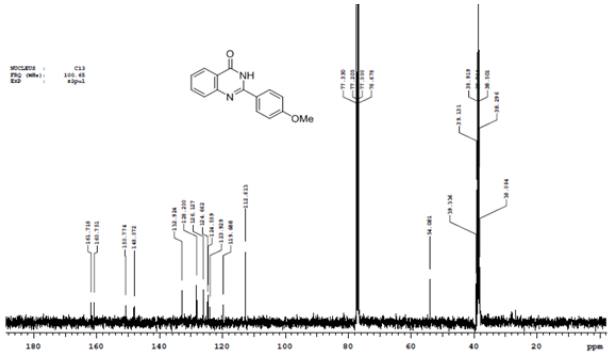
^{1}H-NMR, ^{13}C-NMR, and Mass spectra of some selected compounds



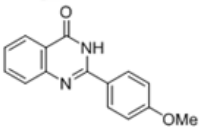








Mass Analysis Report



User Spectra

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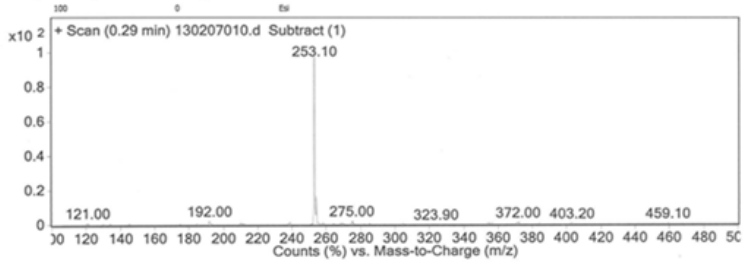

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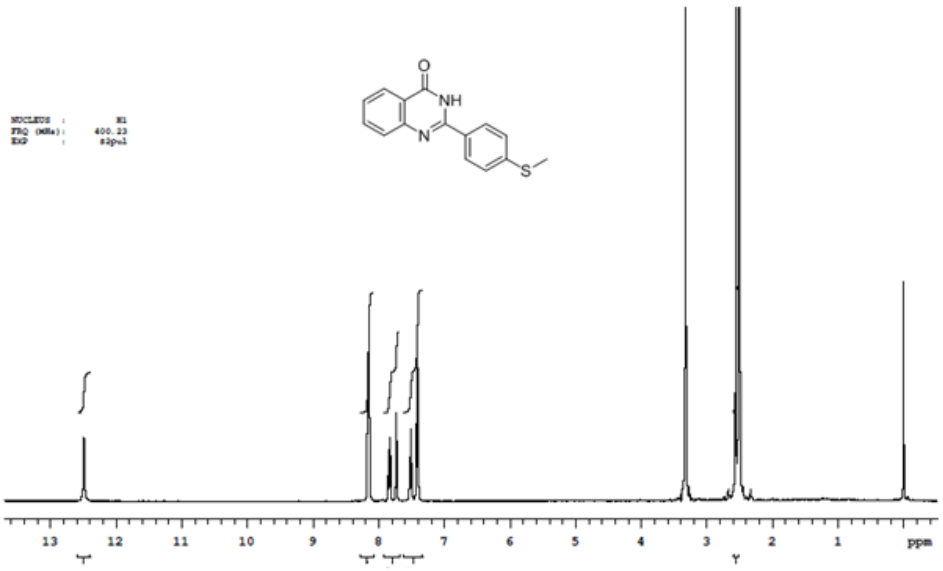

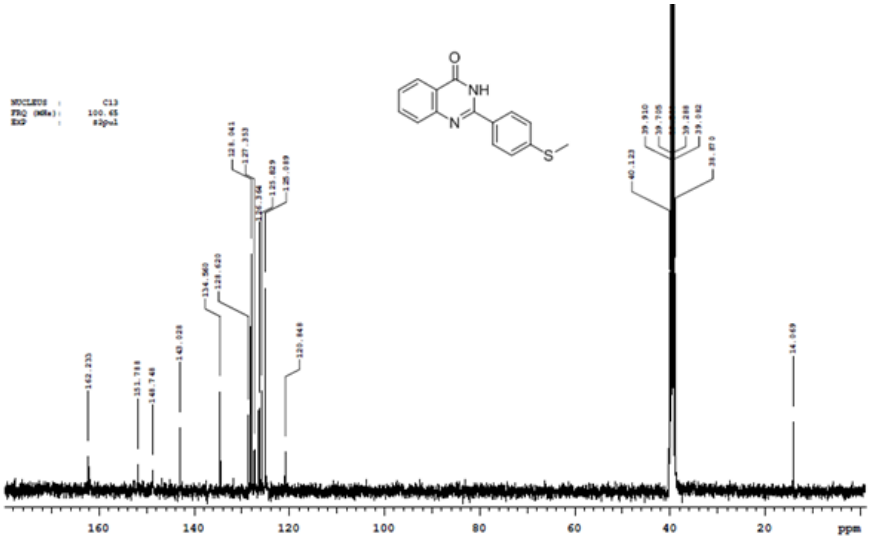

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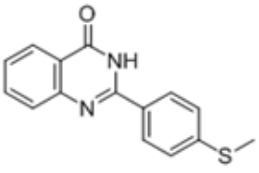

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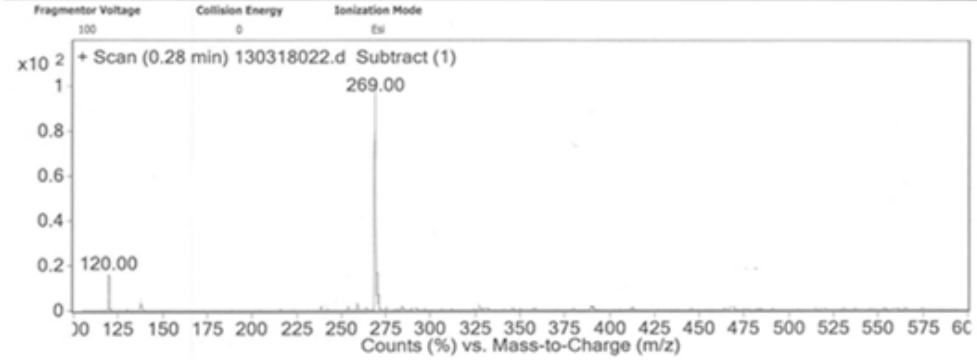

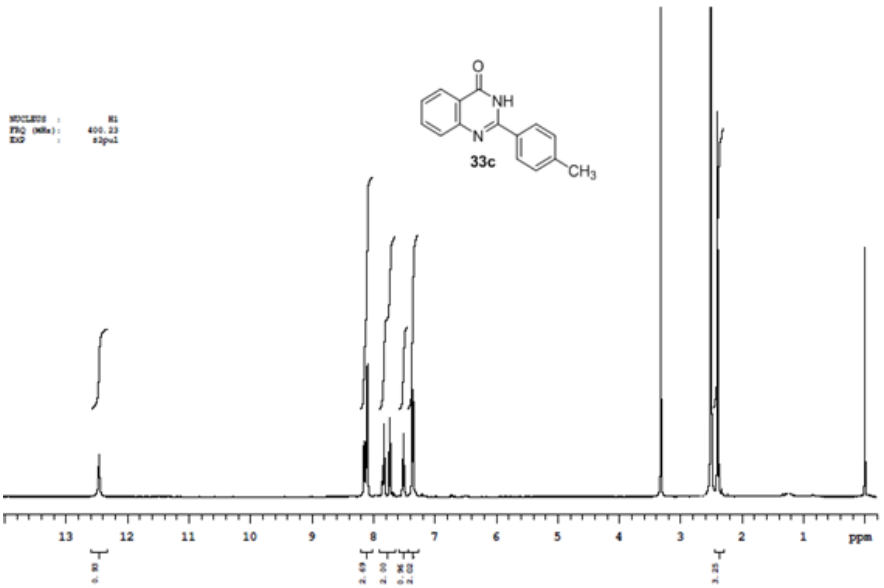

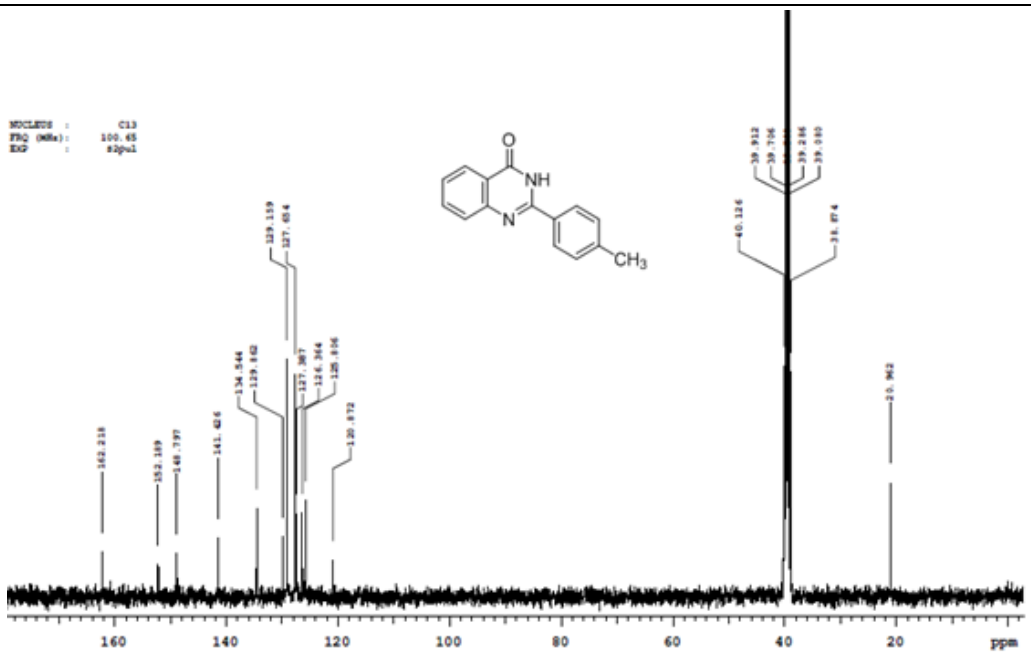
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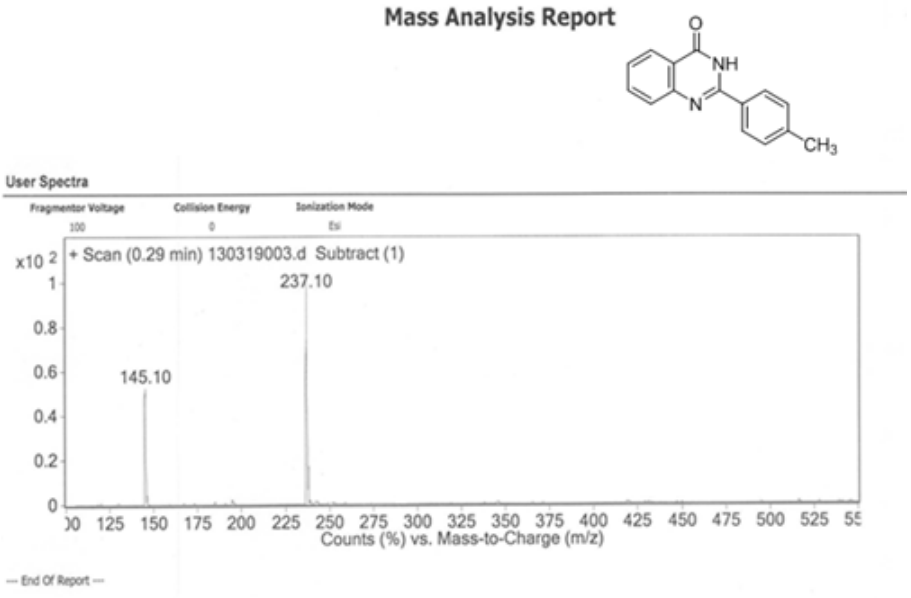
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User Spectra
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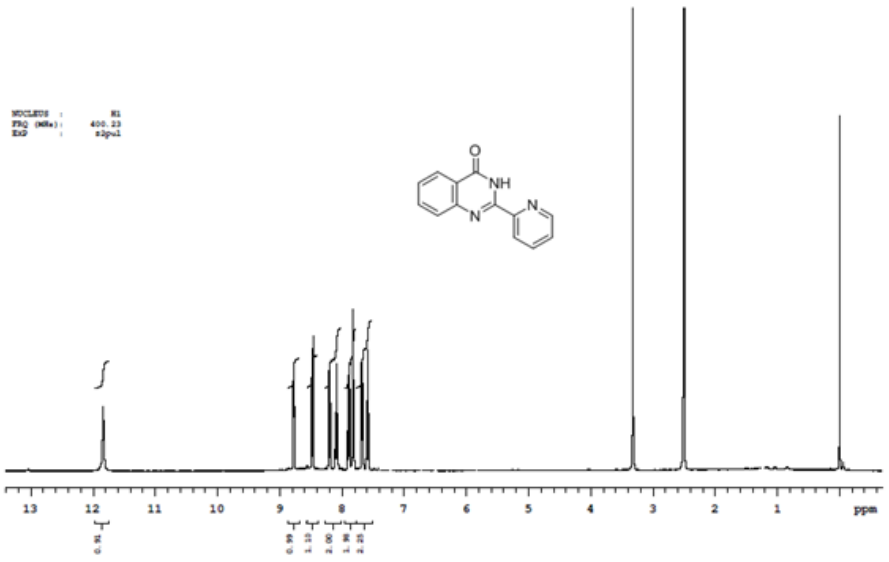
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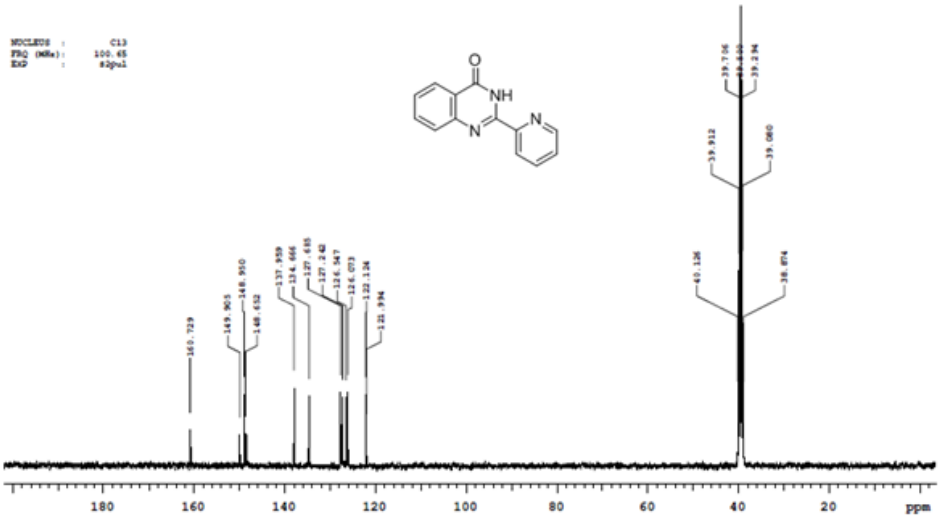
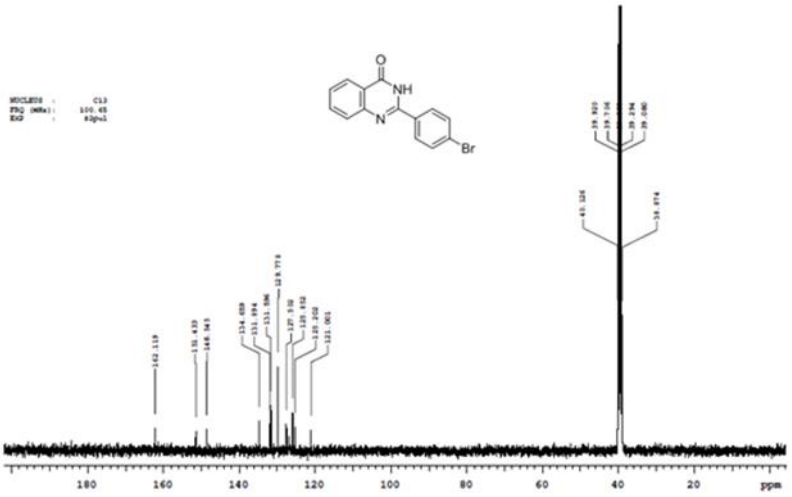
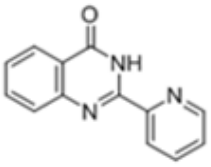




Mass Analysis Report



User Spectra

Fragmentor Voltage	Collision Energy	Ionization Mode
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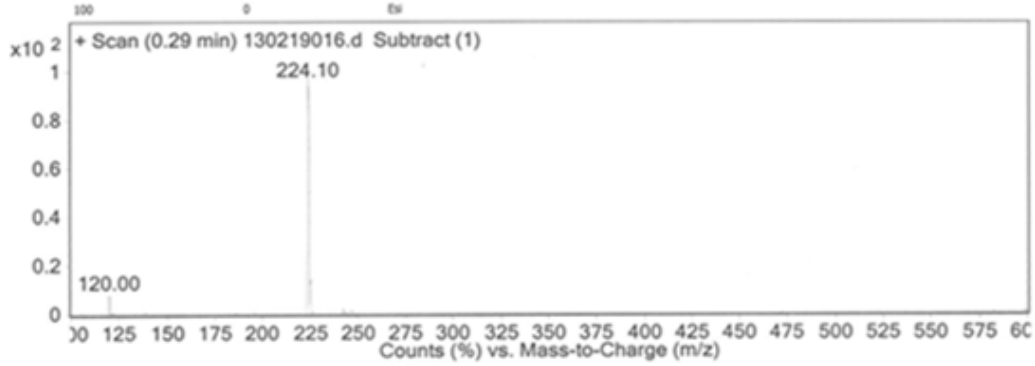

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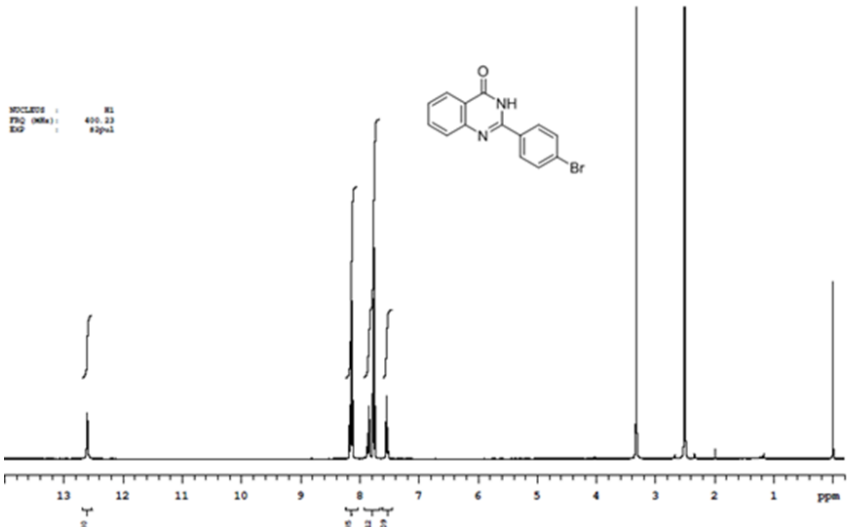

Mass Analysis Report

User Spectra

Spectrum Source	Fragmentor Voltage	Collision Energy	Ionization Mode
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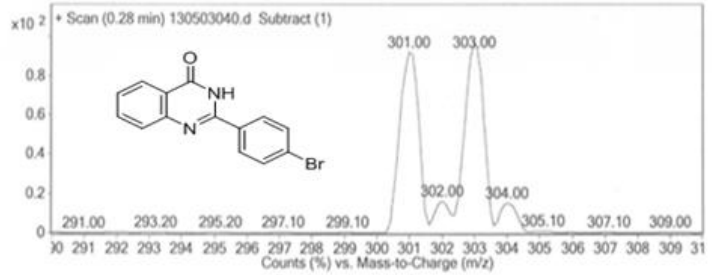

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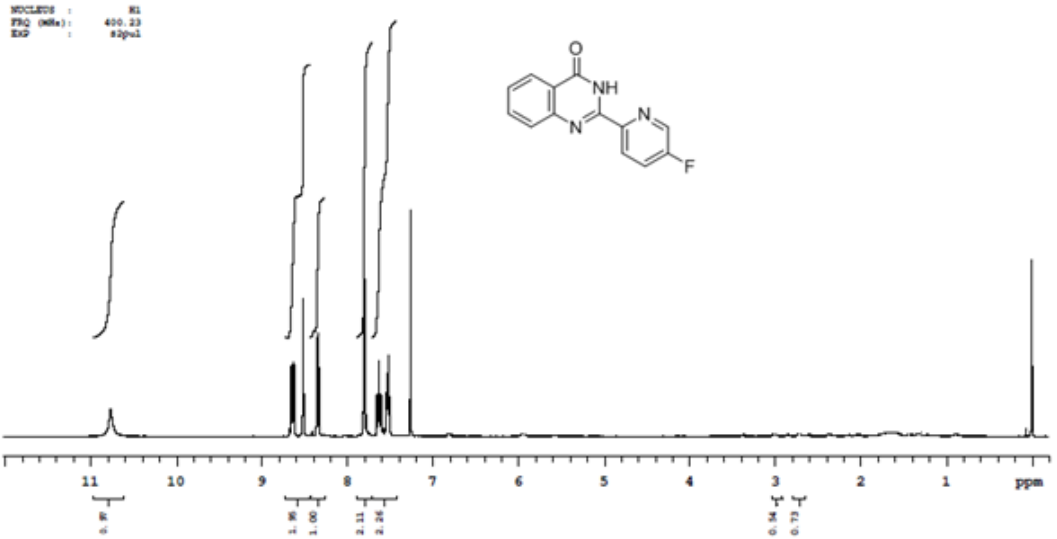

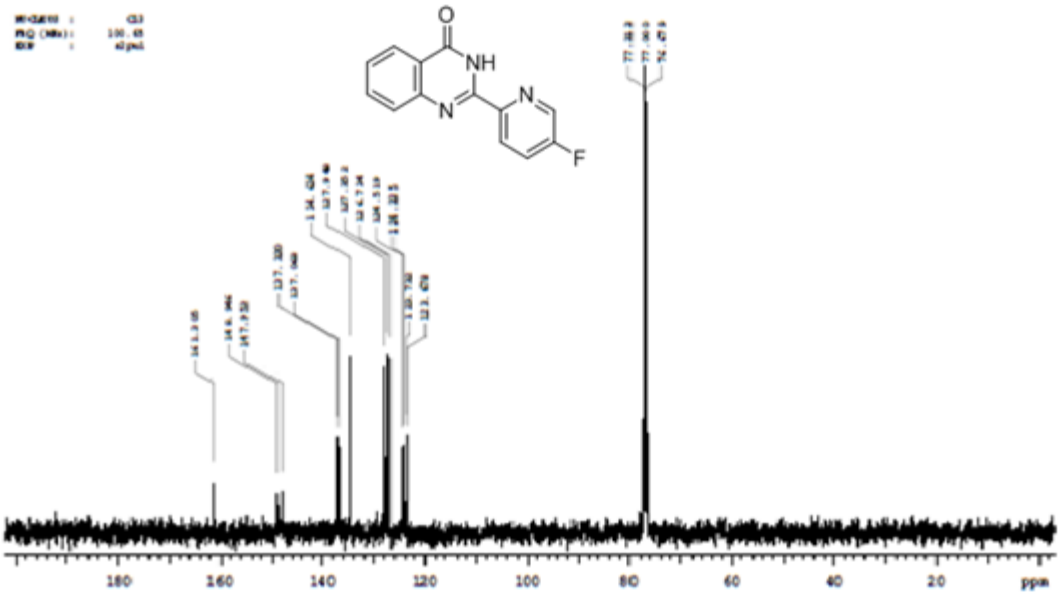
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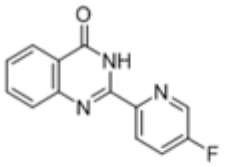
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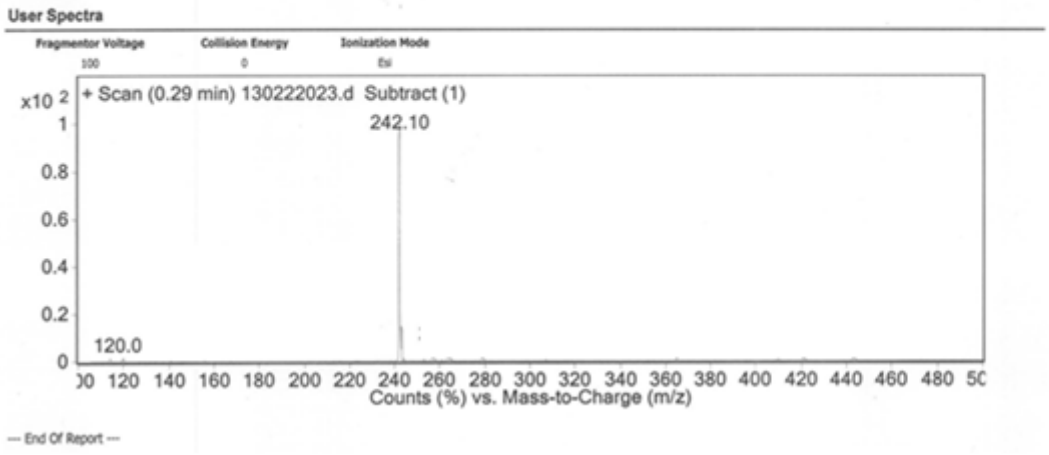
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Counts (%) vs. Mass-to-Charge (m/z)
— End Of Report —