Electronic Supplementary Material (ESI) for New Journal of Chemistry.

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Supplementary Information

Synthesis of 3-Oxadiazole-substituted imidazo[1,2-*a*]pyridines by Nickel Immobolized on Multifunctional Amphiphilic Porous Polysulfonamide-melamine

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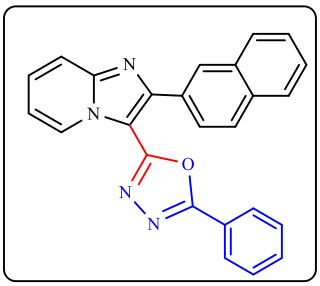
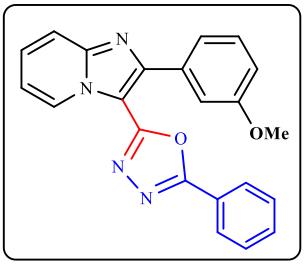
Contents

2-(2-(naphthalen-2-yl) imidazo[1,2-a] pyridin-3-yl)-5-phenyl-1,3,4-oxadiazole.



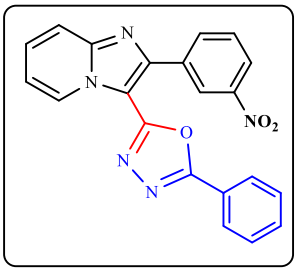
The general procedure was followed. Purification by column chromatography (silica gel, *n*-hexan/EtOAc, 10:5) gave the desired product as a white solid, M.p. 282–284 °C; ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.56 (dd, J = 11.7, 7.4 Hz, 3H), 8.19 – 8.15 (m, 1H), 8.12 (dd, J = 8.6, 1.8 Hz, 1H), 8.00 (dd, J = 11.4, 8.3 Hz, 2H), 7.93 (d, J = 9.5 Hz, 1H), 7.71–7.59 (m, 3H), 7.59–7.44 (m, 3H), 7.28 (t, J = 6.7 Hz, 1H), 6.92 (t, J = 6.7 Hz, 1H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 164.5, 145.4, 144.7, 133.7, 133.2, 133.1, 132.5, 131.9, 131.8, 129.8, 128.6, 128.5, 128.1, 127.4, 127.1, 126.8, 126.4, 125.5, 124.5, 124.5, 124.3, 123.8, 118.3, 117.1, 112.7, 110.1, 109.9; MS *m/z:* 388.4.

2-(2-(3-methoxyphenyl) imidazo[1,2-a]pyridin-3-yl)-5-phenyl-1,3,4-oxadiazole.



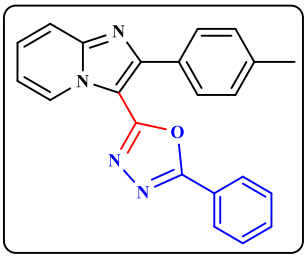
The general procedure was followed. Purification by column chromatography (silica gel, *n*-hexan/EtOAc, 10:5) gave the desired product as a white solid, M.p. 269–270 °C; ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.84-8.87 (m, 1H), 8.11 (d, 2H), 7.94 (s, 2H), 7.58-7.65 (m, 4H), 7.46-7.52 (m, 3H), 7.10 (dq, J = 6.9, 2.2 Hz, 1H), 3.85 (s, 3H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 164.4, 160.3, 140.6, 135.8, 133.9, 132.4, 131.2, 129.8, 129.5, 128.2, 127.1, 123.77, 118.8, 117.7, 116.4, 112.6, 112.0, 111.9, 55.9. MS *m/z*: 368.4.

2-(2-(3-nitrophenyl) imidazo[1,2-a] pyridin-3-yl)-5-phenyl-1,3,4-oxadiazole.



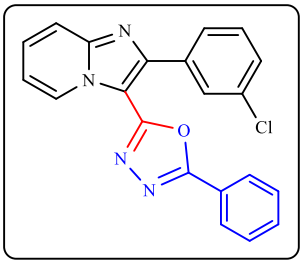
The general procedure was followed. Purification by column chromatography (silica gel, *n*-hexan/EtOAc, 10:5) gave the desired product as a white solid. M.p. 279–281 °C; ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.78 (s, 1H), 8.64 (m, 1H), 8.56 (d, J = 6.9 Hz, 1H), 8.40 (d, J = 7.8 Hz, 1H), 8.16 (t, J = 11 Hz, 3H), 7.75 (t, J = 8.2 Hz, 1H), 7.63-7.66 (m, 3H), 7.32 (t, J = 8.2 Hz, 1H), 6.96 (t, J = 6.9 Hz, 1H). ^{13}C NMR (126 MHz, DMSO-d_6) δ 164.4, 148.8, 145.4, 142.3, 136.0, 132.5, 132.1, 130.8, 129.8, 127.6, 127.1, 126.2, 123.8, 122.6, 120.2, 117.2, 113.3, 111.0. MS *m/z*: 383.4. MS *m/z*: 383.1.

2-phenyl-5-(2-(p-tolyl) imidazo[1,2-a]pyridin-3-yl)-1,3,4-oxadiazole.



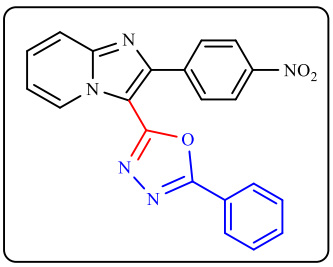
The general procedure was followed. Purification by column chromatography (silica gel, *n*-hexan/EtOAc, 10:5) gave the desired product as a white solid, M.p. 277–279 °C; ^{1}H NMR (250 MHz, DMSO-d_6) δ 8.51 (d, J = 6.8 Hz, 1H), 8.38-8.41 (m, 1H), 7.97 (dd, J = 10.5, 7.8 Hz, 3H), 7.56 (d, J = 9.1 Hz, 1H), 7.41 (d, J = 7.6 Hz, 4H), 7.31 (d, J = 7.1 Hz, 1H), 7.23 (t, J = 7.5 Hz, 1H), 6.87 (t, J = 7.5 Hz, 1H).2.38 (s, 3H). ^{13}C NMR (63 MHz, DMSO-d_6) δ 165.5, 142.5, 130.4, 129.1, 128.1, 127.3, 127.0, 125.9, 125.3, 121.0, 117.0, 112.7, 109.5, 21.5. MS *m/z*: 352.4.

2-(2-(3-chlorophenyl) imidazo[1,2-a]pyridin-3-yl)-5-phenyl-1,3,4-oxadiazole.



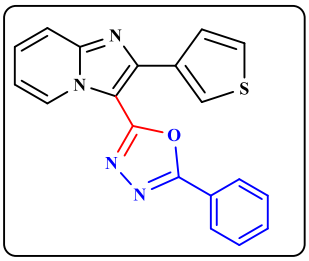
The general procedure was followed. Purification by column chromatography (silica gel, *n*-hexan/EtOAc, 10:5) gave the desired product as a light pink solid, M.p. 240–242 °C; ^{1}H NMR (250 MHz, DMSO-d_6) δ 8.48 (s, 2H), 8.10 (s, 2H), 8.06 – 7.77 (m, 2H), 7.58 (s, 3H), 7.23-7.43 (m, 3H), 6.88 (s, 1H). ^{13}C NMR (63 MHz, DMSO-d_6) δ 164.4, 145.3, 143.2, 136.5, 134.0, 132.4, 131.0, 129.8, 127.8, 127.4, 127.1, 125.7, 125.5, 124.5, 123.7, 117.1, 112.9, 110.4. MS *m/z*: 372.8.

2-(2-(4-nitrophenyl) imidazo[1,2-a]pyridin-3-yl)-5-phenyl-1,3,4-oxadiazole.



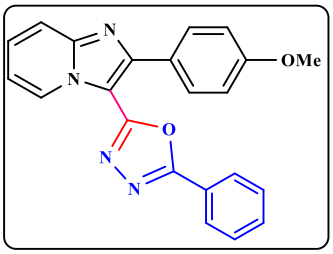
The general procedure was followed. Purification by column chromatography (silica gel, *n*-hexan/EtOAc, 10:5) gave the desired product as a white solid, M.p. 326–328 °C; ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.90 (s, 1H), 8.81 (d, J = 6.8 Hz, 1H), 8.43 (d, J = 8.8 Hz, 1H), 8.23 (d, J = 8.7 Hz, 1H), 8.15 (dd, J = 7.3, 2.2 Hz, 2H), 7.87 (d, J = 10 Hz, 1H), 7.76 (t, J = 5 Hz, 1H), 7.63-7.69 (m, 4H), 7.34 (t, J = 6.8 Hz, 1H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 164.5, 147.9, 132.5, 129.9, 129.0, 127.4, 127.1, 125.0, 123.8, 113.3. MS *m/z*: 383.4.

2-phenyl-5-(2-(thiophen-3-yl)imidazo[1,2-a]pyridin-3-yl)-1,3,4-oxadiazole.



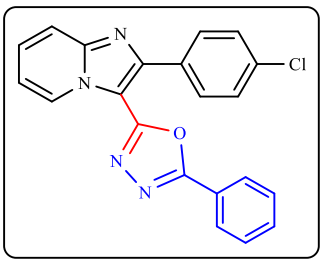
The general procedure was followed. Purification by column chromatography (silica gel, *n*-hexan/EtOAc, 10:5) gave the desired product as a dark orange solid. M.p. 330–332 °C; ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.49 (d, J = 6.8 Hz, 1H), 8.26 (s, 1H), 8.13 (d, J = 5 Hz, 2H), 7.69 – 7.58 (m, 2H), 7.59 – 7.44 (m, 3H), 7.23 (ddd, J = 8.8, 6.8, 1.4 Hz, 1H), 7.11 (dd, J = 5.1, 3.4 Hz, 1H), 6.88 (t, J = 6.8 Hz, 1H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 164.4, 144.9, 140.1, 138.2, 132.5, 129.8, 128.4, 127.2, 127.1, 125.9, 125.5, 124.0, 123.8, 116.7, 112.8, 108.6, MS *m/z*: 344.4.

2-(2-(4-methoxyphenyl)imidazo[1,2-a]pyridin-3-yl)-5-phenyl-1,3,4-oxadiazole.



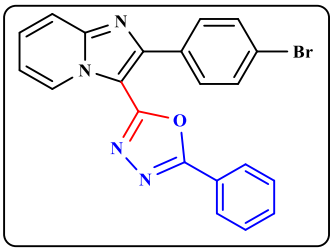
The general procedure was followed. Purification by column chromatography (silica gel, *n*-hexan/EtOAc, 10:5) gave the desired product as a white solid, M.p. 298–300 °C; ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.85 (d, J = 6.6 Hz, 1H), 8.68 (d, J = 4.0 Hz, 1H), 8.13 (d, J = 6.9 Hz, 3H), 7.89 (dd, J = 13.4, 6.2 Hz, 1H), 7.64 (d, J = 10 Hz, 6H), 7.16 (d, J = 8.2 Hz, 1H), 3.84 (s, 3H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 164.4, 161.2, 140.5, 136.4, 133.1, 132.5, 129.8, 129.2, 128.1, 127.1, 123.8, 117.4, 115.3, 112.5, 110.4, 55.9. MS *m/z*: 368.2.

2-(2-(4-chlorophenyl)imidazo[1,2-a]pyridin-3-yl)-5-phenyl-1,3,4-oxadiazole.



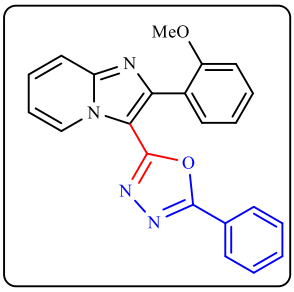
The general procedure was followed. Purification by column chromatography (silica gel, *n*-hexan/EtOAc, 10:5) gave the desired product as a white solid, M.p. 302–305 °C; ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.57 – 8.46 (d, J=5 Hz, 1H), 8.44 (s, 1H), 8.15 (dd, J = 5, 10 Hz, 2H), 7.98 (d, 2H), 7.72 – 7.63 (m, 3H), 7.59 (d, J = 9.1 Hz, 1H), 7.50 (d, J = 10 Hz, 2H), 7.27 (ddd, J = 8.6, 6.8, 1.5 Hz, 1H), 6.92 (t, J = 6.7 Hz, 1H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 164.5, 133.2, 132.5, 132.5, 129.8, 129.2, 127.7, 127.4, 127.1, 125.7, 123.8, 117.1, 112.9, 109.9. MS *m/z*: 372.8.

2-(2-(4-bromophenyl)imidazo[1,2-a]pyridin-3-yl)-5-phenyl-1,3,4-oxadiazole.



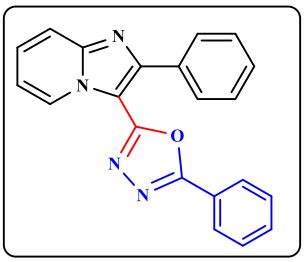
The general procedure was followed. Purification by column chromatography (silica gel, *n*-hexan/EtOAc, 10:5) gave the desired product as a white solid, M.p. 290–292 °C; ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.93 – 8.81 (m, 2H), 8.13 (d, *J*= 10 Hz, 2H), 7.95 (d, *J* = 4.0 Hz, 1H), 7.92 – 7.80 (dd, *J* = 10, 12 Hz, 3H), 7.70– 7.60 (m, 4H), 7.54 – 7.46 (m, 1H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 164.4, 140.9, 135.3, 133.8, 132.9, 132.5, 129.8, 129.5, 128.5, 127.1, 126.5, 124.1, 123.8, 117.6, 112.8, 112.1. MS *m/z*: 417.2.

2-(2-(2-methoxyphenyl)imidazo[1,2-a]pyridin-3-yl)-5-phenyl-1,3,4-oxadiazole.

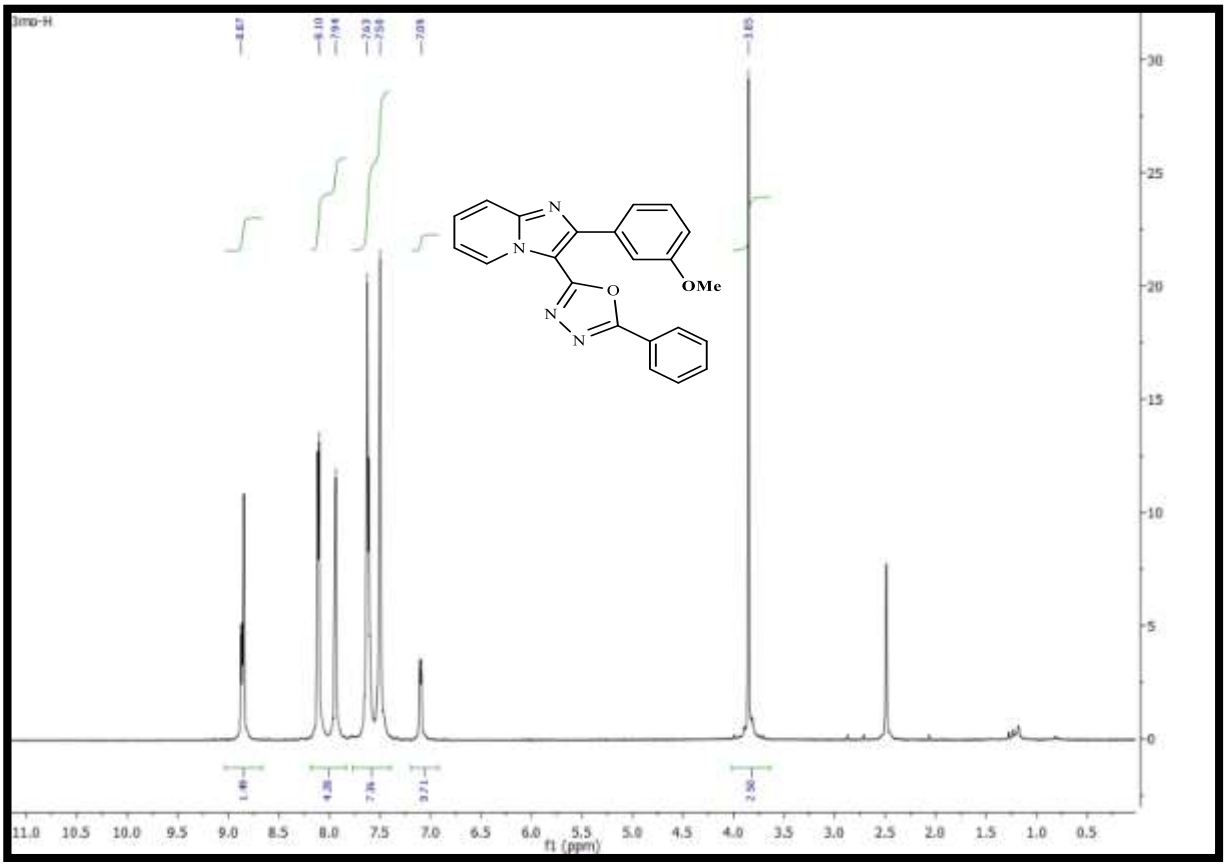


The general procedure was followed. Purification by column chromatography (silica gel, *n*-hexan/EtOAc, 10:5) gave the desired product as a white solid, M.p. 284–285 °C; ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.89 (d, *J* = 6.7 Hz, 1H), 8.44 (s, 1H), 8.22 (d, *J* = 10 Hz, 1H), 8.13 (d, *J* = 5.0 Hz, 3H), 8.03 (t, *J* = 10 Hz, 1H), 7.68 – 7.57 (m, 4H), 7.55 (s,1H), 7.00 (s, 1H), 3.92 (s, 3H). ^{13}C NMR (125 MHz, DMSO-d_6) δ 164.4, 148.3, 146.3, 140.0, 138.3, 133.5, 132.5, 129.9, 129.4, 127.1, 123.8, 121.7, 117.8, 117.2, 116.6, 116.2, 113.0, 112.2, 111.9, 32.6. MS *m/z*: 369.0.

2-phenyl-5-(2-phenylimidazo[1,2-a]pyridin-3-yl)-1,3,4-oxadiazole.



The general procedure was followed. Purification by column chromatography (silica gel, *n*-hexan/EtOAc, 5:1) gave the desired product as a white solid, M.p. 279–281 °C; ^{1}H NMR (250 MHz, DMSO-d_6) δ 8.51 (d, *J* = 7.5 Hz, 1H), 8.37 (d, *J* = 4.1 Hz, 1H), 8.19 – 8.04 (m, 2H), 7.95 (d, *J* = 7.5 Hz, 2H), 7.54-7.63 (m, 3H), 7.49 – 7.34 (m, 2H), 7.25 (dt, *J* = 21.8, 7.2 Hz, 2H), 6.99 – 6.74 (m, 1H). ^{13}C NMR (63 MHz, DMSO-d_6) δ 164.4, 145.2, 144.7, 134.3, 132.5, 129.8, 129.1, 128.1, 127.3, 127.1, 126.0, 125.3, 123.7, 117.0, 112.7, 109.5. MS *m/z*: 338.3.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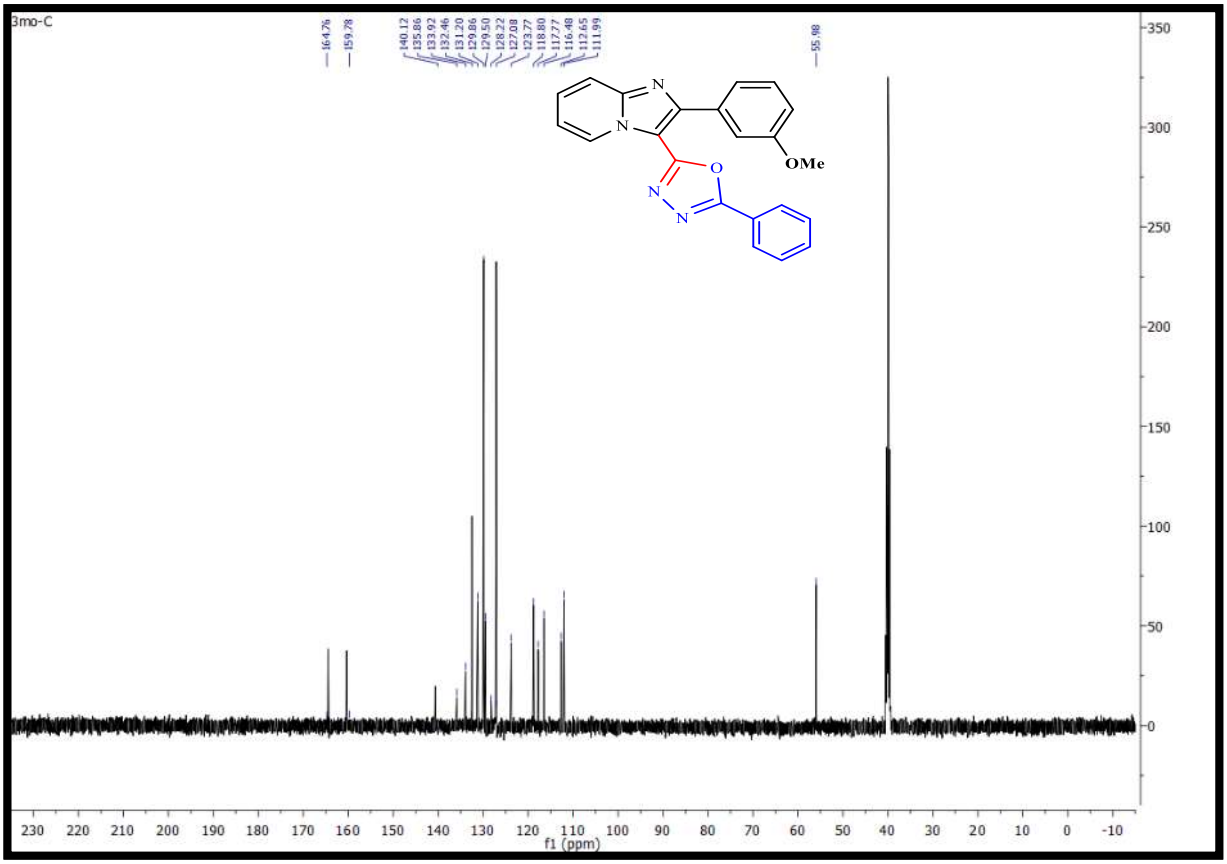
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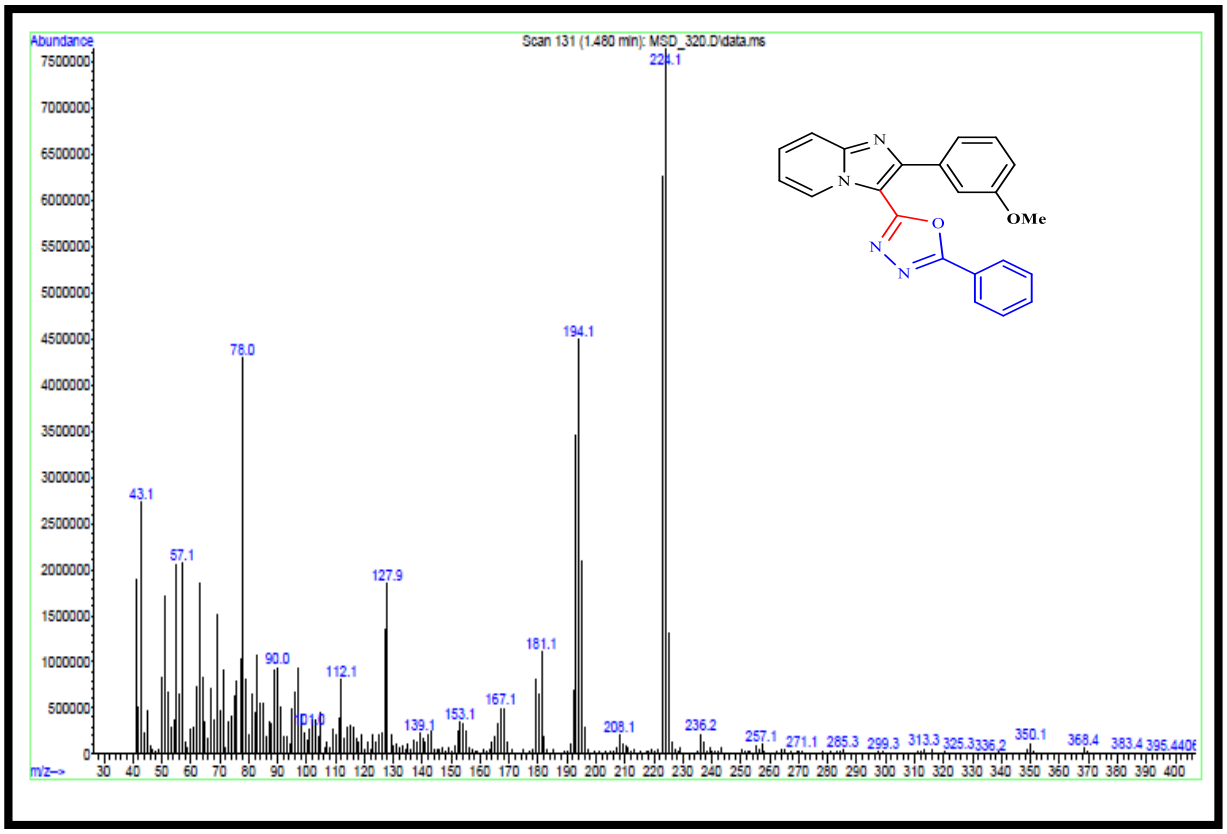
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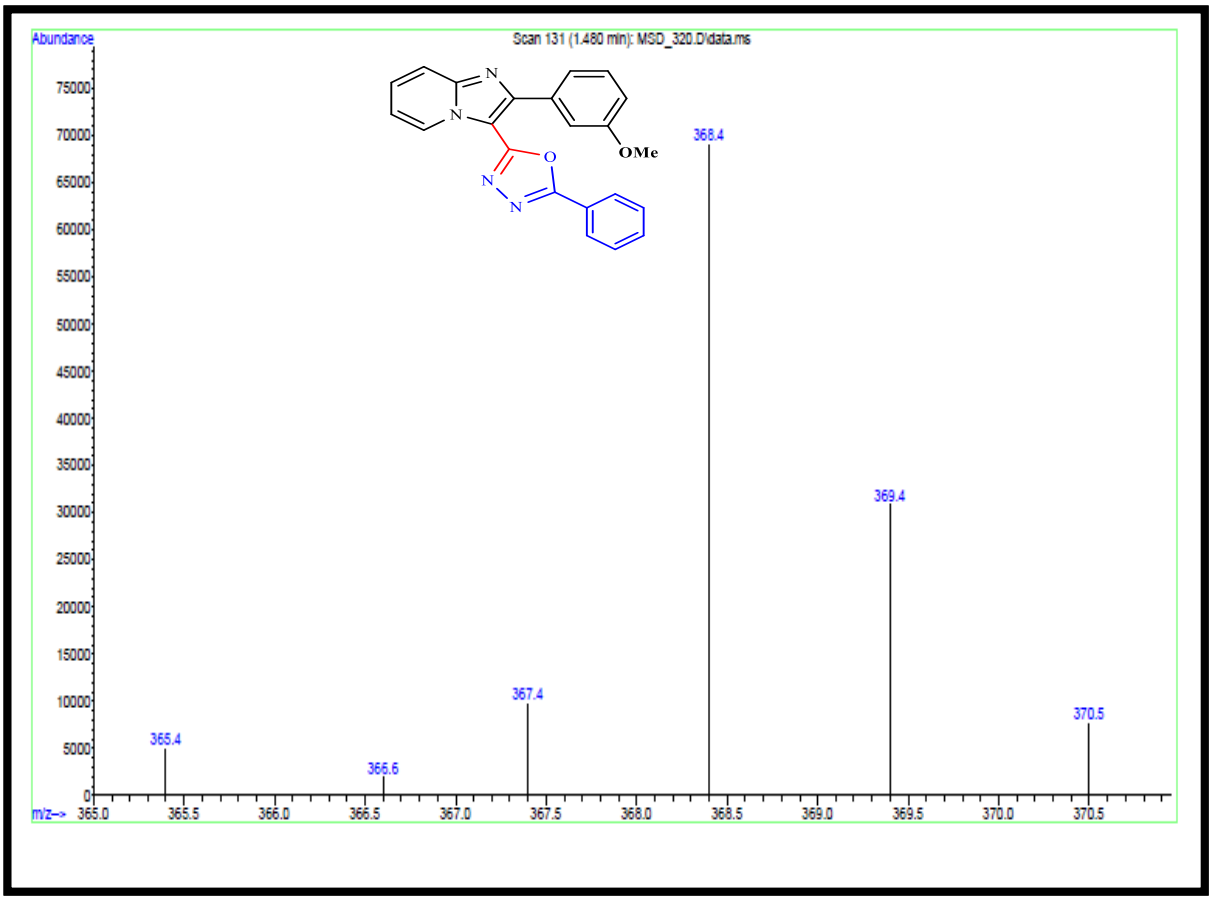
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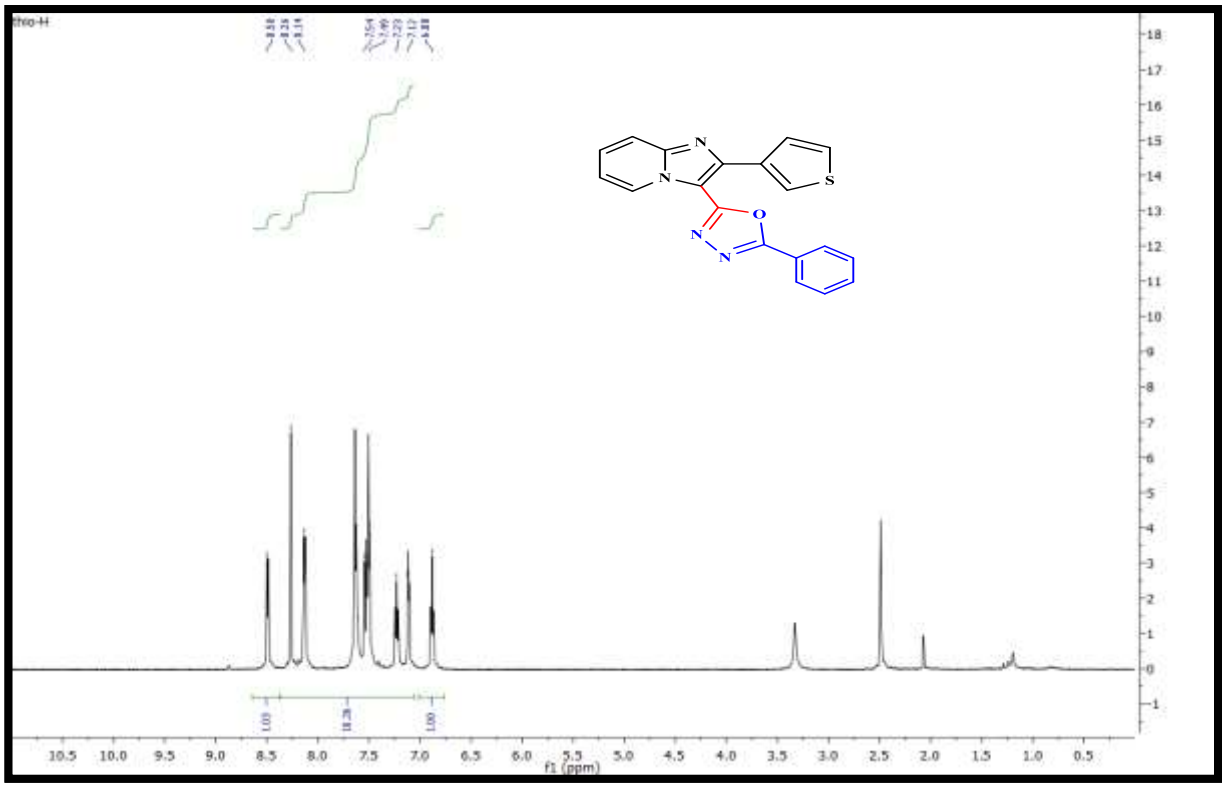
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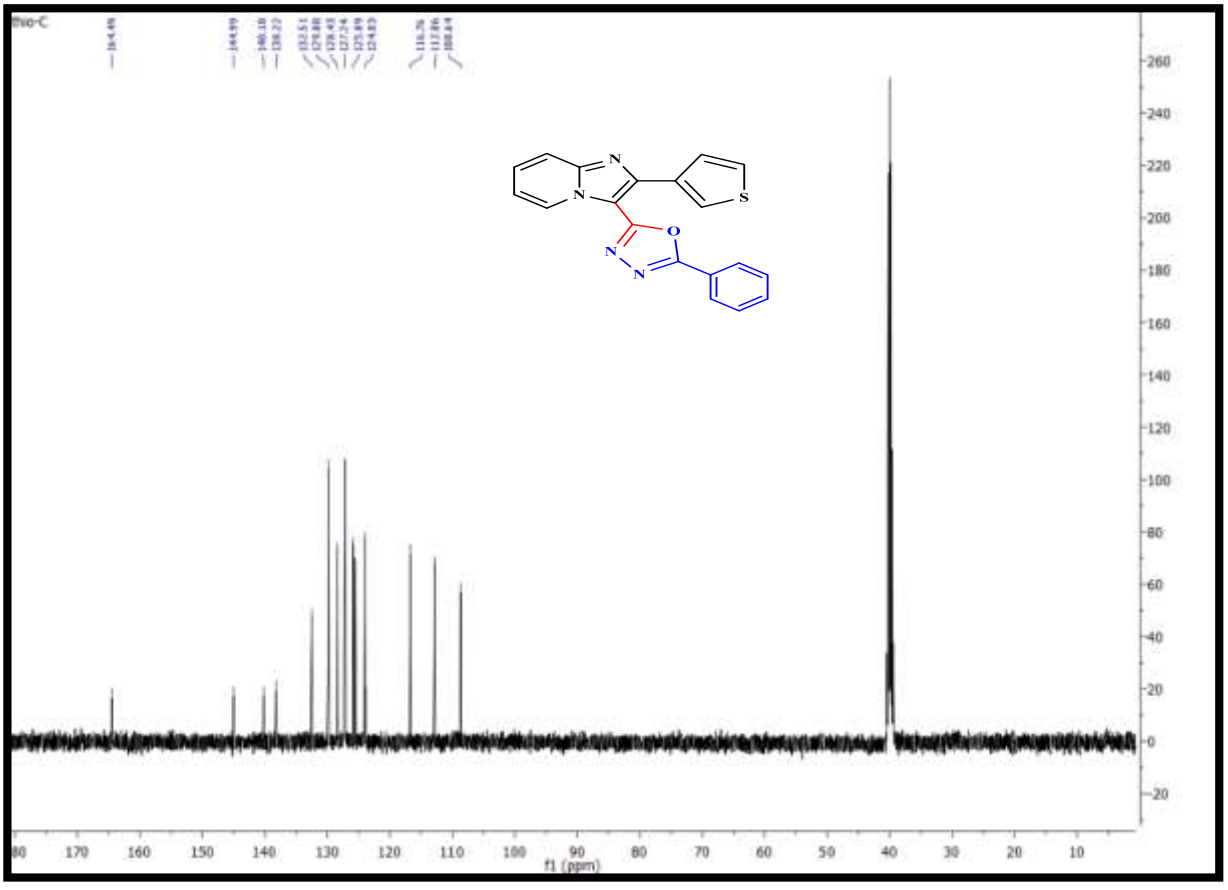
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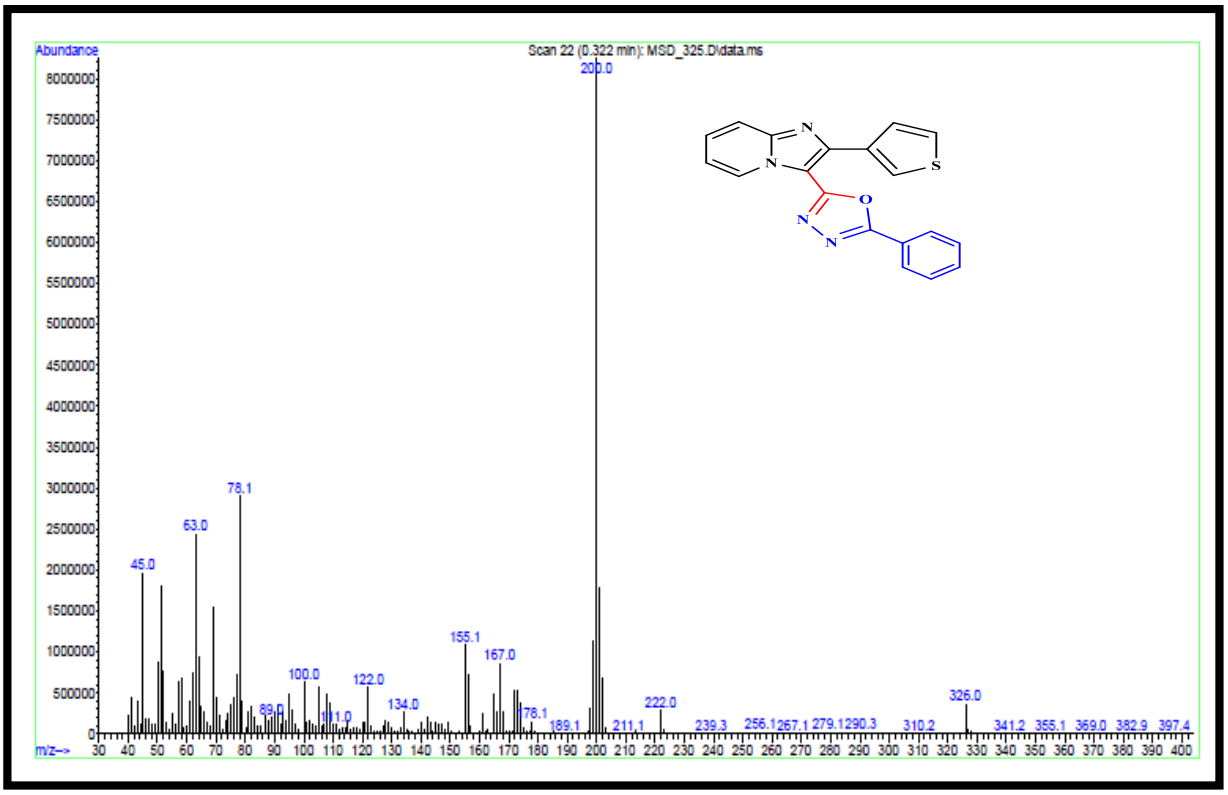
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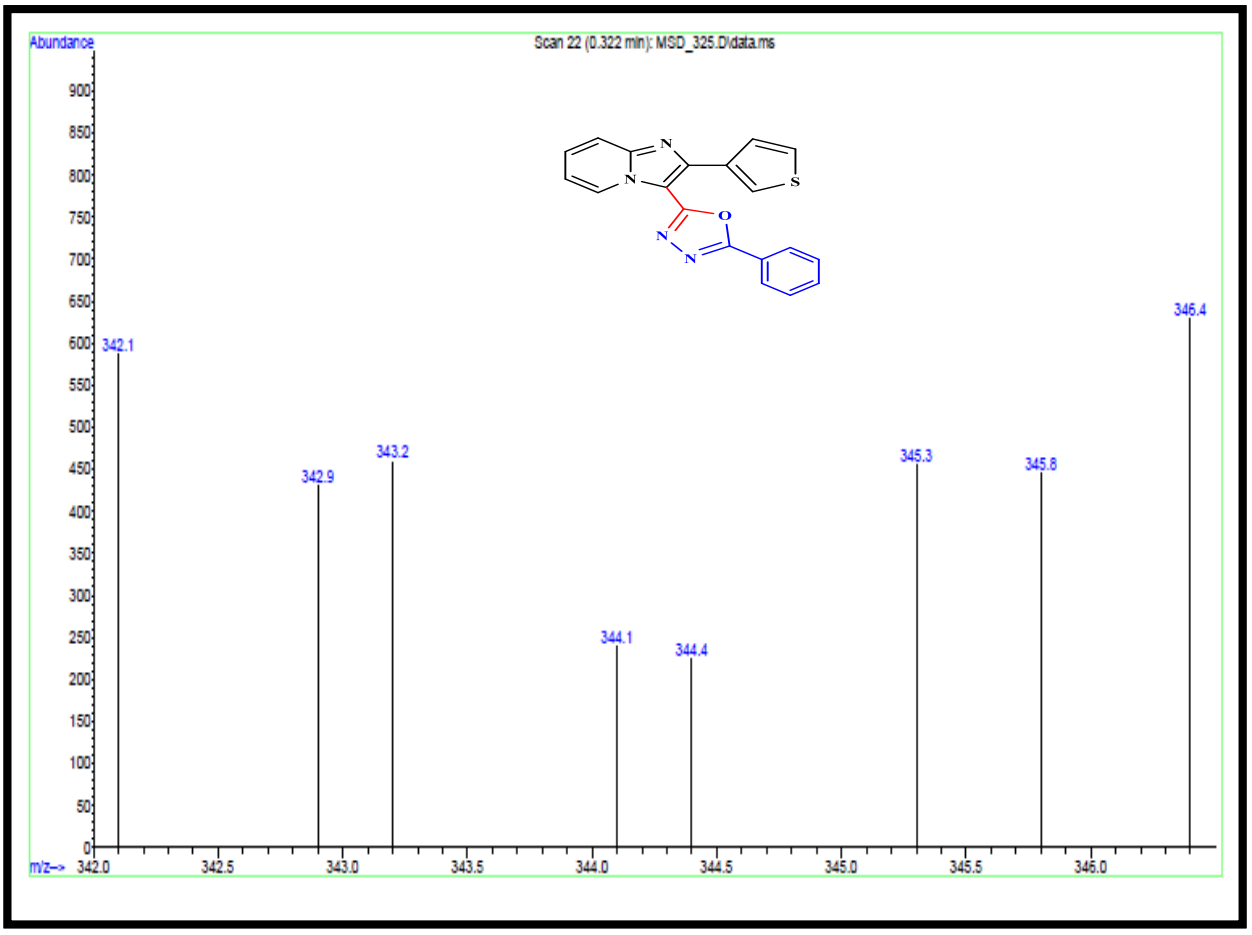
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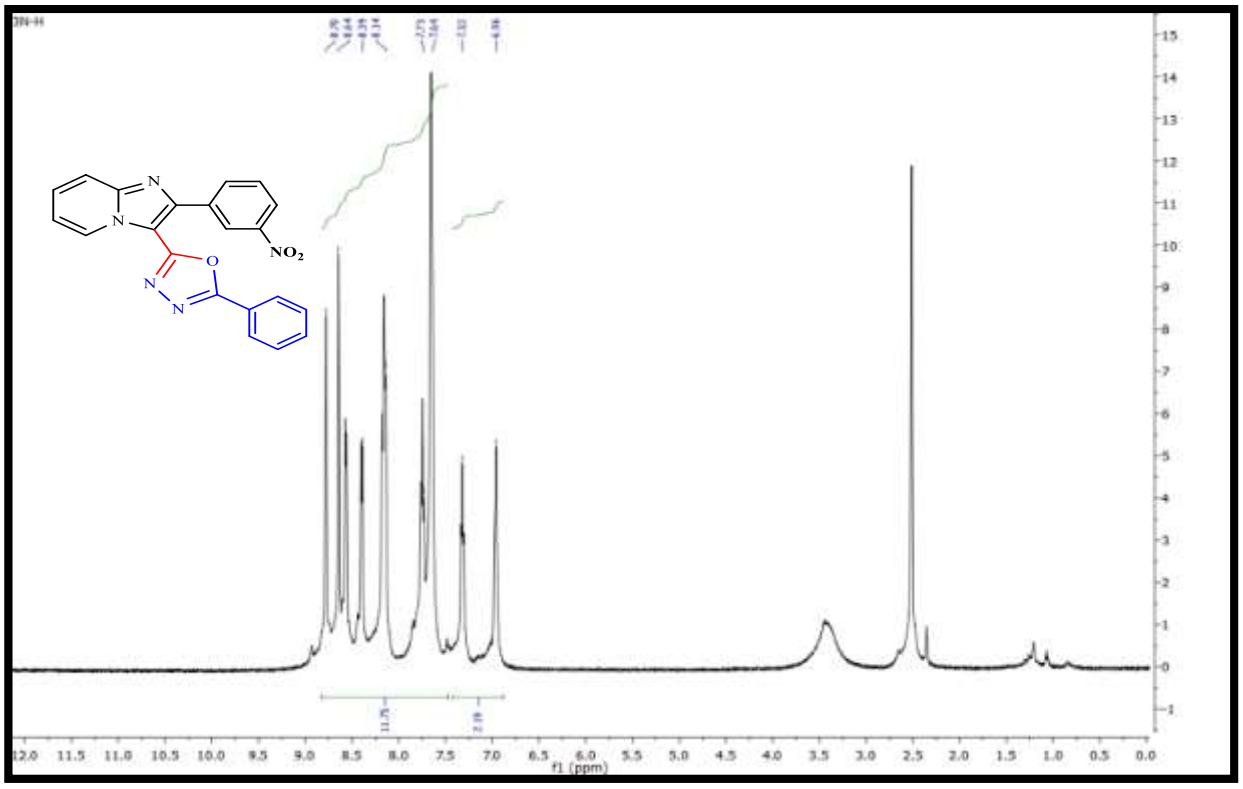
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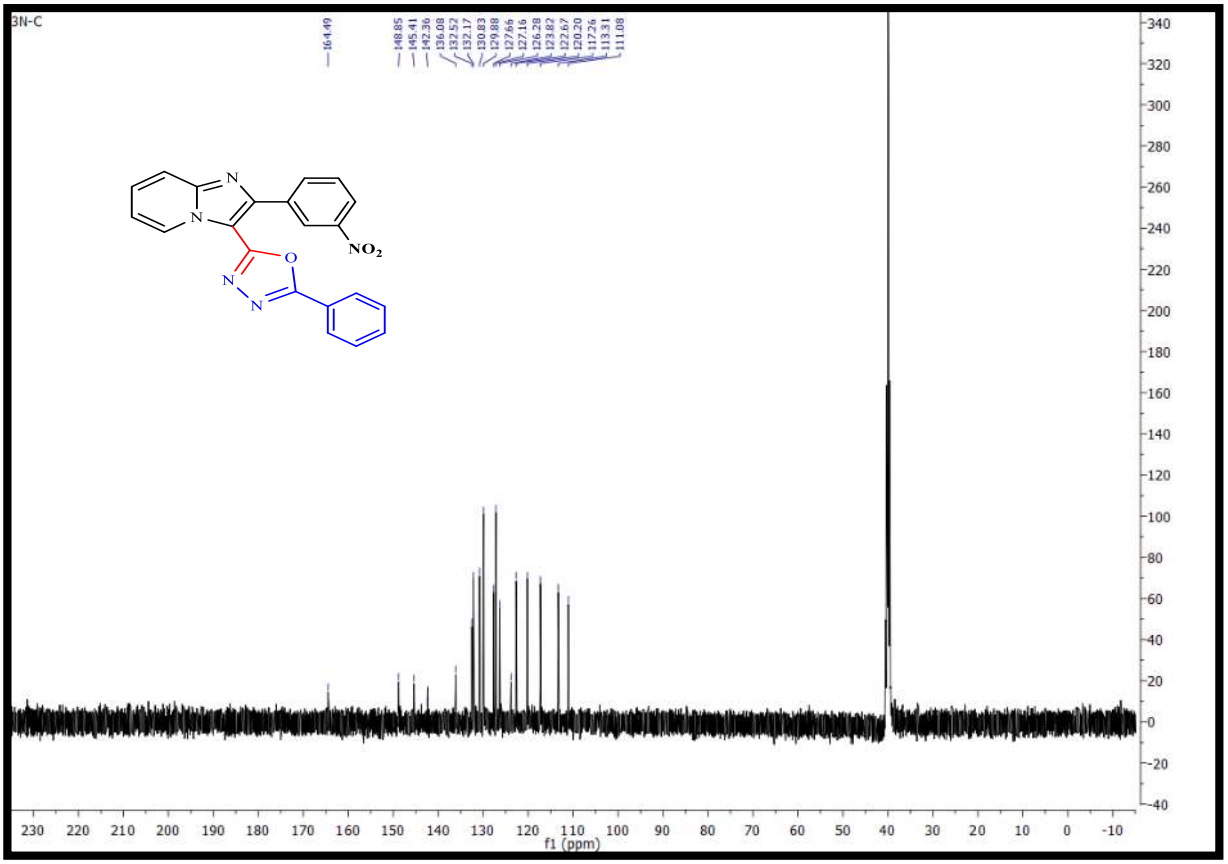
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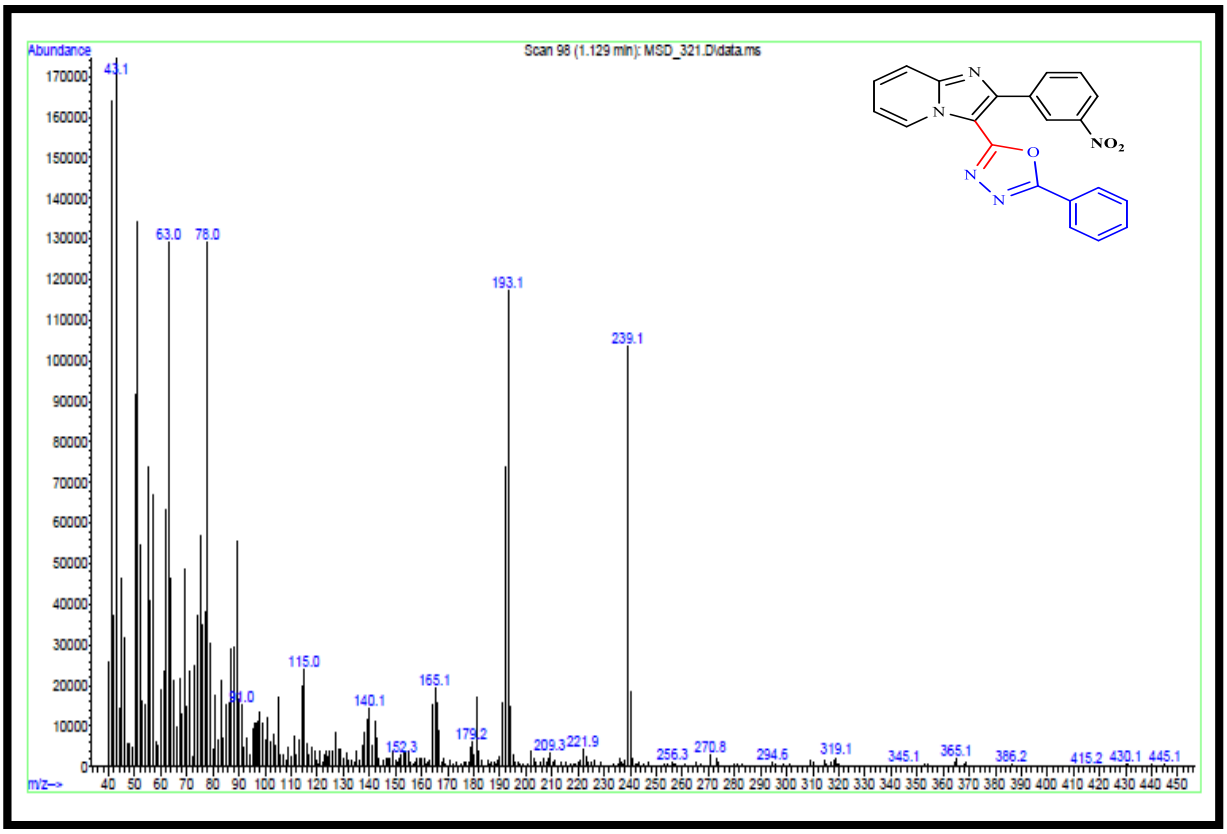
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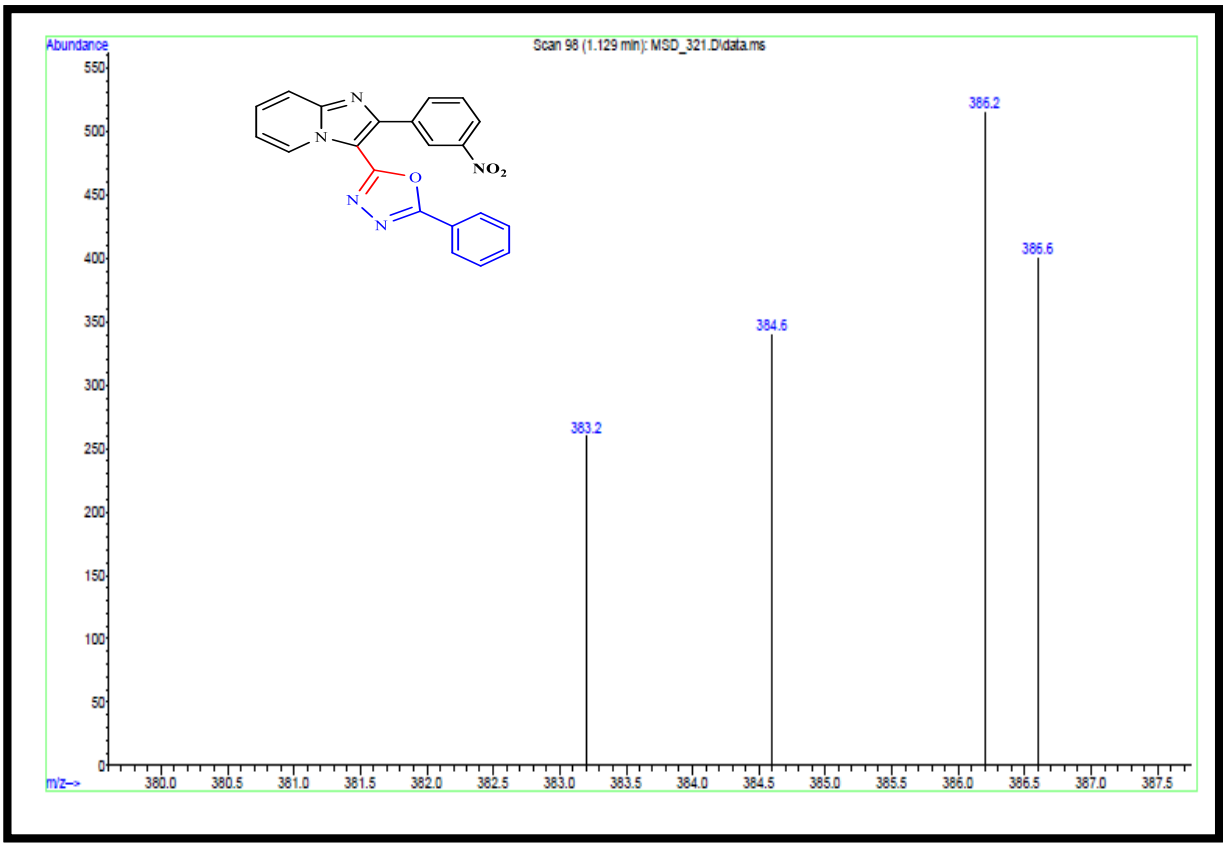
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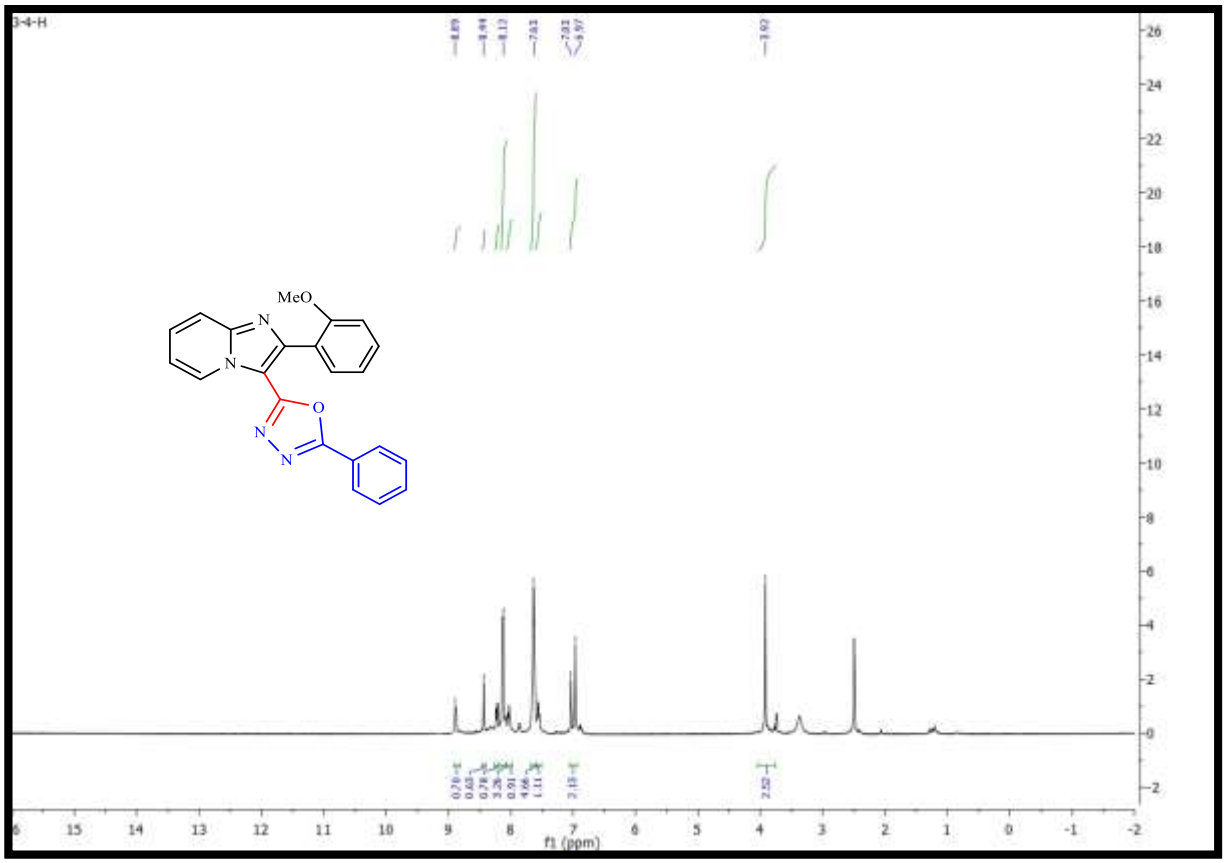
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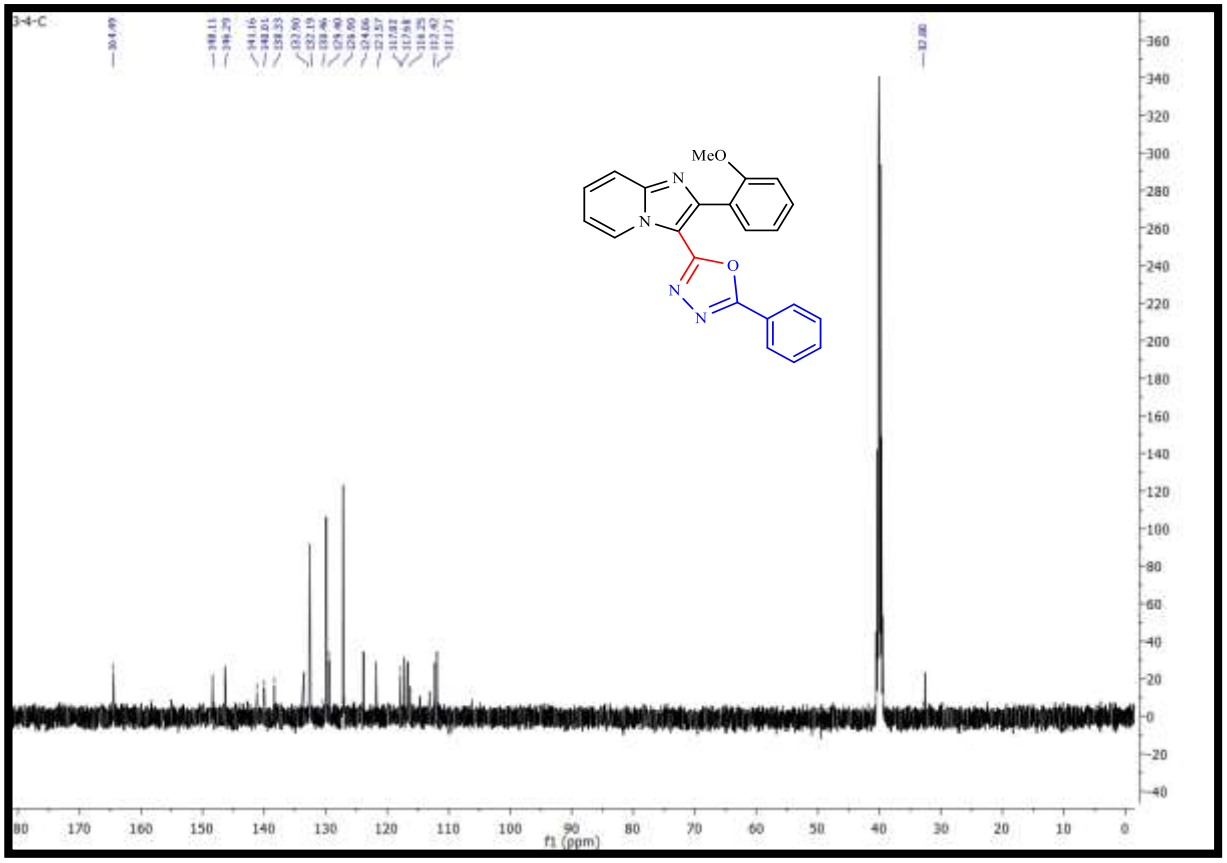
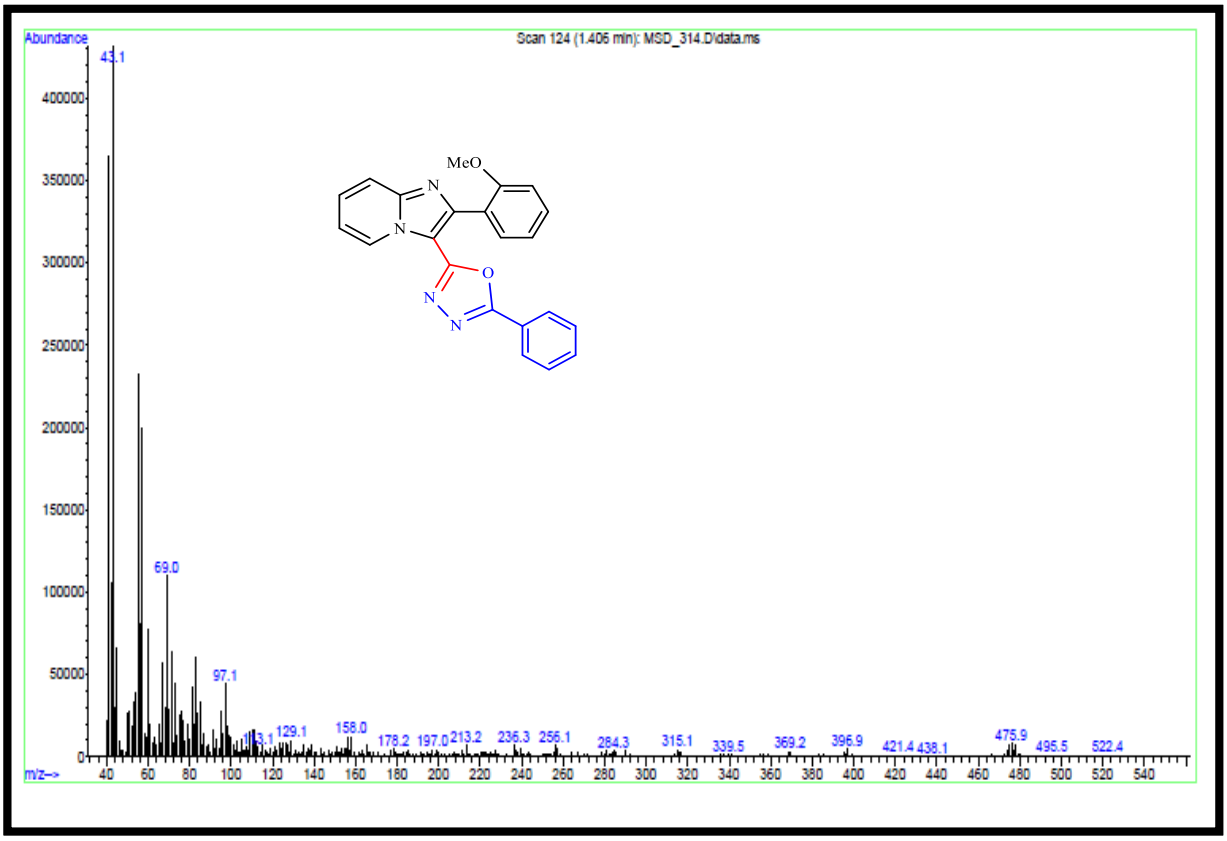
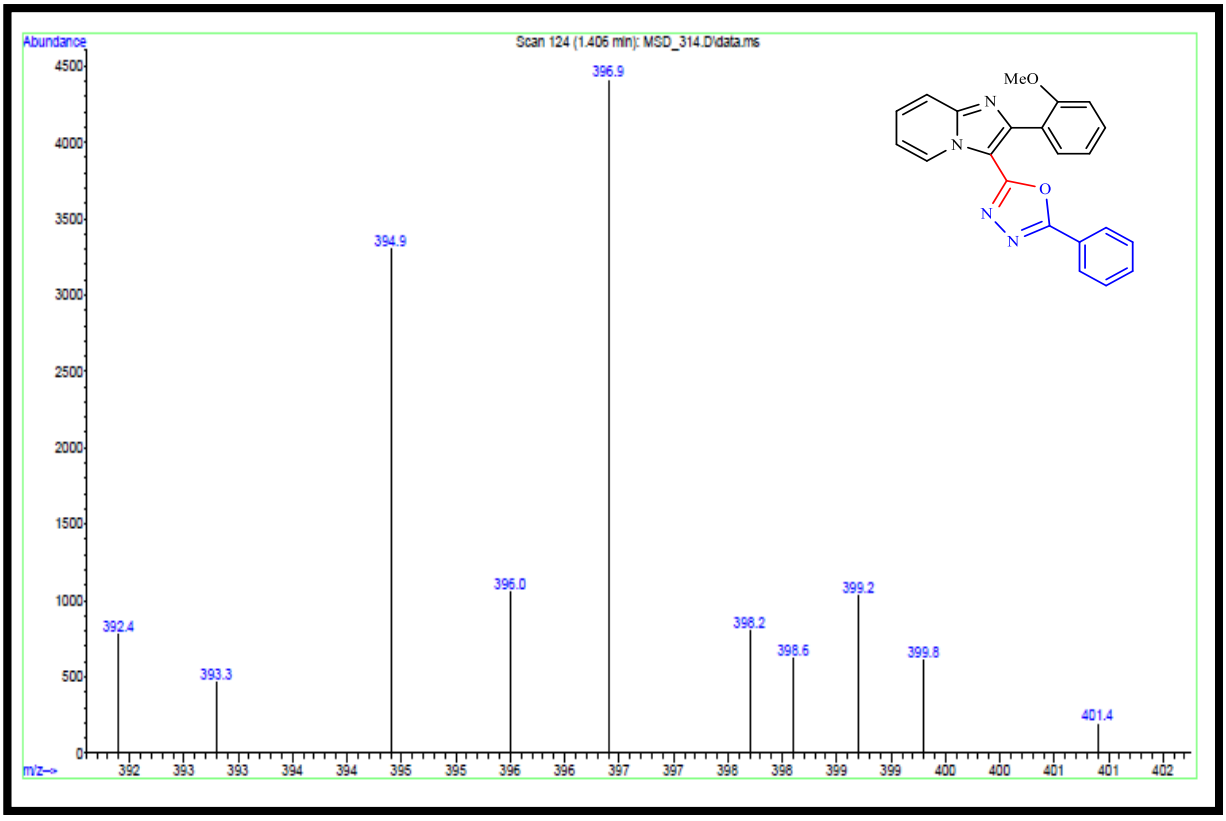
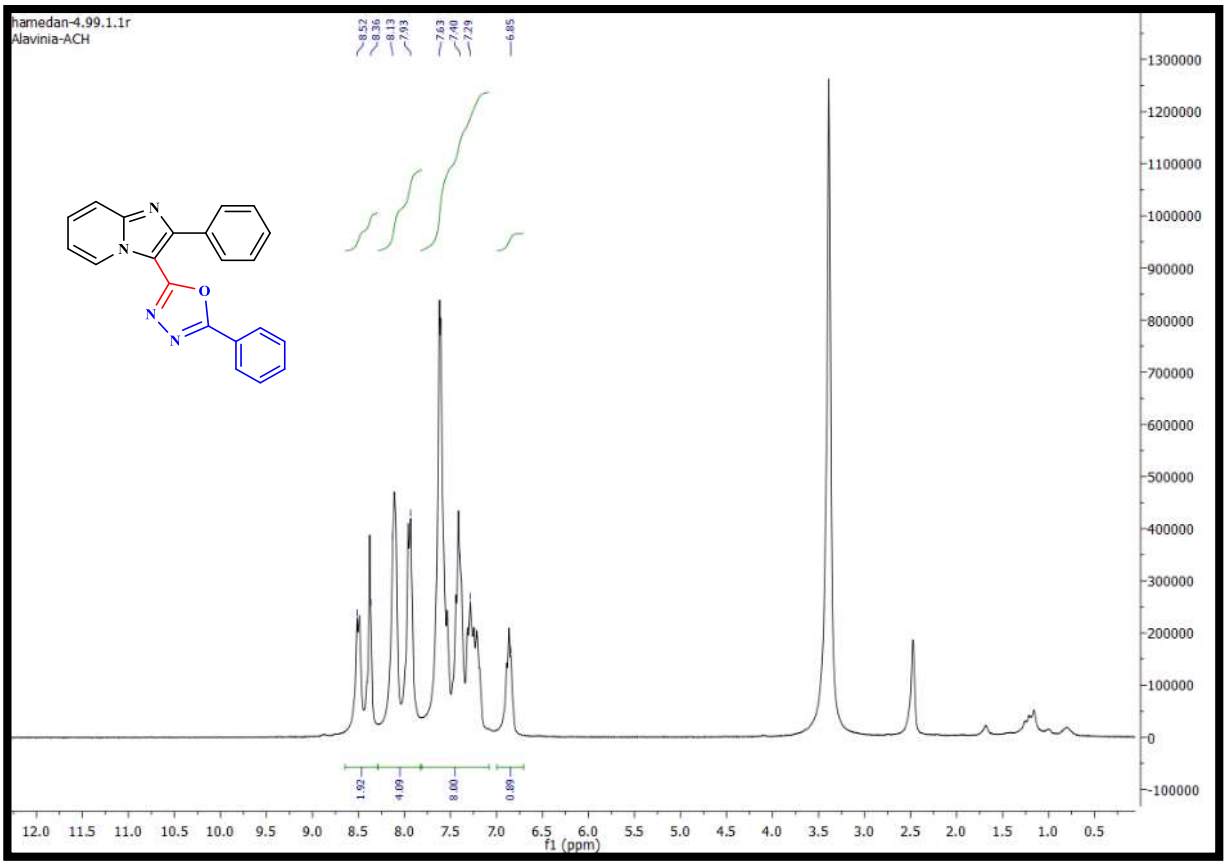
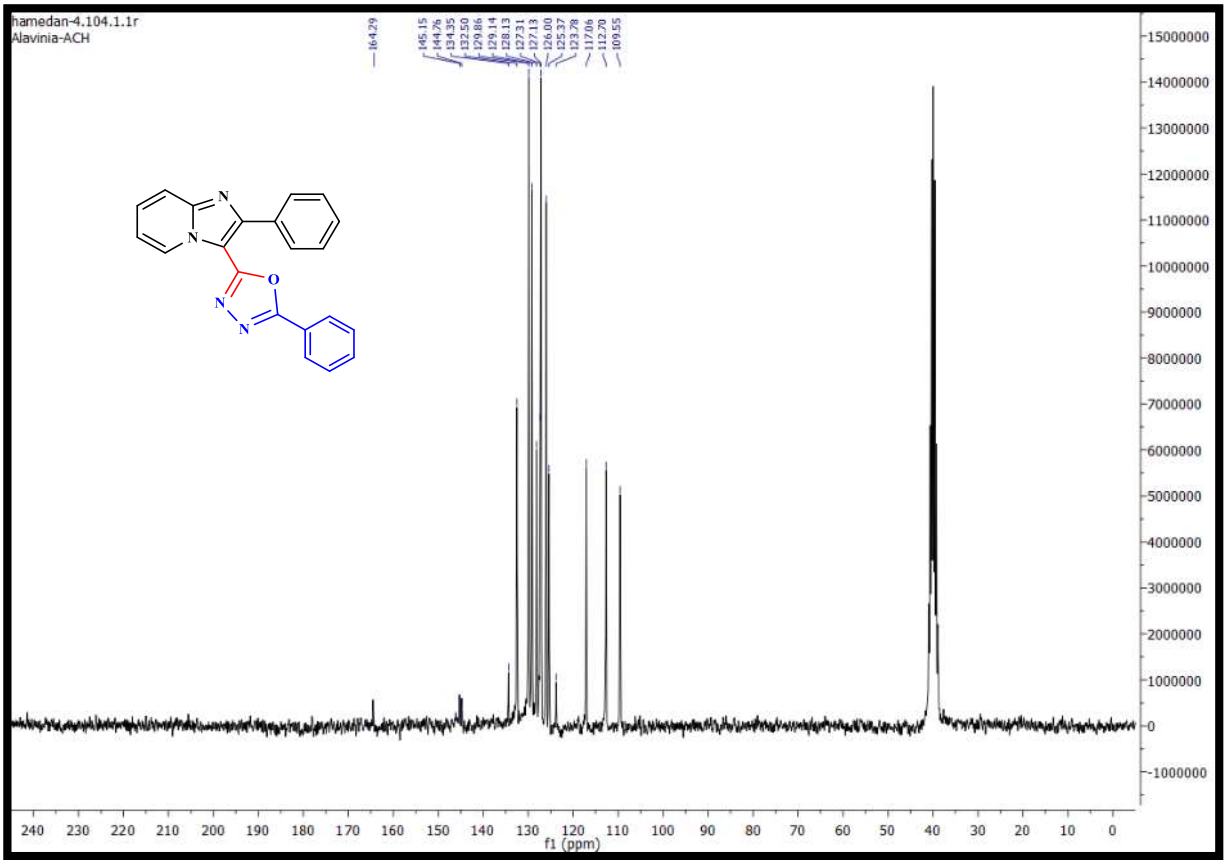
N
N
f1 (ppm)

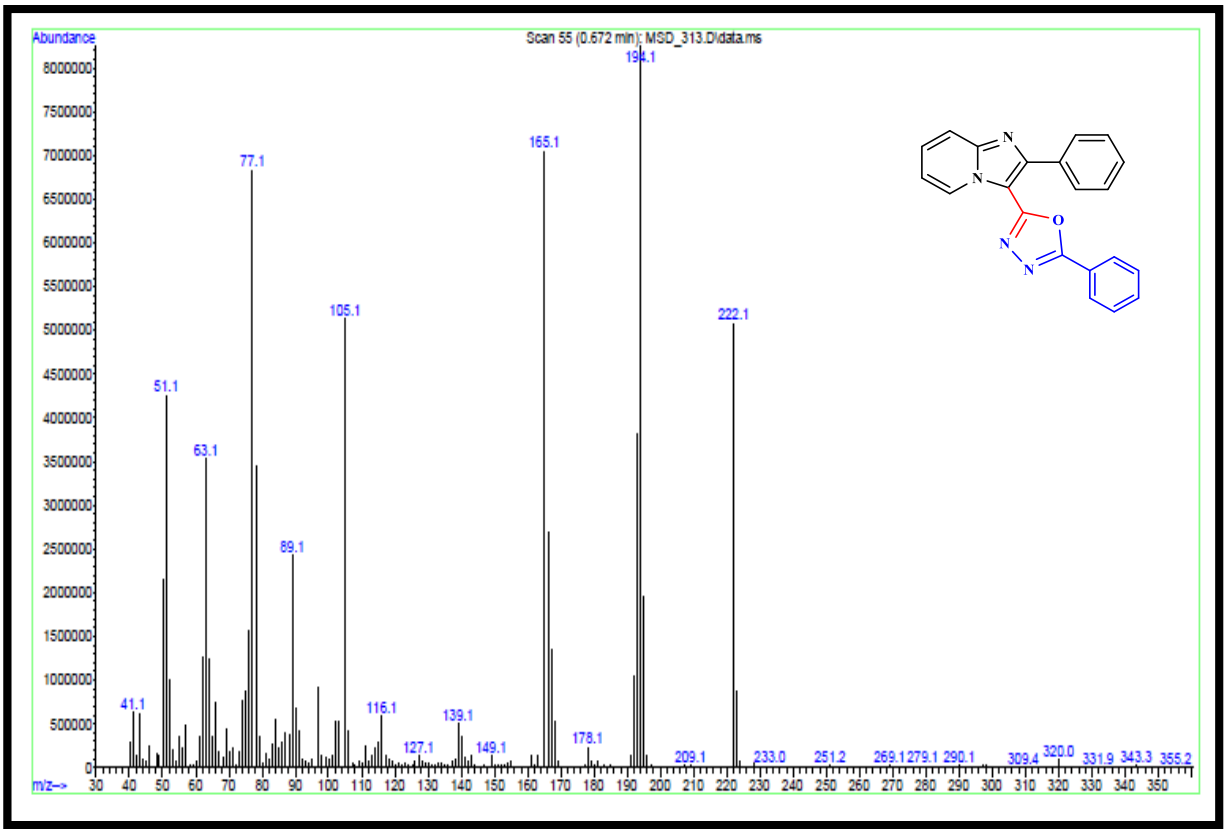

3-4-C
MeO
N
N
O
N
N
f1 (ppm)

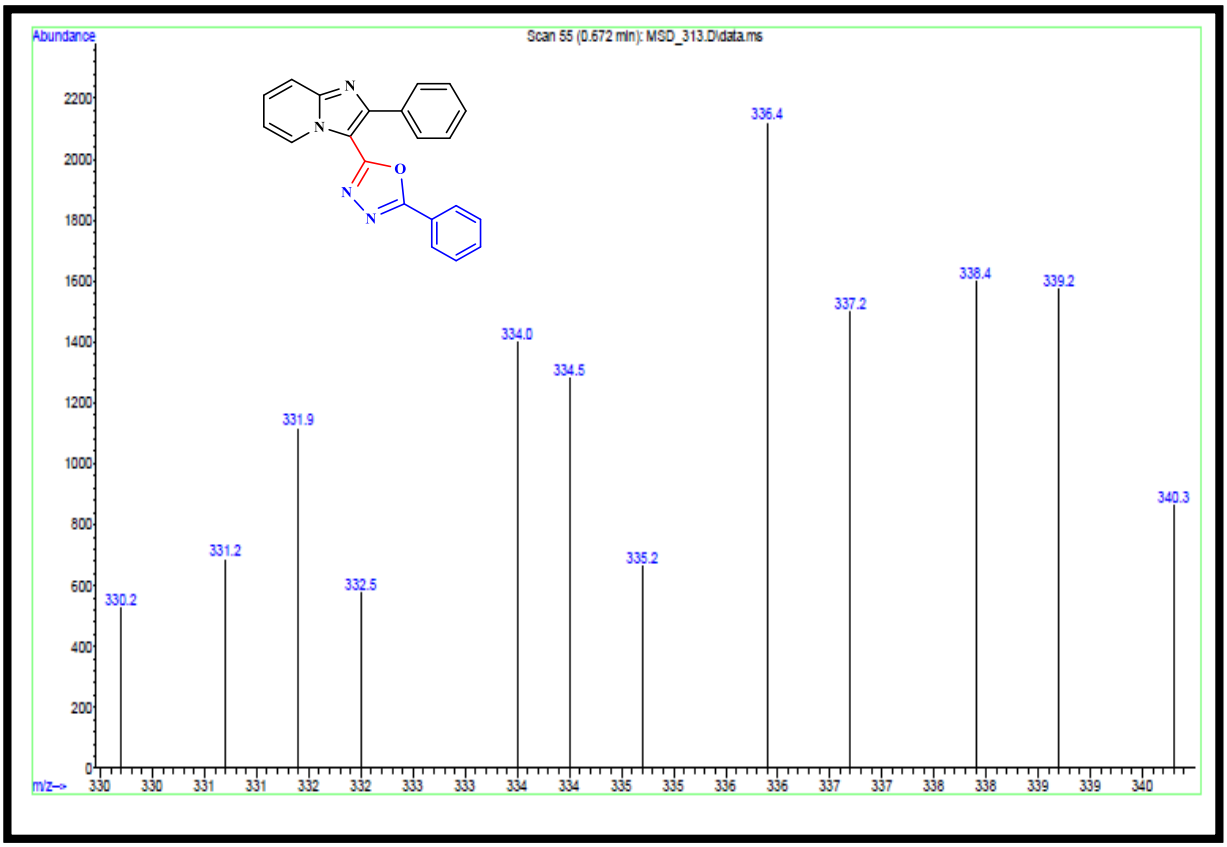

Abundance
Scan 124 (1.406 min): MSD_314.D\data.ms
43.1
69.0
97.1
MeO
N
O
m/z-->

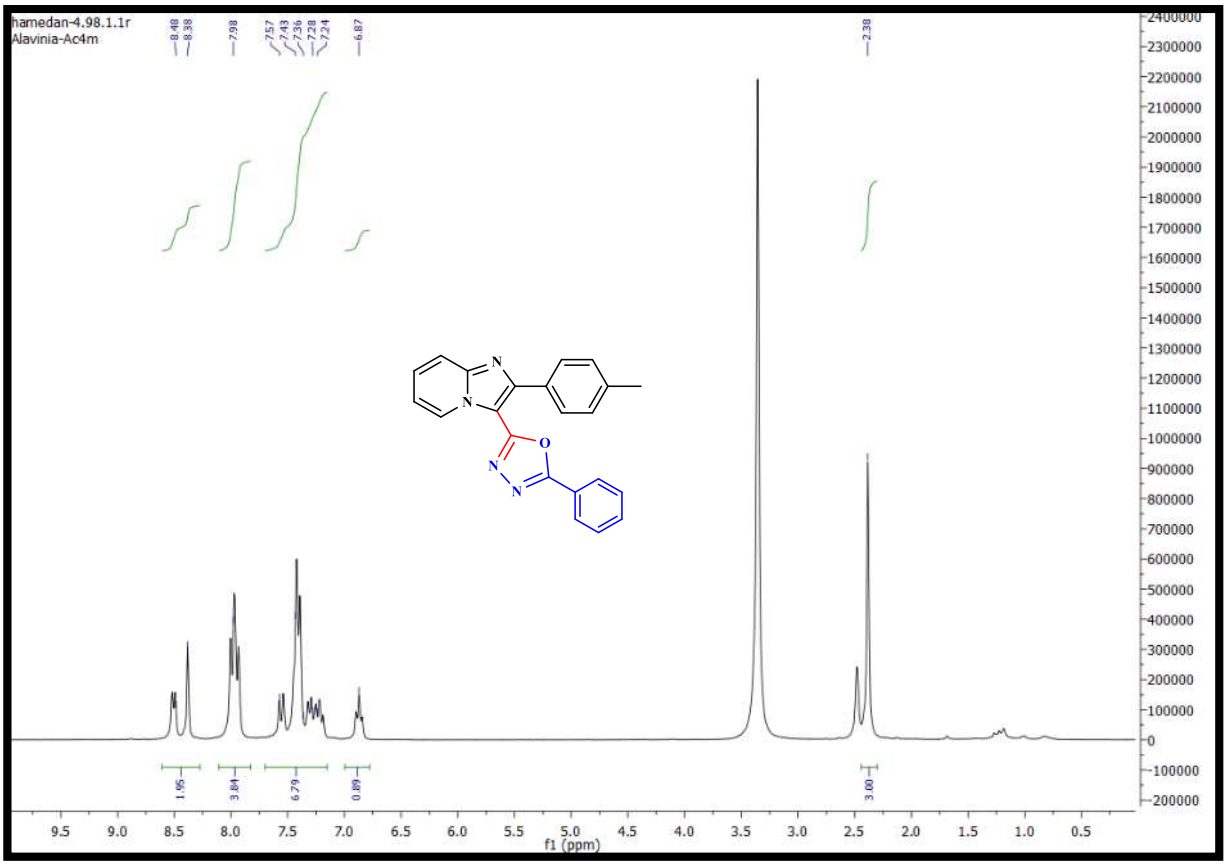

Abundance
Scan 124 (1.406 min): MSD_314.D\data.ms
396.9
394.9
396.0
392.4
393.3
398.2
398.6
399.2
399.8
401.4
MeO
N
O
m/z-->

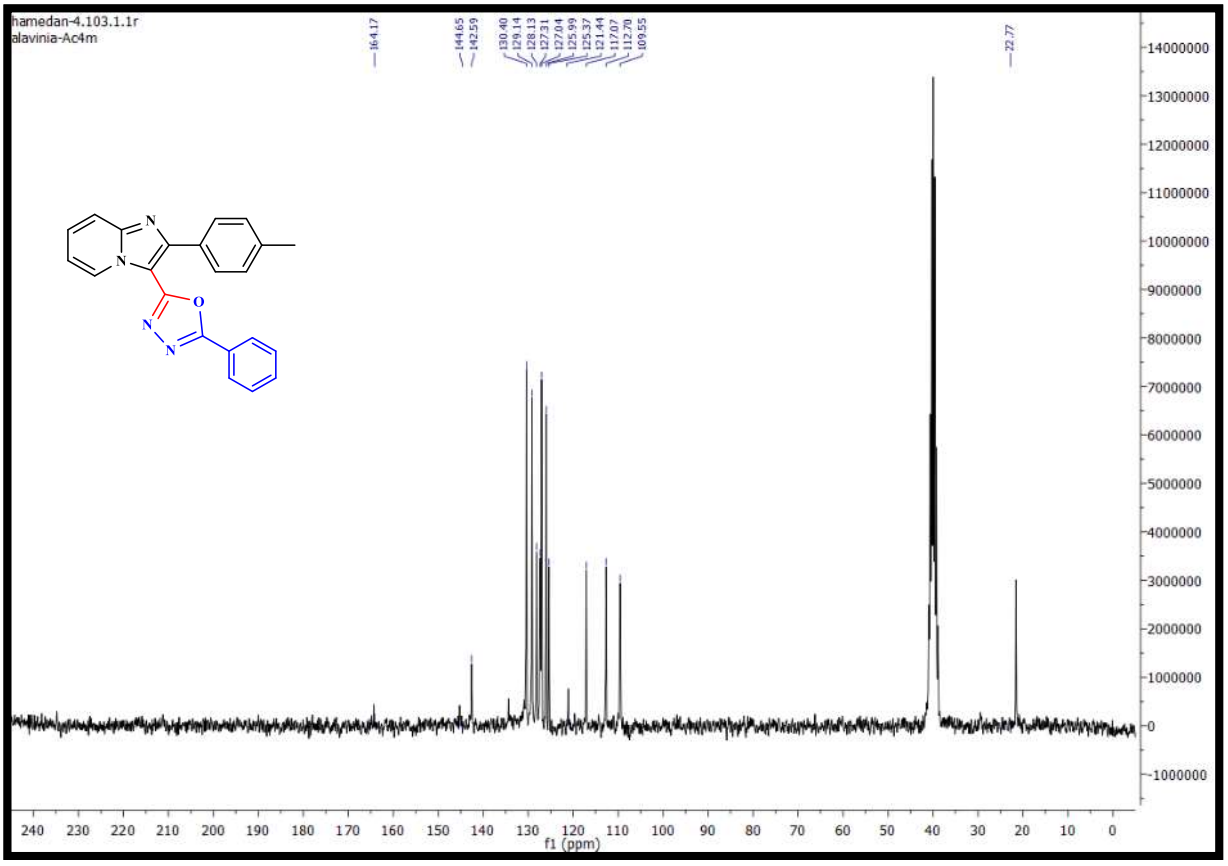

hamedan-4.99.1.1r
Alavinia-ACH
8.52
8.36
8.13
7.93
7.63
7.40
7.29
6.85
1.92
4.09
8.00
0.89
f1 (ppm)

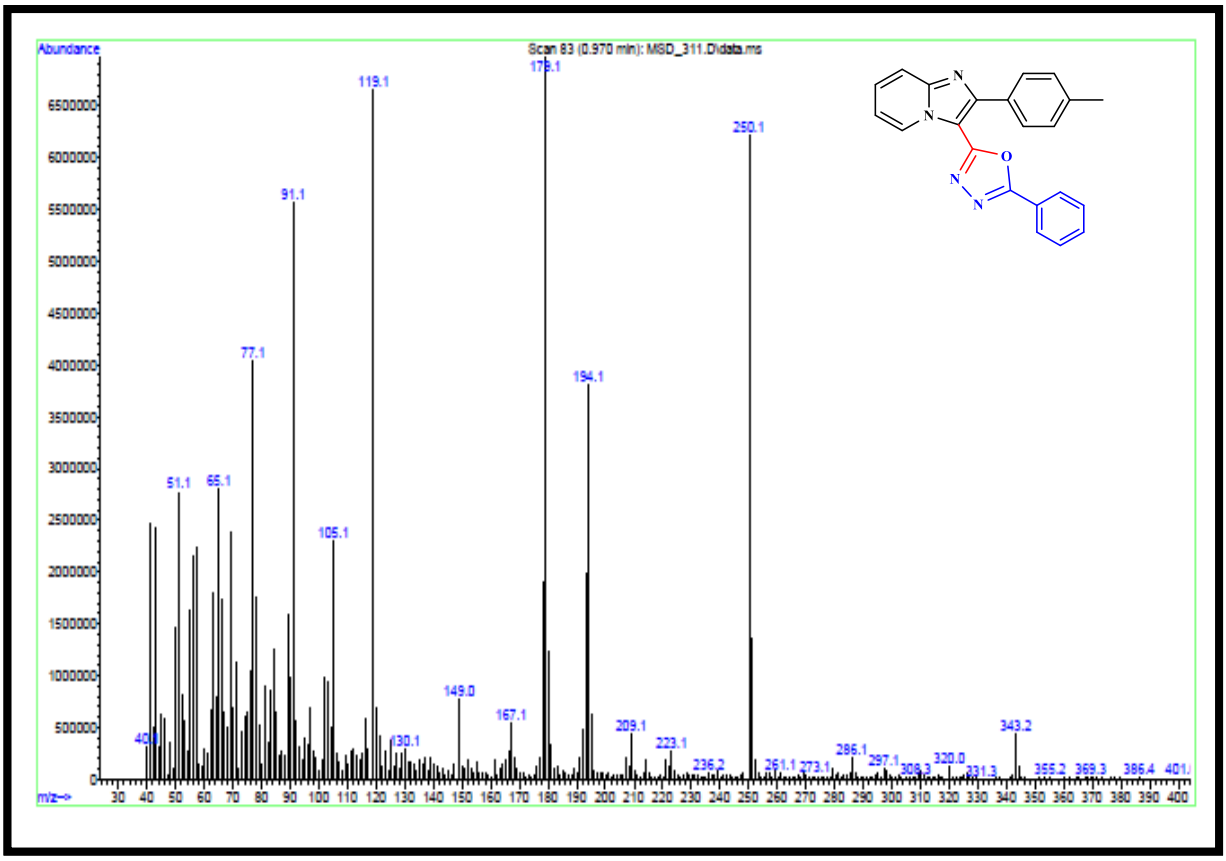

hamedan-4.104.1.1r
Alavinia-ACH
164.29
145.15
144.76
134.35
132.50
129.86
129.14
128.13
127.31
127.13
126.00
125.37
123.78
117.06
112.70
109.55
f1 (ppm)

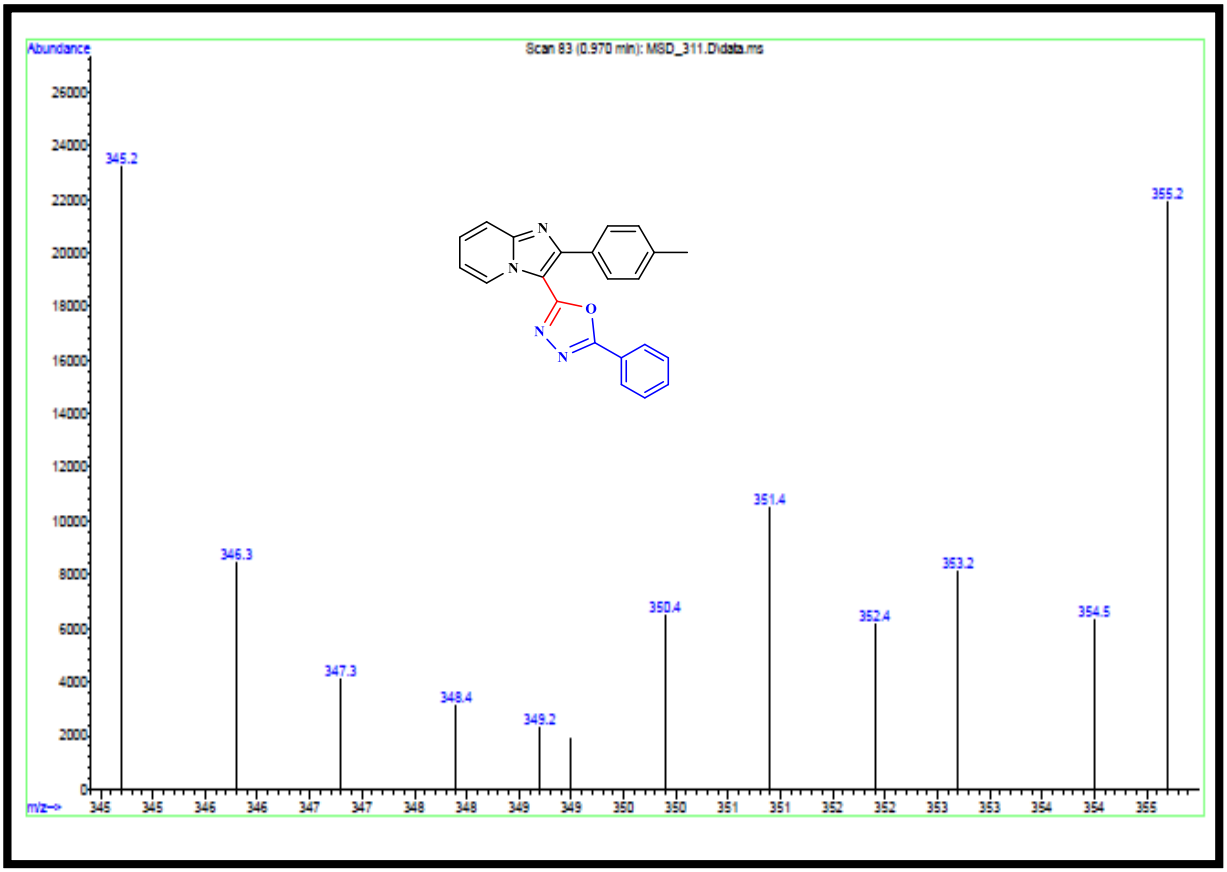

Abundance
Scan 55 (0.672 min): MSD_313.D\data.ms
194.1
165.1
77.1
105.1
222.1
51.1
63.1
89.1
41.1
116.1
139.1
127.1
149.1
178.1
209.1
233.0
251.2
269.1
279.1
290.1
309.4
320.0
331.9
343.3
355.2
m/z-->

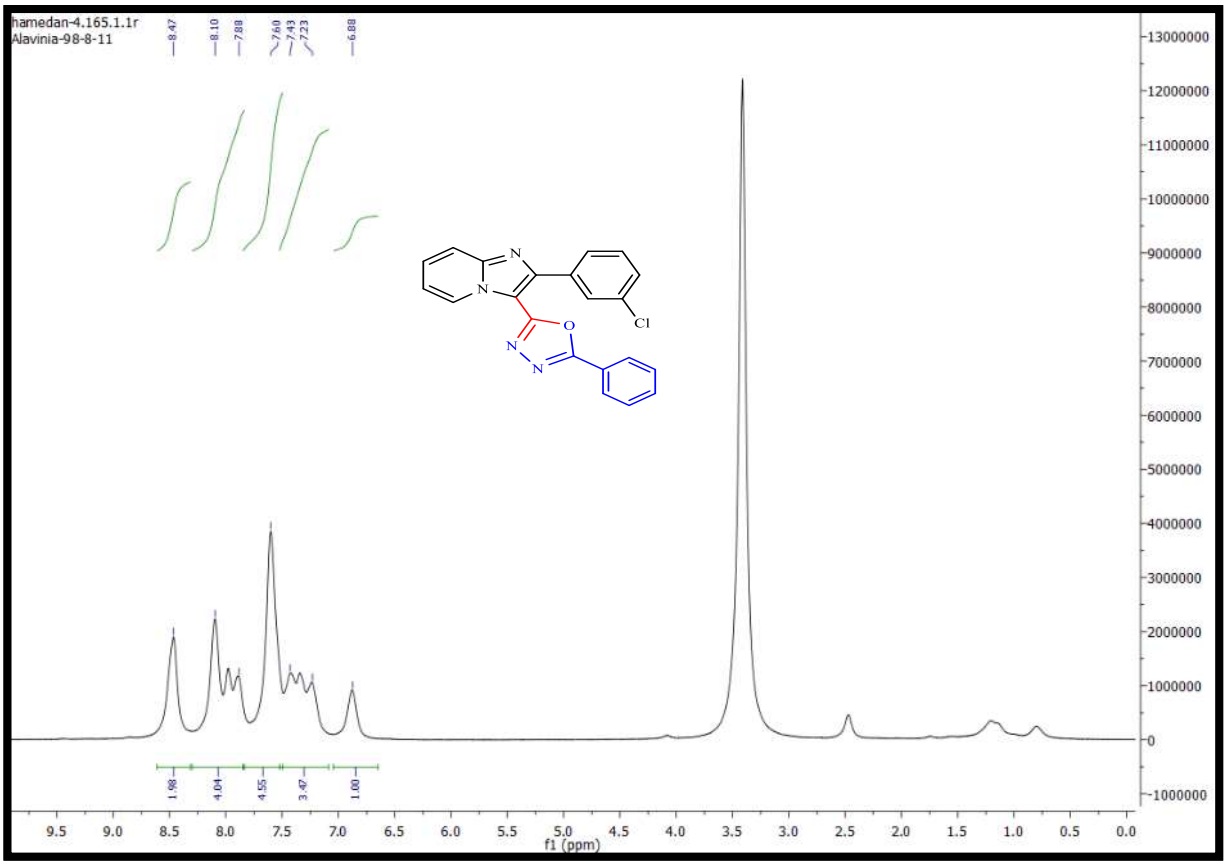

Abundance
Scan 55 (0.672 min): MSD_313.D\data.ms
336.4
338.4
339.2
337.2
334.0
334.5
331.9
340.3
331.2
335.2
332.5
330.2
m/z-->

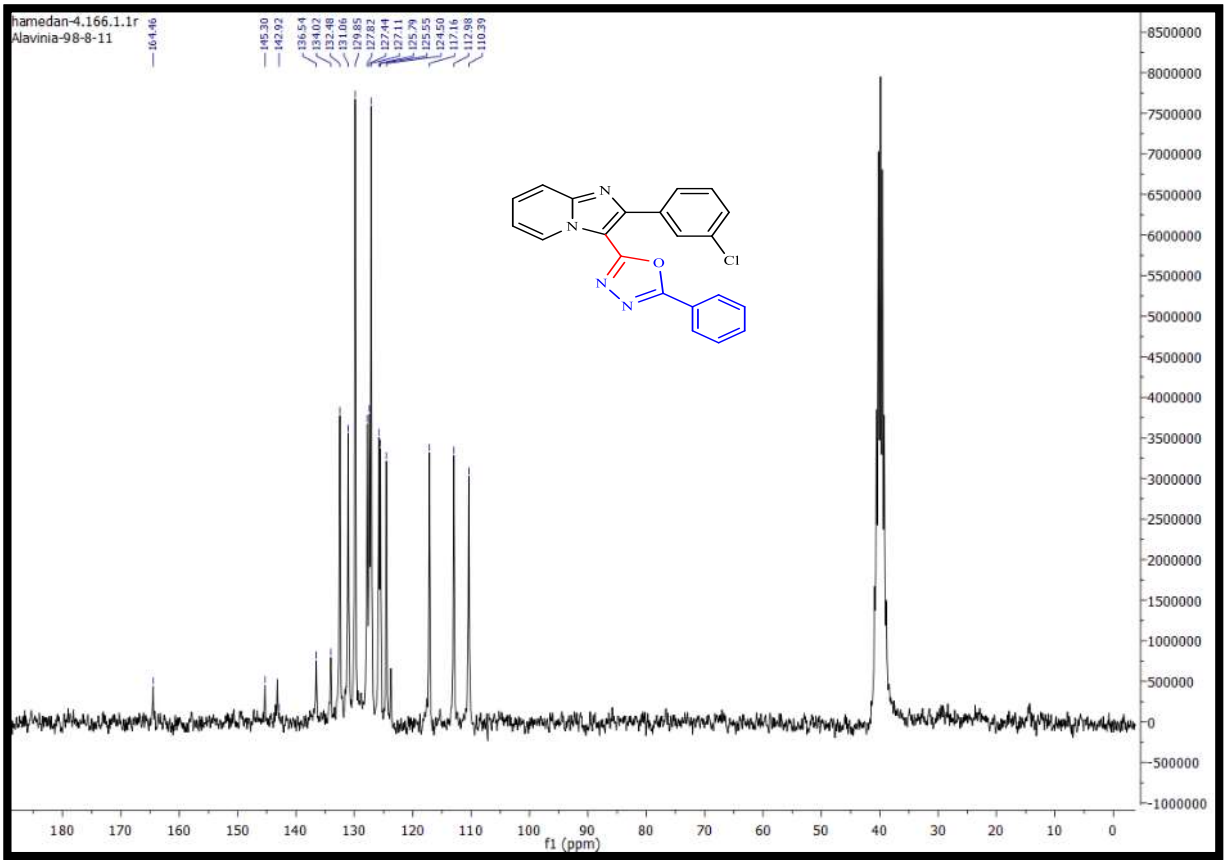

hamedan-4.98.1.1r
Alavinia-Ac4m
8.48
8.38
7.98
7.57
7.43
7.36
7.28
7.24
6.87
2.38
1.95
3.84
6.79
0.89
3.00
f1 (ppm)

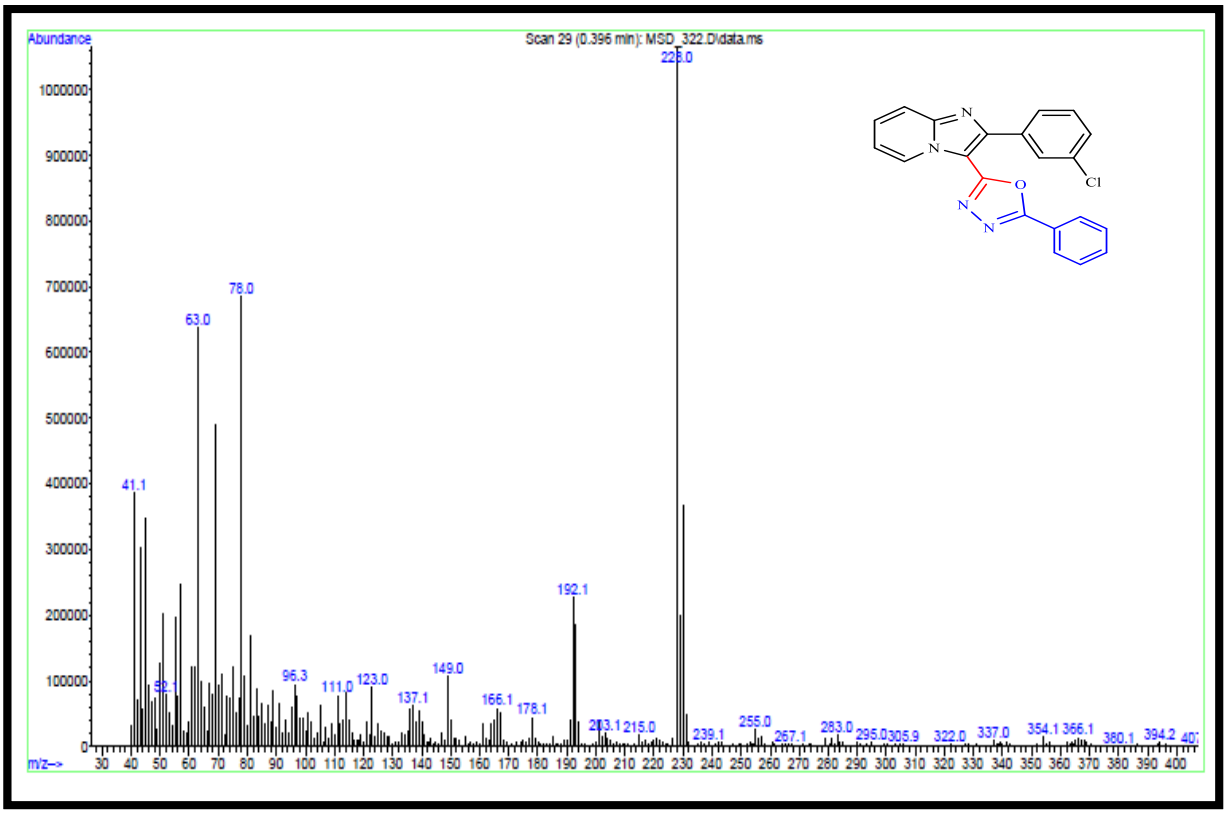

hamedan-4.103.1.1r
alavinia-Ac4m
164.17
144.65
142.59
130.40
129.14
128.13
127.31
127.04
125.99
125.37
121.44
117.07
112.70
109.55
22.77
f1 (ppm)

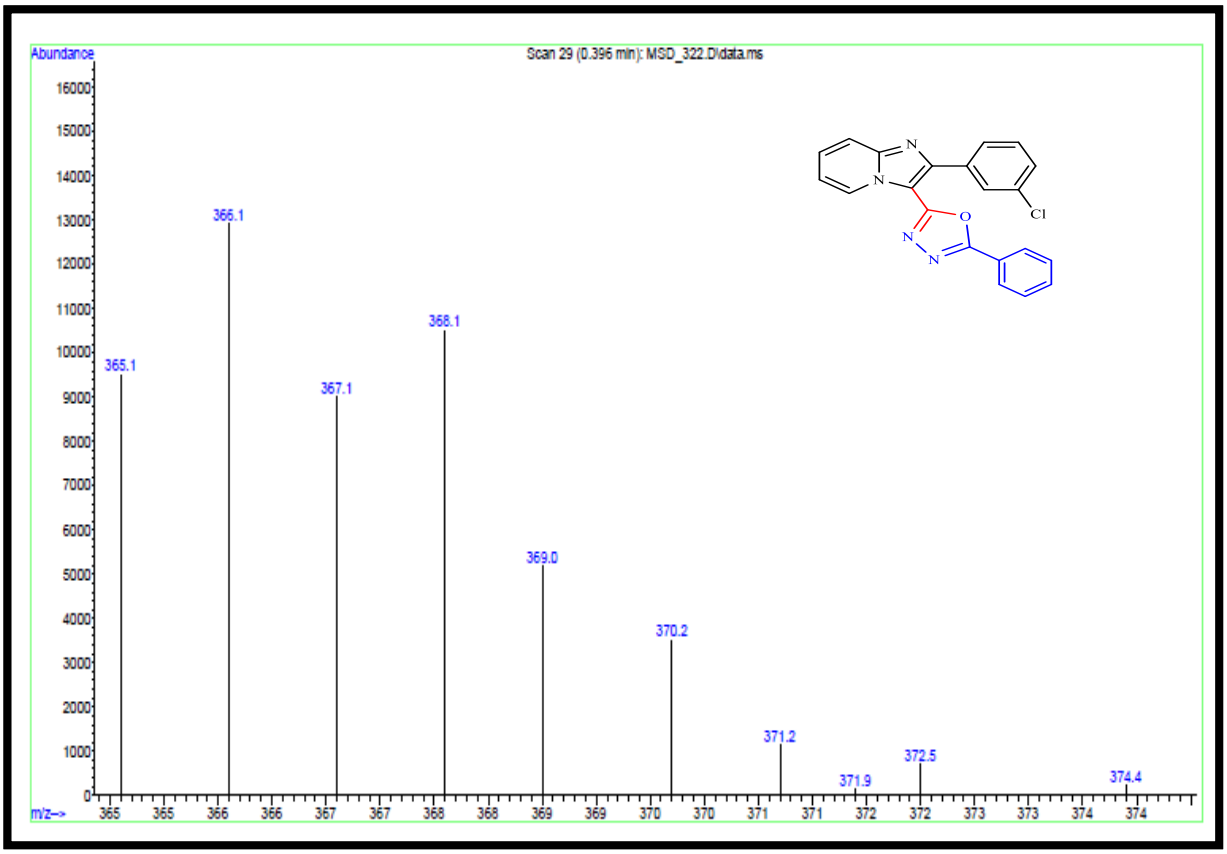

Abundance
Scan 83 (0.970 min): MSD_311.D\data.ms
178.1
119.1
250.1
91.1
77.1
194.1
51.1
65.1
105.1
149.0
167.1
209.1
223.1
343.2
m/z-->

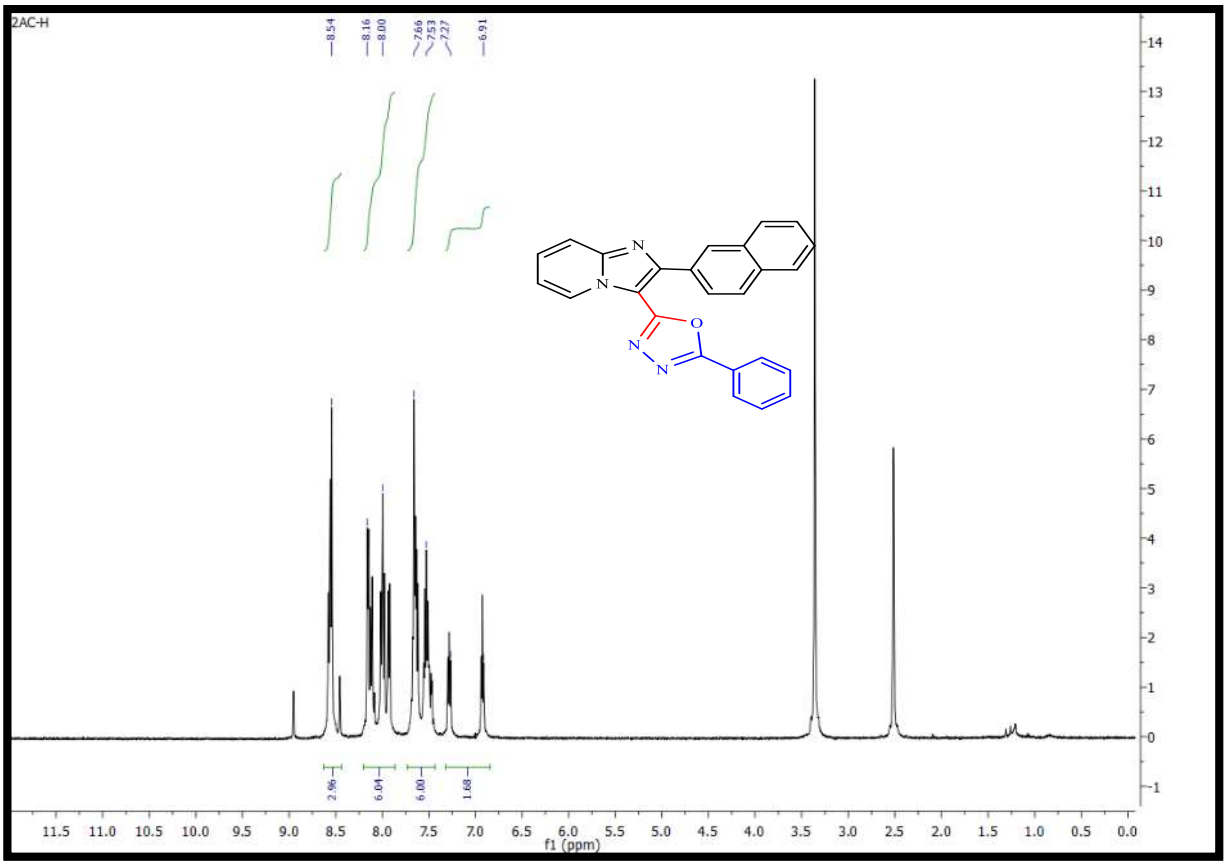

Abundance
Scan 83 (0.970 min): MSD_311.D\data.ms
345.2
355.2
351.4
346.3
353.2
350.4
352.4
354.5
347.3
348.4
349.2
m/z-->

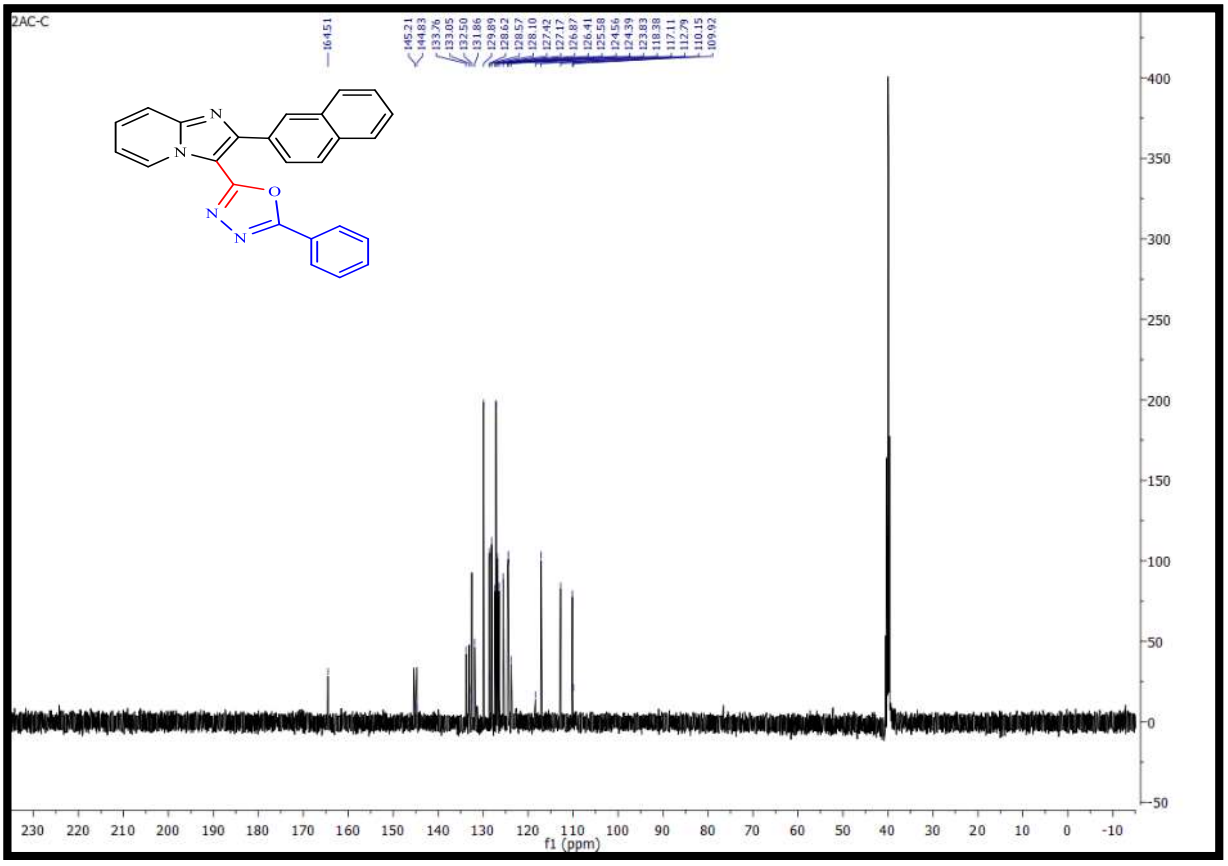

hamedan-4.165.1.1r
Alavinia-98-8-11
8.47
8.10
7.88
7.60
7.43
7.23
6.88
Cl
1.98
4.04
4.55
3.47
1.00
f1 (ppm)

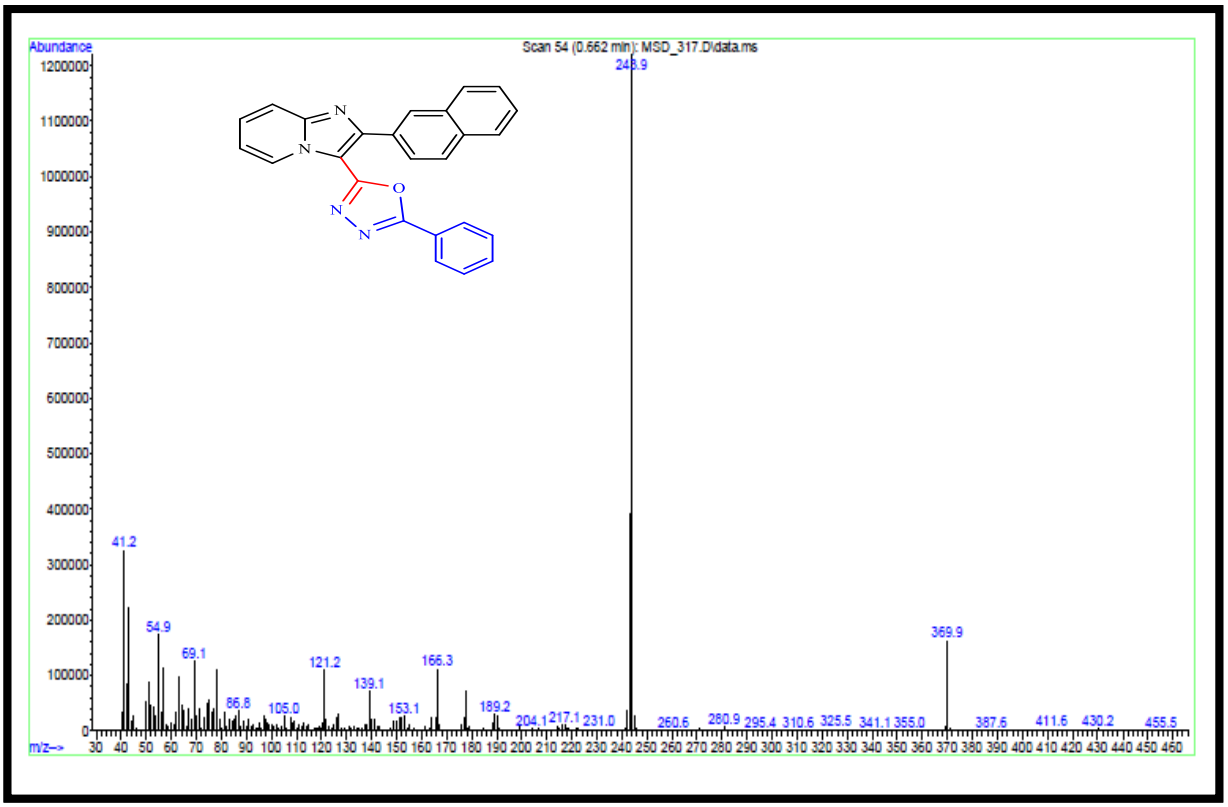

hamedan-4.166.1.1r
Alavinia-98-8-11
164.46
145.30
142.92
136.54
134.02
132.48
131.06
129.85
127.82
127.44
127.11
125.79
125.55
124.50
117.16
112.98
110.39
Cl
f1 (ppm)

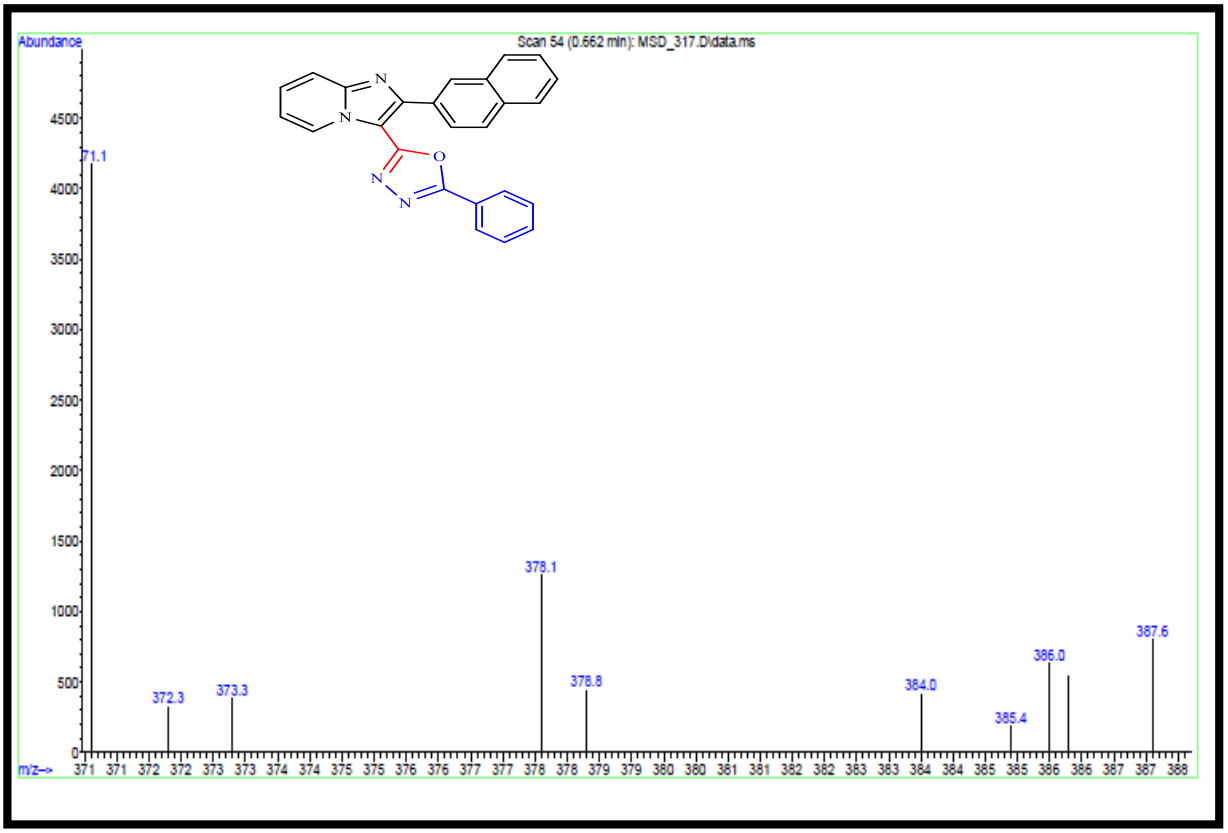

Abundance
Scan 29 (0.396 min): MSD_322.D\data.ms
225.0
78.0
63.0
41.1
192.1
96.3
111.0
123.0
149.0
137.1
166.1
178.1
255.0
283.0
354.1
366.1
m/z-->

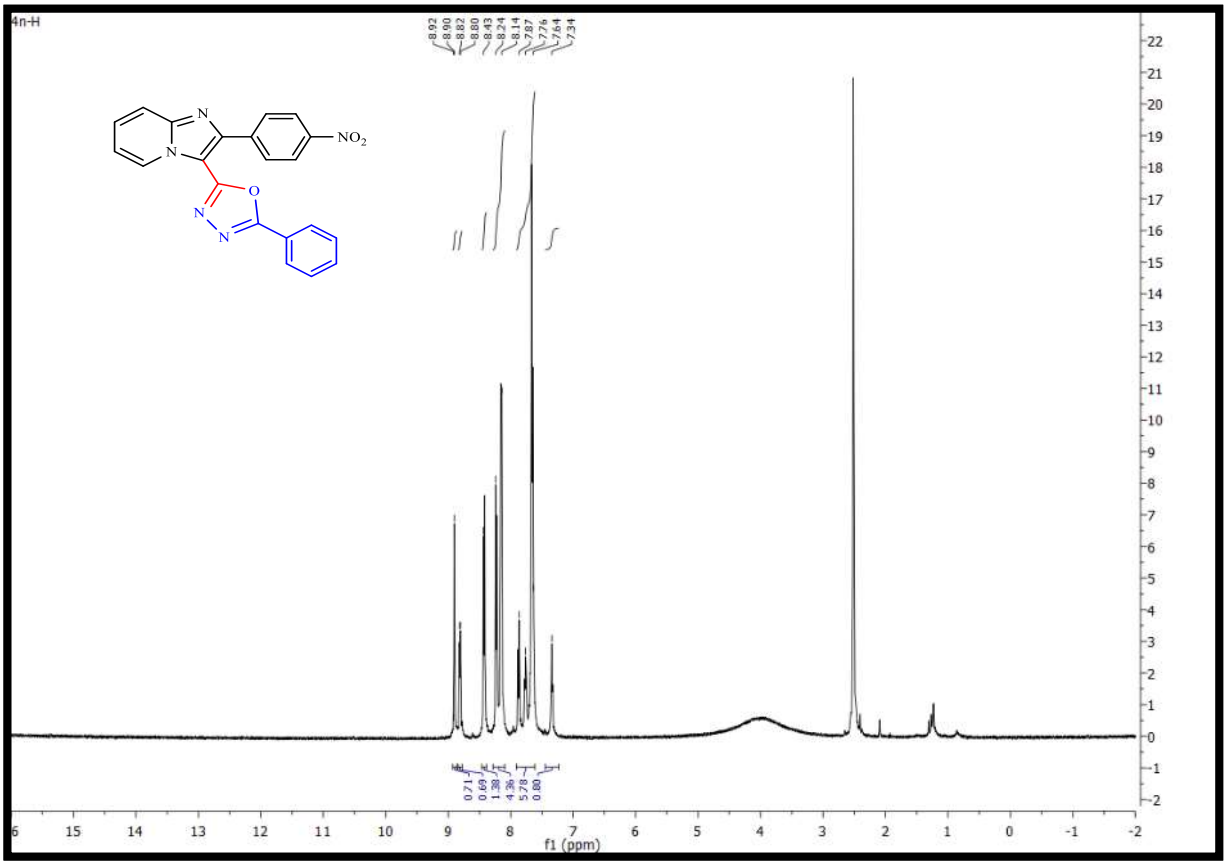

Abundance
Scan 29 (0.396 min): MSD_322.D\data.ms
366.1
365.1
367.1
368.1
369.0
370.2
371.2
372.5
371.9
374.4
m/z-->

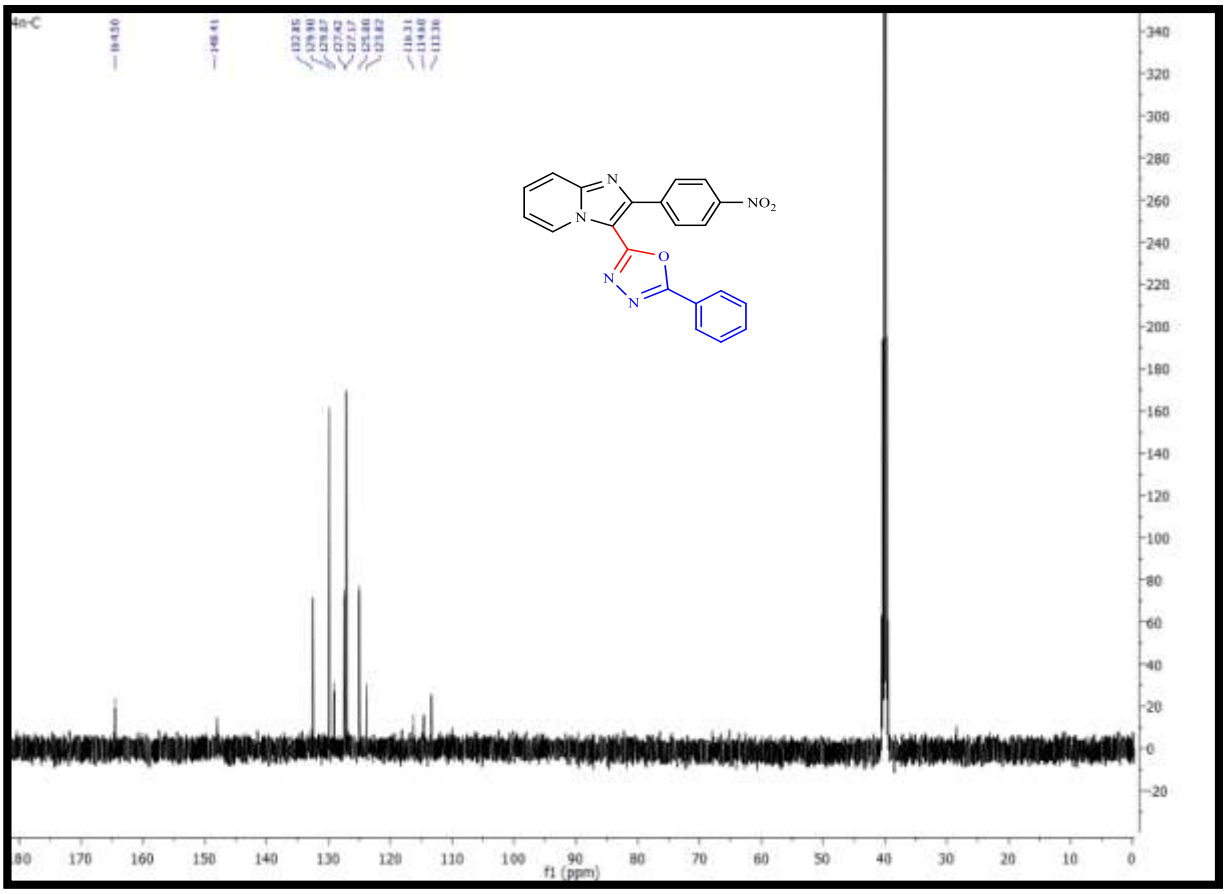

2AC-H
8.54
8.16
8.00
7.66
7.53
7.27
6.91
2.96
6.04
6.00
1.68
f1 (ppm)

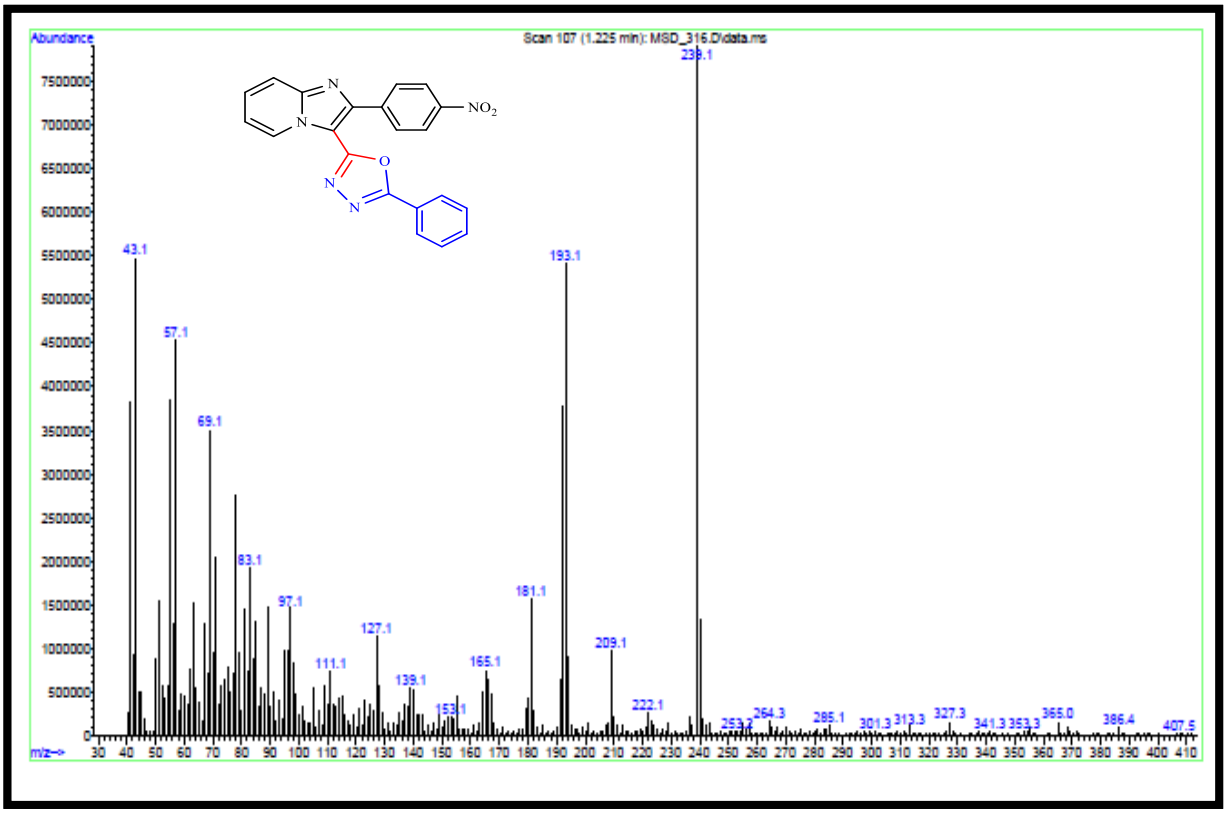

2AC-C
f1 (ppm)

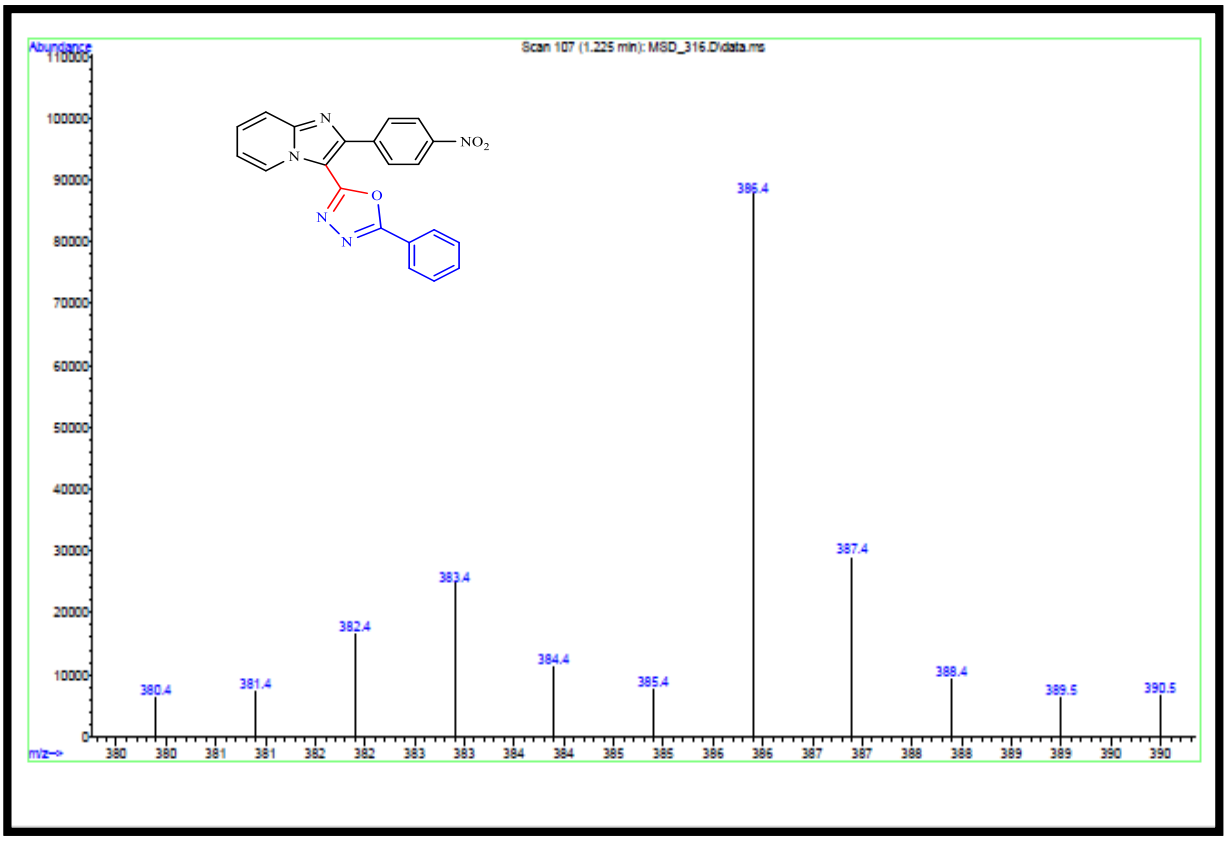

Abundance
Scan 54 (0.662 min): MSD_317.D\data.ms
243.9
41.2
54.9
69.1
86.8
105.0
121.2
139.1
153.1
165.3
189.2
204.1
217.1
231.0
260.6
280.9
295.4
310.6
325.5
341.1
355.0
369.9
387.6
411.6
430.2
455.5
m/z-->

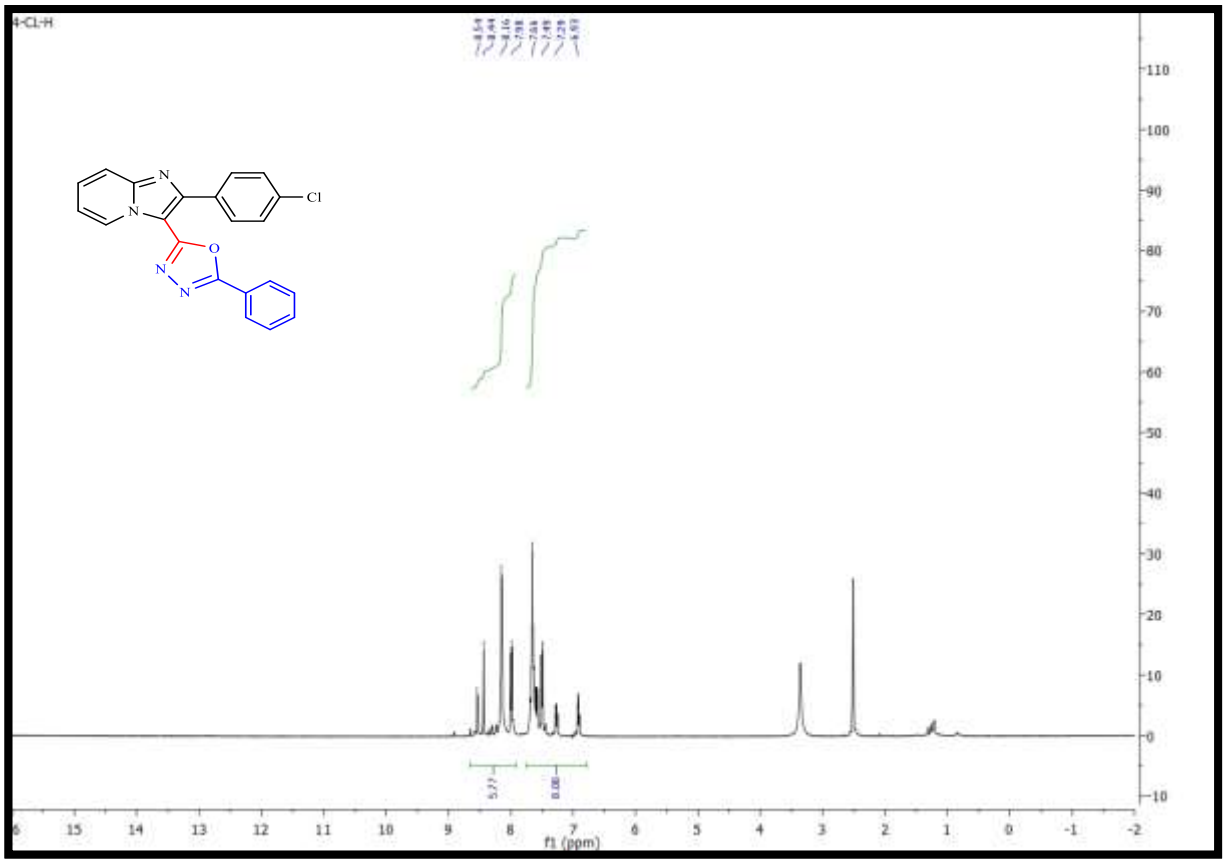

Abundance
Scan 54 (0.662 min): MSD_317.D\data.ms
71.1
372.3
373.3
378.1
378.8
384.0
385.4
386.0
387.6
m/z-->

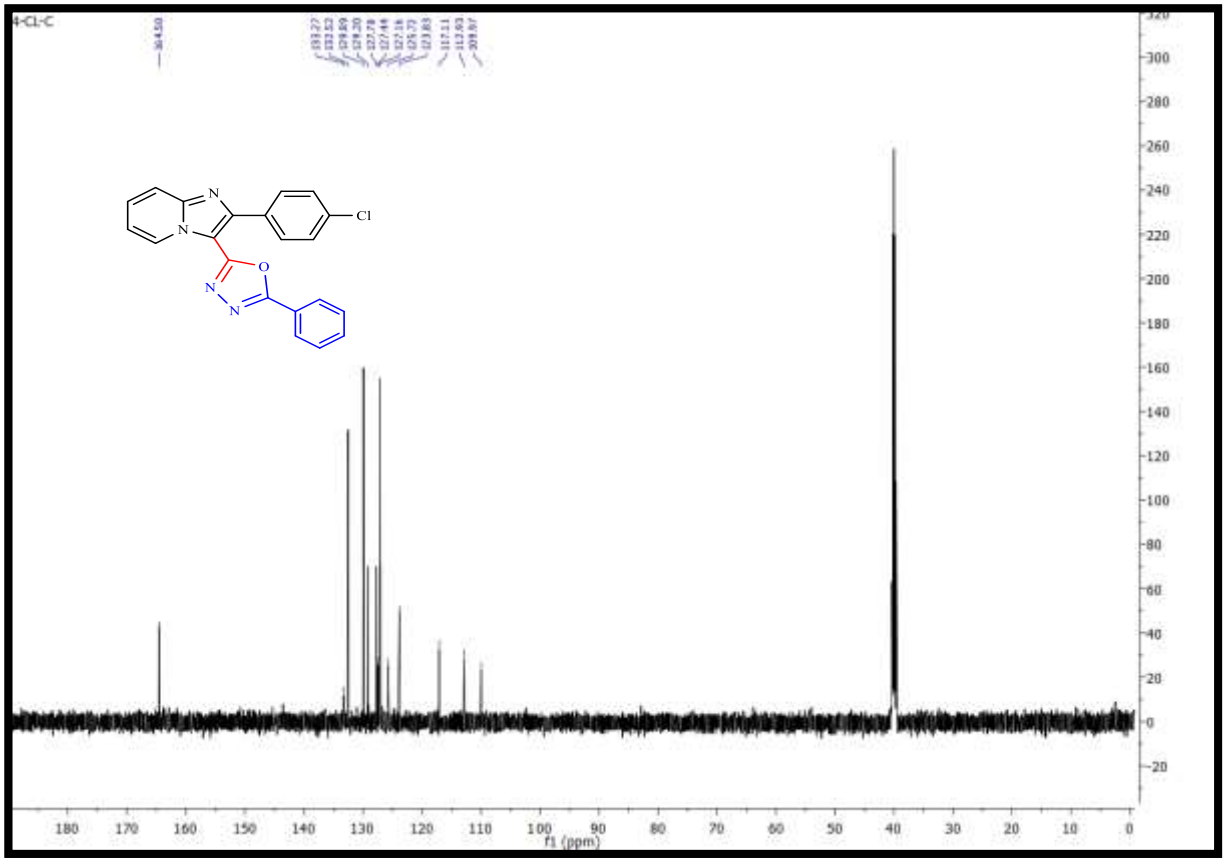

4n-H
8.92
8.90
8.82
8.80
8.43
8.24
8.14
7.87
7.76
7.64
7.34
NO2
f1 (ppm)

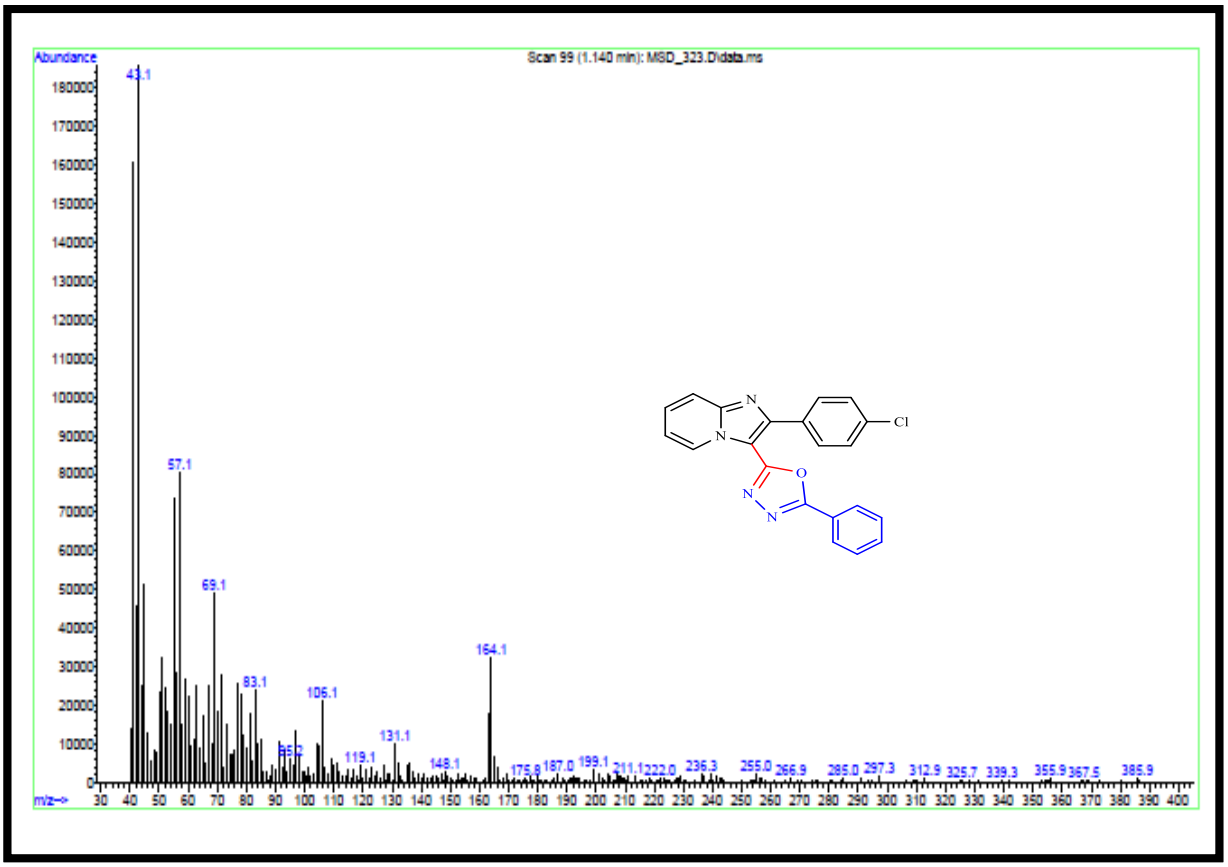

4n-C
NO2
f1 (ppm)

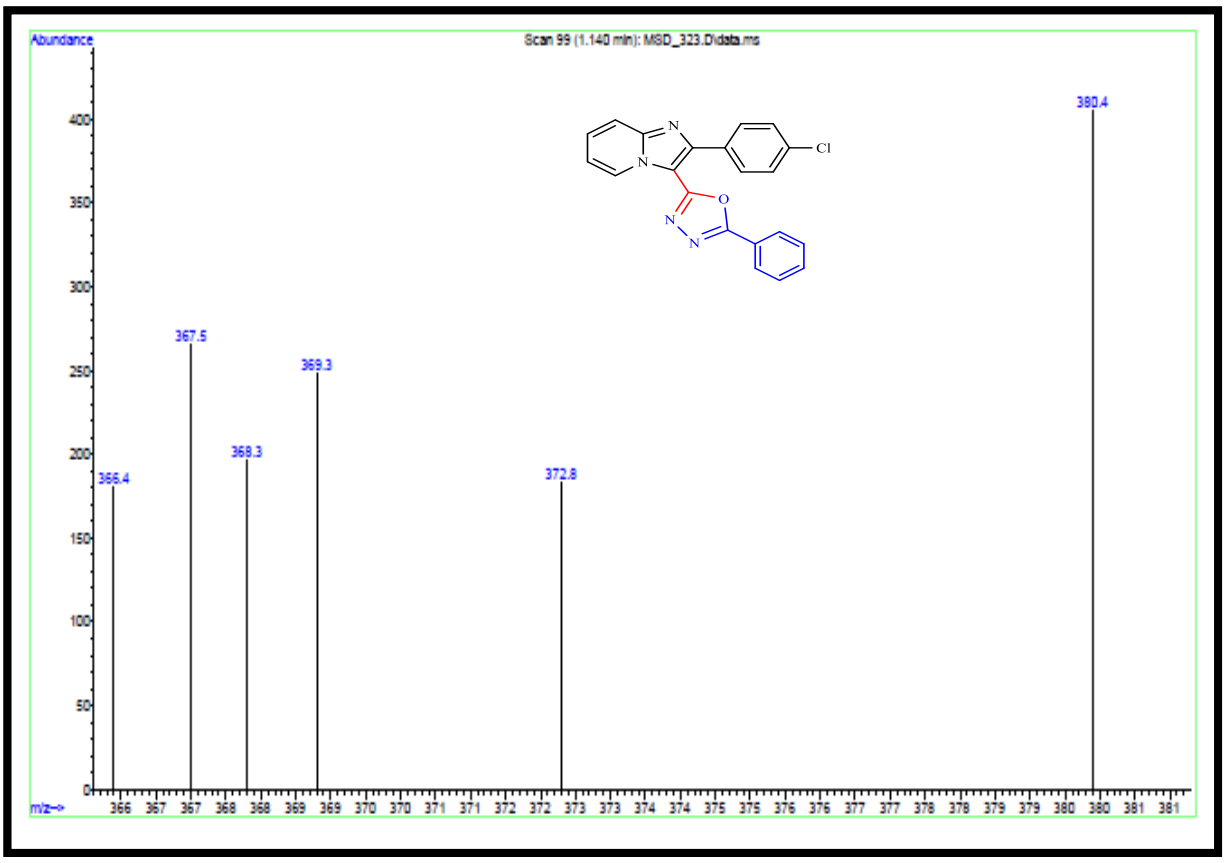

Abundance
Scan 107 (1.225 min): MSD_316.D\data.ms
238.1
193.1
43.1
57.1
69.1
83.1
97.1
111.1
127.1
139.1
153.1
165.1
181.1
209.1
222.1
253.2
264.3
285.1
301.3
313.3
327.3
341.3
353.3
365.0
386.4
407.5
m/z-->

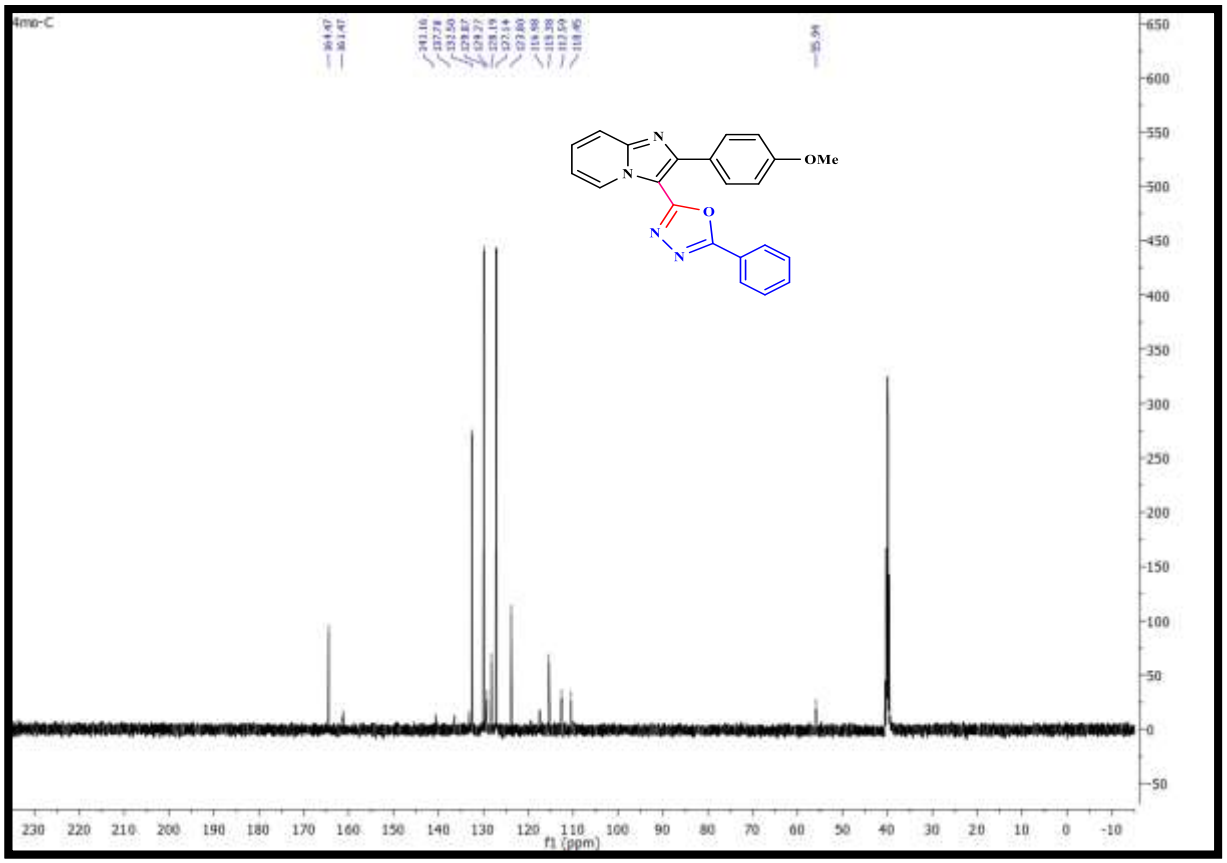

Abundance
Scan 107 (1.225 min): MSD_316.D\data.ms
386.4
387.4
383.4
382.4
384.4
385.4
388.4
380.4
381.4
389.5
390.5
m/z-->

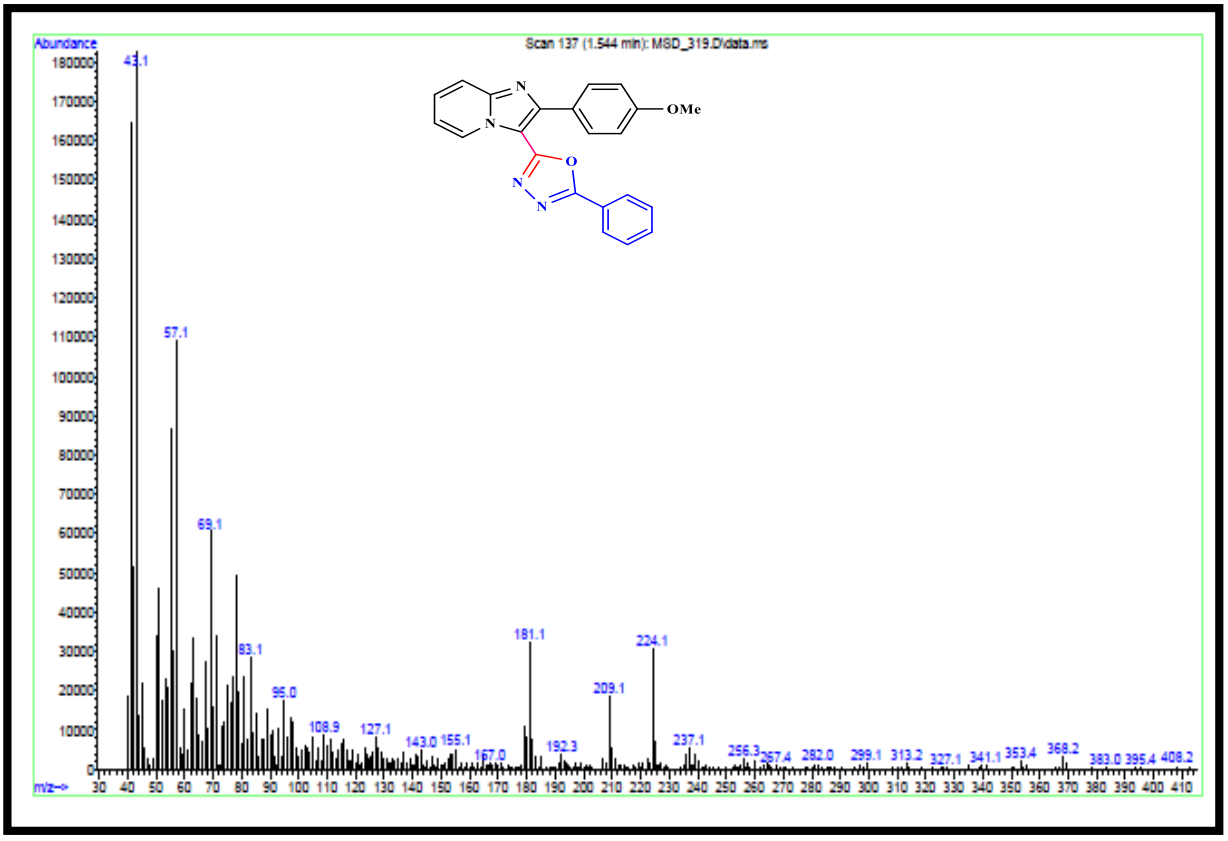

4-Cl-H
Cl
N
O
f1 (ppm)

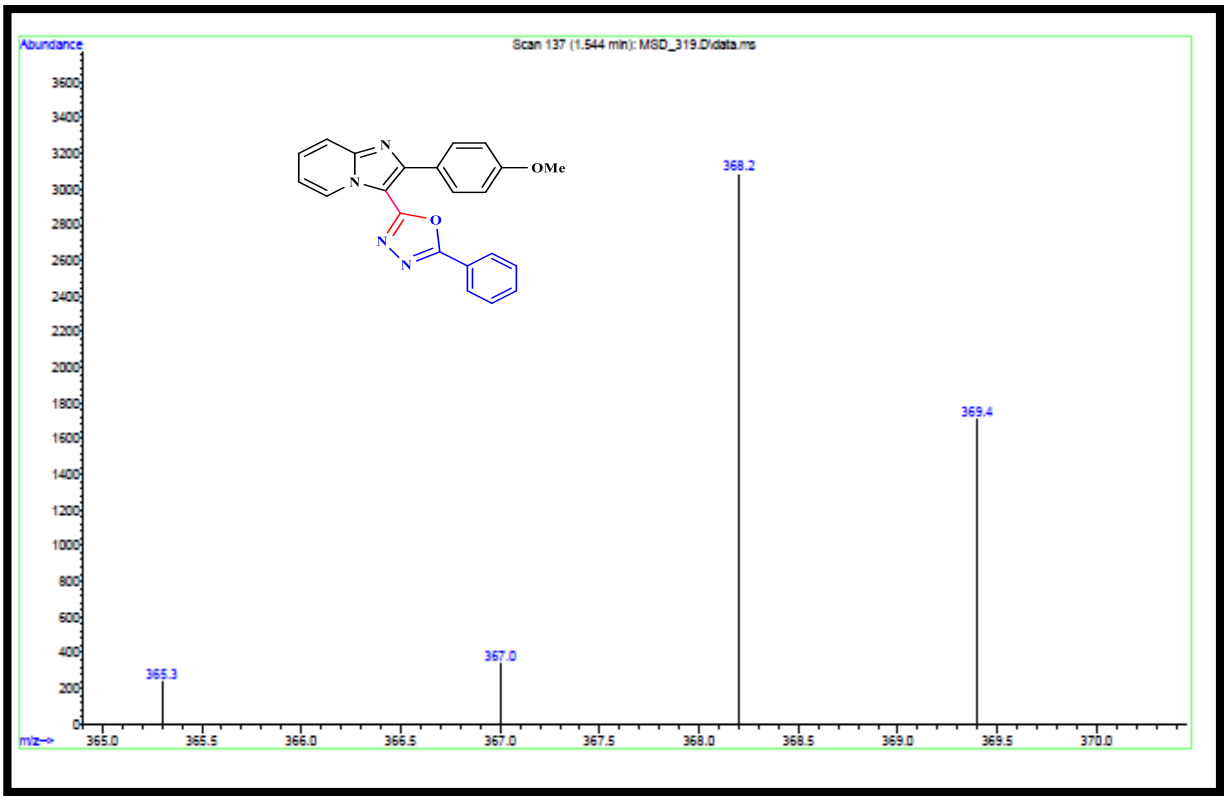

4-Cl-C
Cl
N
O
f1 (ppm)

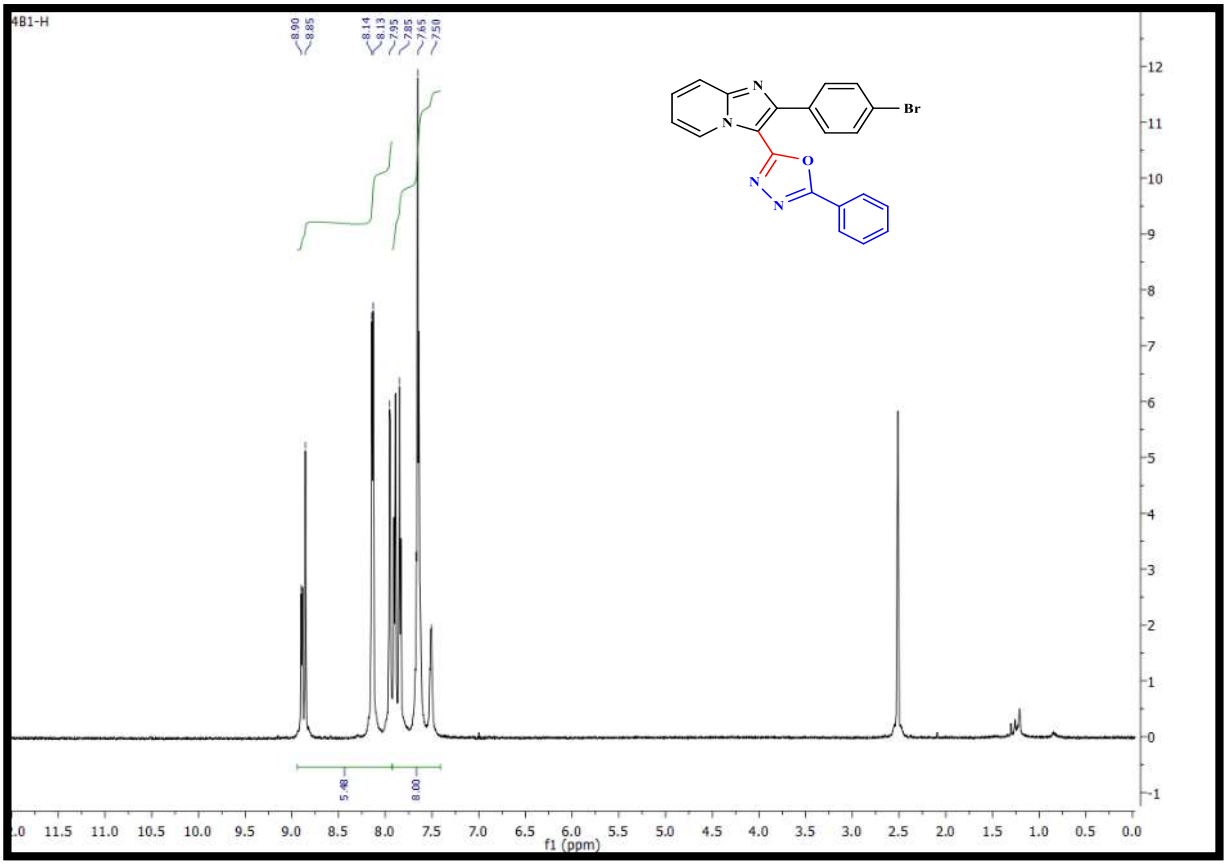

Abundance
Scan 99 (1.140 min): MSD_323.D\data.ms
43.1
57.1
69.1
83.1
105.1
131.1
164.1
m/z-->

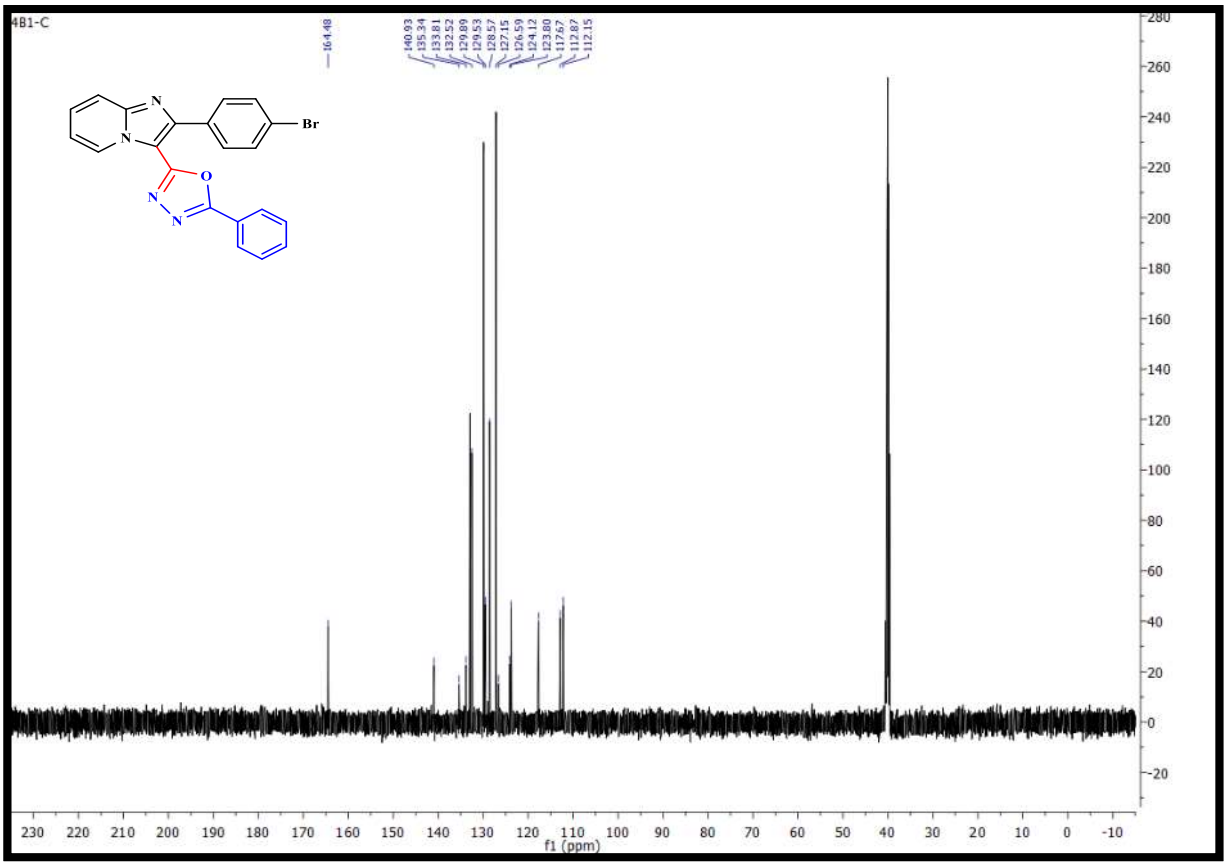

Abundance
Scan 99 (1.140 min): MSD_323.D\data.ms
380.4
367.5
369.3
368.3
365.4
372.8
m/z-->

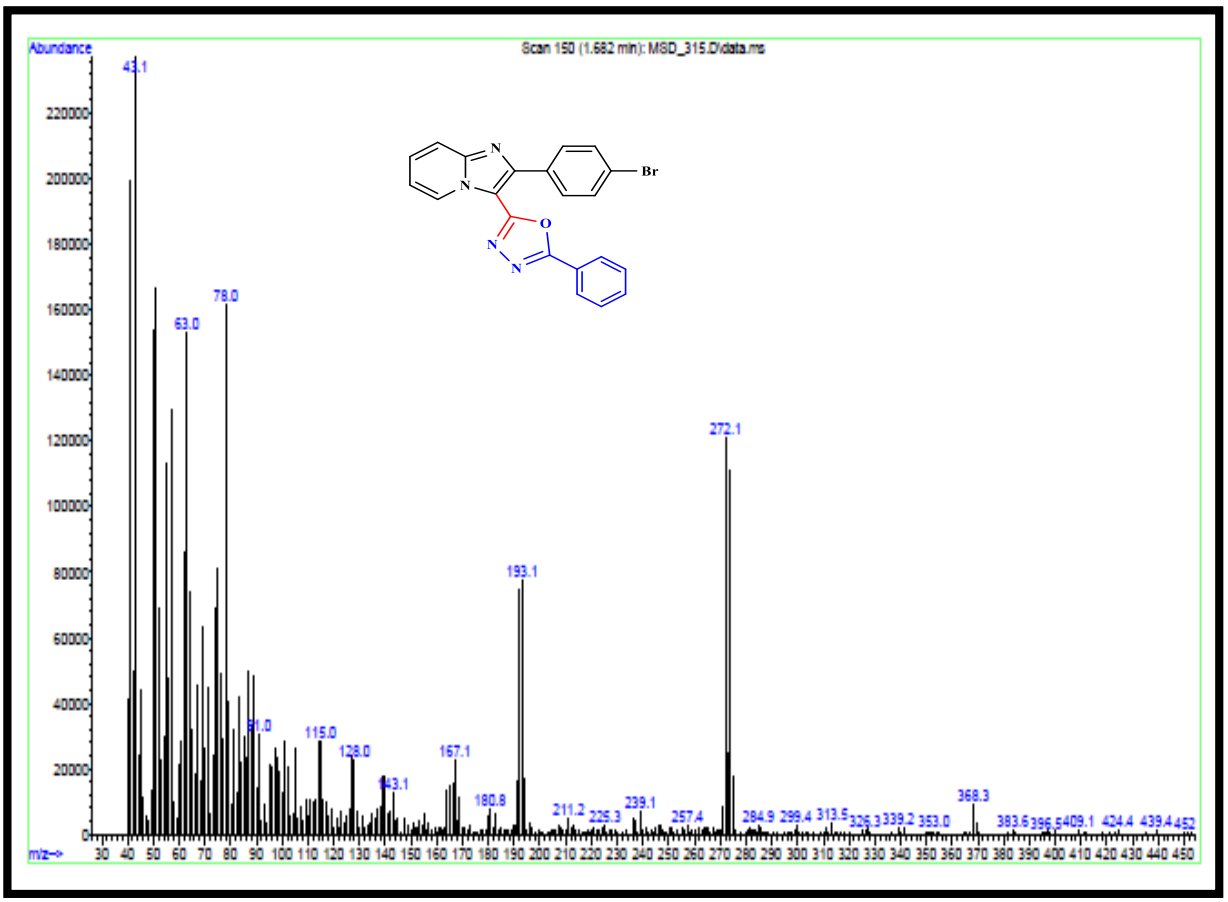

4mo-C
OMe
f1 (ppm)

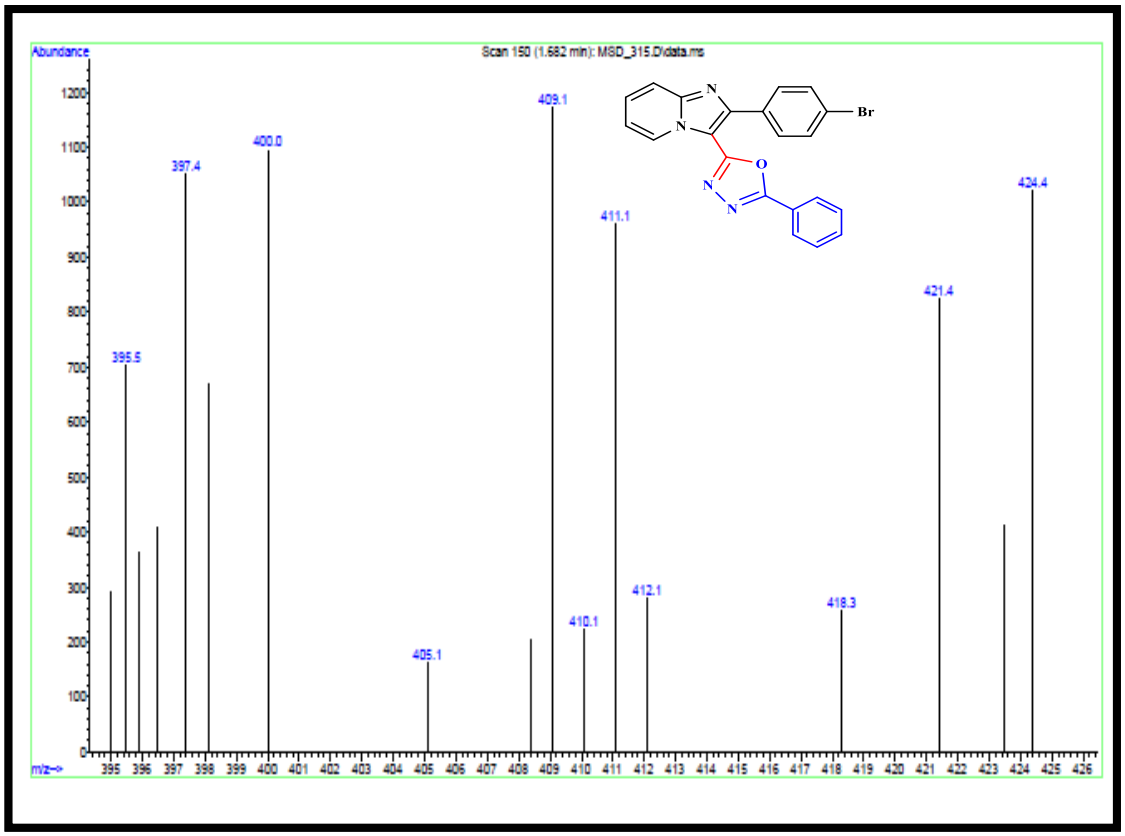

Abundance
Scan 137 (1.544 min): MSD_319.D\data.ms
OMe
m/z-->


Abundance
Scan 137 (1.544 min): MSD_319.D\data.ms
OMe
368.2
369.4
367.0
365.3
m/z-->


4B1-H
Br
f1 (ppm)


4B1-C
164.48
140.93
135.94
133.61
132.52
129.99
129.53
128.57
127.15
126.59
124.12
123.80
117.67
112.87
112.15
Br
f1 (ppm)


Abundance
Scan 150 (1.682 min): MSD_315.D\data.ms
Br
m/z-->


Abundance
Scan 150 (1.682 min): MSD_315.D\data.ms
409.1
400.0
397.4
411.1
424.4
421.4
395.5
412.1
418.3
410.1
405.1
m/z-->
Br
N
N
O
N
N