

Electronic Supplemental Information (ESI)

Transition metal and base free synthesis of 2-aryl-2-oxazolines from aldehydes with β -amino alcohols catalyzed by Potassium Iodide

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Table of contents

1. General information.....	2
2. Experimental section.....	2
3. Spectroscopic data of products and Copies of ¹ H NMR, ¹³ C NMR and GC-MS for Products	4
4. References	34

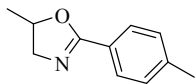
General Information

All chemicals were purchased from Sigma-Aldrich and S.D Fine Chemicals, Pvt. Ltd. India and used as received. ACME silica gel (100–200 mesh) was used for column chromatography and thin-layer chromatography was performed on Merck-precoated silica gel 60-F₂₅₄ plates. All the other chemicals and solvents were obtained from commercial sources and purified using standard methods. The ¹H spectra were recorded on a Varian-Gemini 200 MHz, Bruker-Avance 300 MHz Spectrometer. Chemical shifts (δ) are reported in ppm, using TMS ($\delta = 0$) as an internal standard in CDCl₃. GC were recorded on Shimadzu-2014 using BP-01 (30M X 0.25 mm X 1.0 μ m) column. GC-MS spectra were recorded on Thermo Trace DSQ GC-MS spectrometer using BP-01 (30M X 0.25 mm X 1.0 μ m) column.

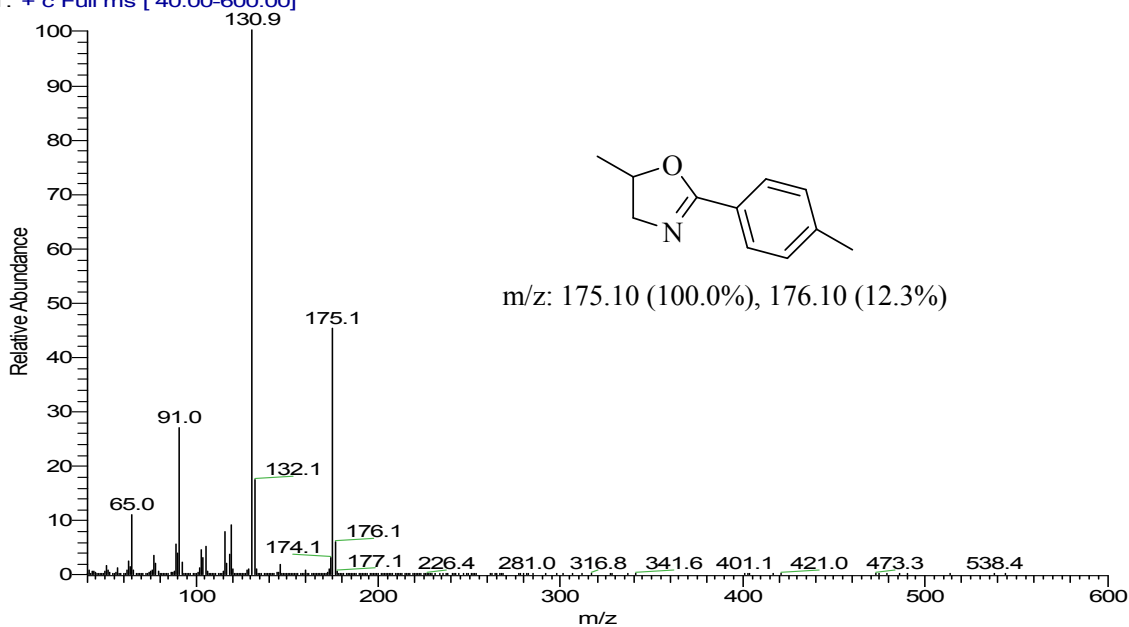
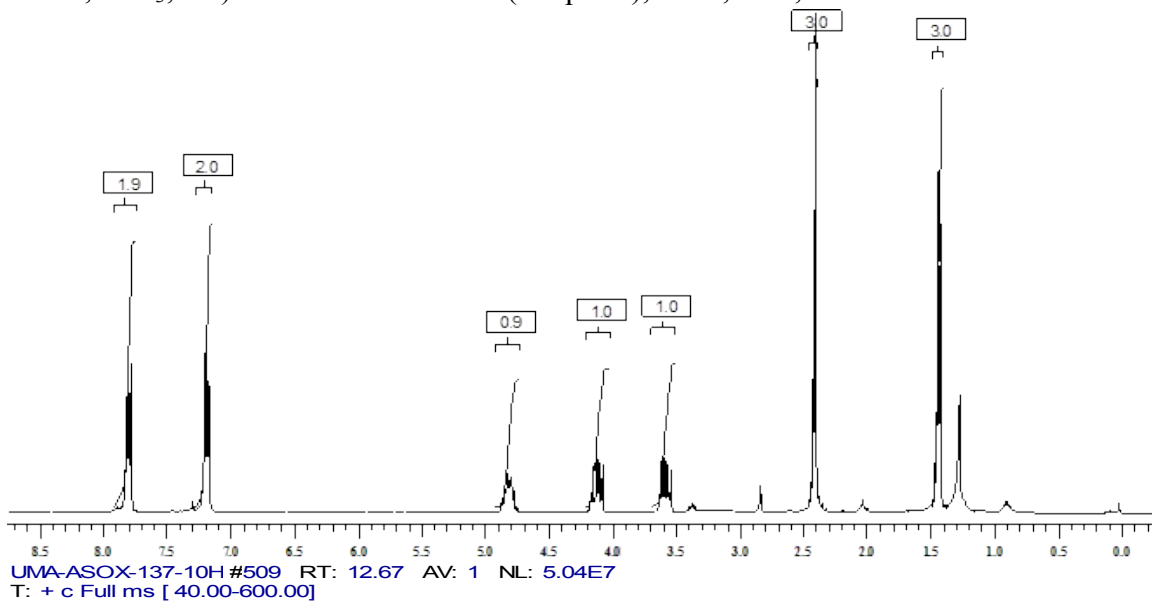
Typical experimental procedure for synthesis of 2-oxazoline from aldehyde and amino alcohol: To a solution of aldehyde (1.0 mmol), potassium iodide (0.2 mmol) and amino-alcohol (1.2 mmol) in 3 mL of CH₂Cl₂, a solution of 70% aqueous TBHP (3.0 mmol) was added dropwise over a period of 30 min and stirred at room temperature. The mixture was quenched with saturated aqueous Na₂S₂O₃ after 8h, washed with brine, extracted with ethyl acetate and dried over anhydrous Na₂SO₄. Removal of the solvent under vacuum afforded the crude product, which was further purified by column chromatography using hexane / ethyl acetate mixture and was analyzed by ¹H NMR, GC and GC-MS. Similar procedure was followed for the synthesis of chiral oxazolines from chiral amino alcohols and benzaldehyde and multi-scale synthesis of chiral 2-Oxazoline **2p**.

Products 3a [1], 3c [2], 3d [3], 3h [4], 3k [5], 3m [6], 3n [7], 3p [6], 3q [8], 3r [8] and 3t [9] have been described in the literature previously. Characterization data for all compounds are given below.

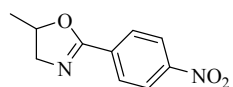
5-methyl-2-p-tolyl-4,5-dihydrooxazole (3a, Table 2, Entry 1) [1]



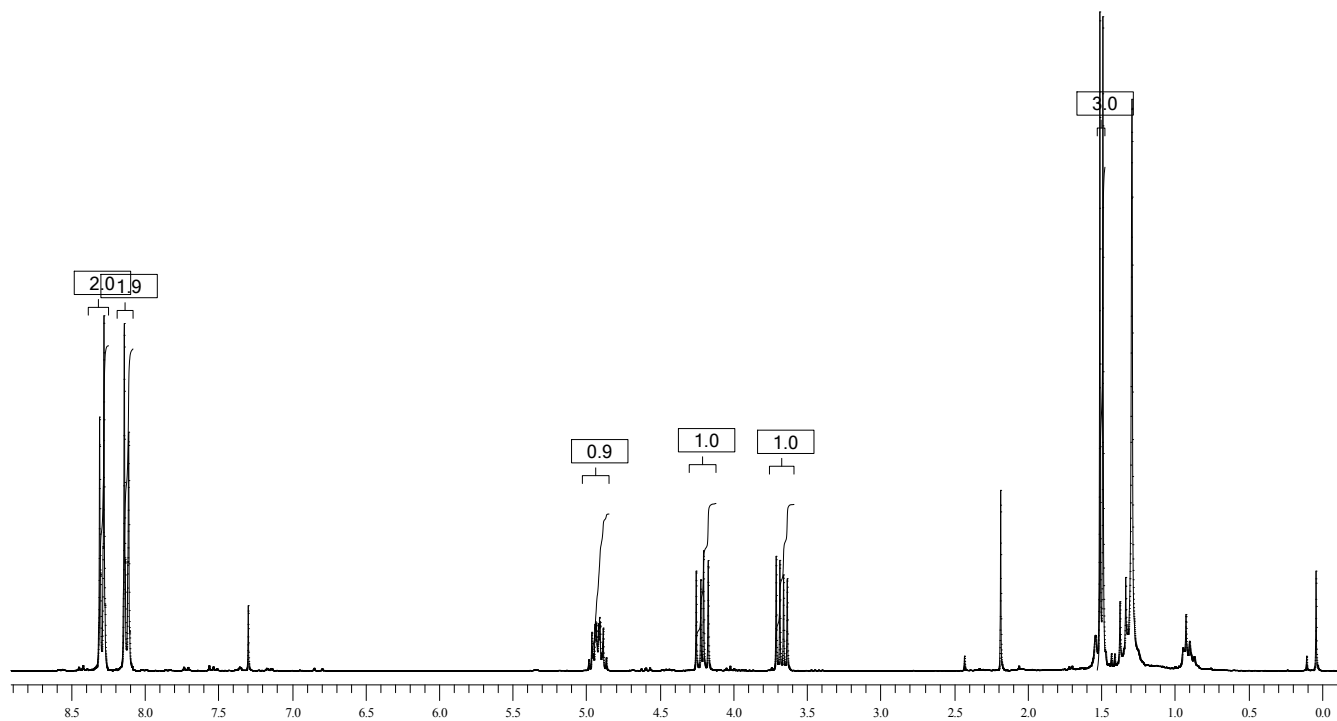
Isolated yield = 61%; $^1\text{H NMR}$ δ (300 MHz, CDCl_3) 7.80 (d, $J = 7.9$ Hz, Ar, 2H), 7.18 (d, $J = 7.9$ Hz, Ar, 2H), 4.77 – 4.87 (m, 1H), 4.11 (dd, $J = 9.3$ Hz, 9.2 Hz, 1H), 3.57 (dd, $J = 7.4$ Hz, 7.2 Hz), 2.4 (s, $-\text{CH}_3$, 3H), 1.43 (d, $J = 6.0$ Hz, $-\text{CH}_3$, 3H). **GC-MS** m/z 175.1 (M^+ peak), 130.9, 91.0, 65.0 .

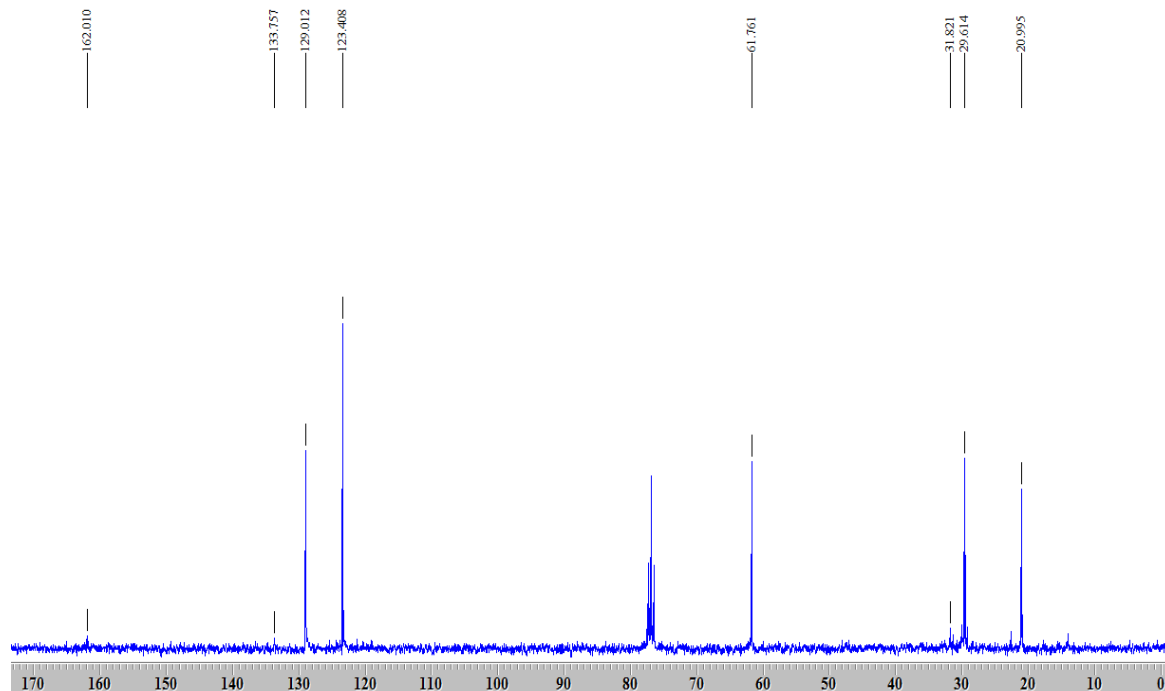


5-methyl-2-(4-nitrophenyl)-4,5-dihydrooxazole (3b, Table 2, Entry 2)

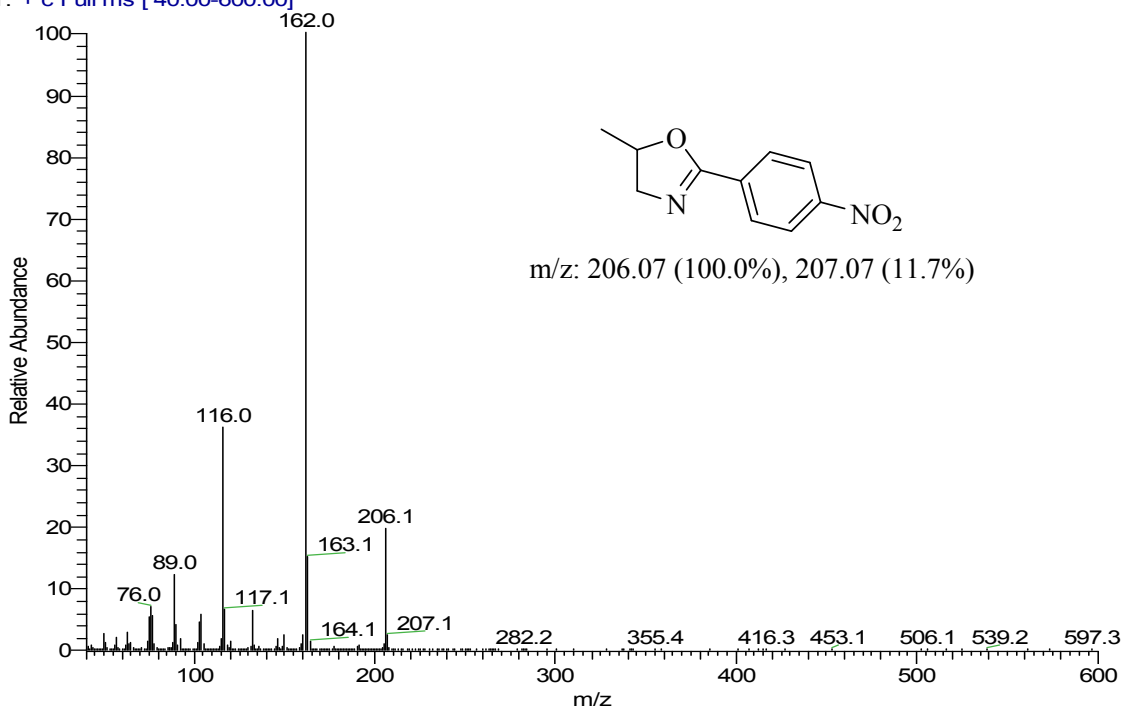


Isolated yield = 96%; yellow solid; mp 122-124 °C. IR (KBr) cm^{-1} : 2927, 2877, 1726, 1647, 1597, 1525, 1343, 1111, 957, 850, 705. $^1\text{H NMR}$ δ (300 MHz, CDCl_3) 8.29 (d, $J = 9.1$ Hz, Ar, 2H), 8.12 (d, $J = 8.9$ Hz, Ar, 2H), 4.86 – 4.98 (m, 1H), 4.21 (dd, $J = 9.4$ Hz, 9.2 Hz, 1H), 3.67 (dd, $J = 7.5$ Hz, 7.5 Hz), 1.50 (d, $J = 6.2$ Hz, $-\text{CH}_3$, 3H). $^{13}\text{C NMR}$ (75 MHz, CDCl_3): 162.0, 133.8, 129.0, 123.4, 61.8, 31.8, 29.6, 20.9. **GC-MS** m/z 206.1 (M^+ peak), 162.0, 117.1, 116.0, 89.0, 76.0 .

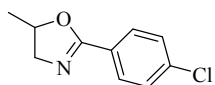




UMA-ASOX-133-5H #678 RT: 15.89 AV: 1 NL: 3.65E7
 T: + c Full ms [40.00-600.00]

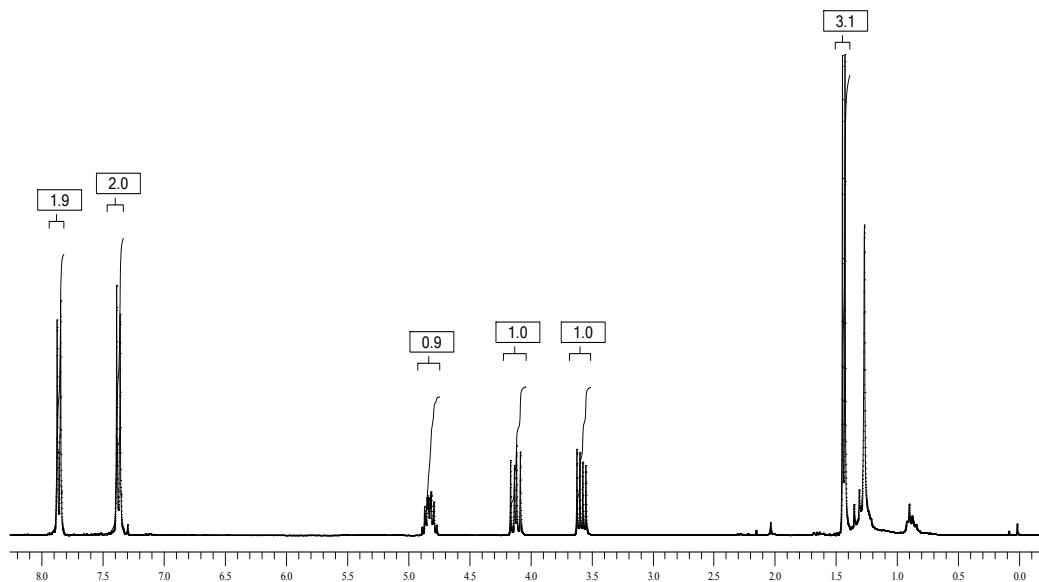


2-(4-chlorophenyl)-5-methyl-4,5-dihydrooxazole (3c, Table 2, Entry 3) [2]

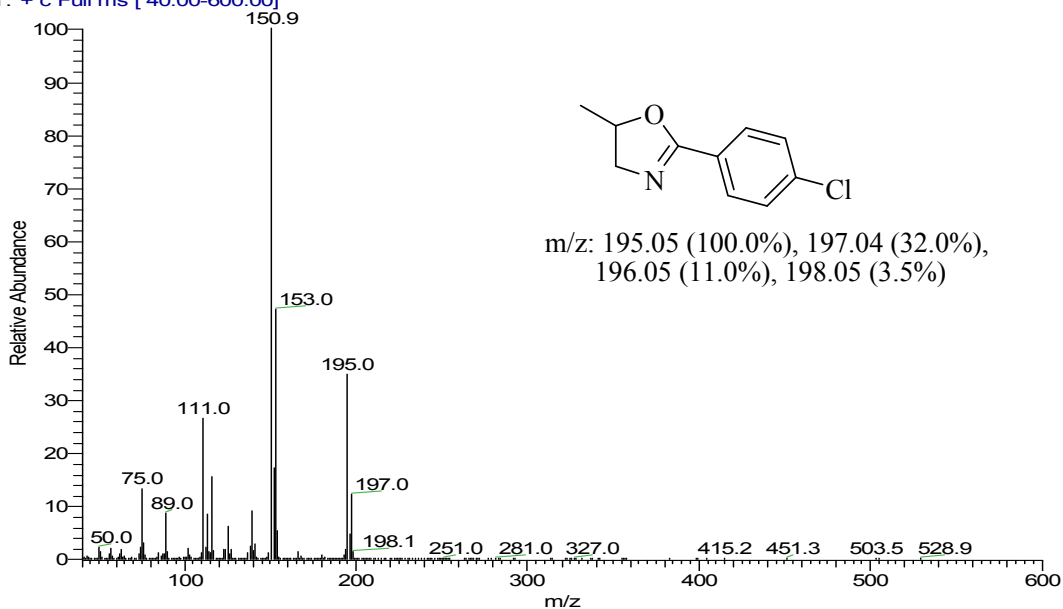


Isolated yield = 86%; $^1\text{H NMR}$ δ (300 MHz, CDCl_3) 7.86 (d, $J = 8.5$ Hz, Ar, 2H), 7.37 (d, $J = 8.5$ Hz, Ar, 2H), 4.77 – 4.89 (m, 1H), 4.12 (dd, $J = 9.4$ Hz, 9.4 Hz, 1H), 3.58 (dd, $J = 7.3$ Hz, 7.5 Hz), 1.43 (d, $J = 6.2$ Hz, $-\text{CH}_3$, 3H).

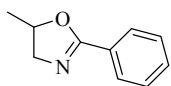
GC-MS m/z 195.0, 153.0, 150.9, 111.0, 89.0, 75.0 .



UMA-ASOX-135P #547 RT: 13.40 AV: 1 NL: 4.33E7
T: + c Full ms [40.00-600.00]

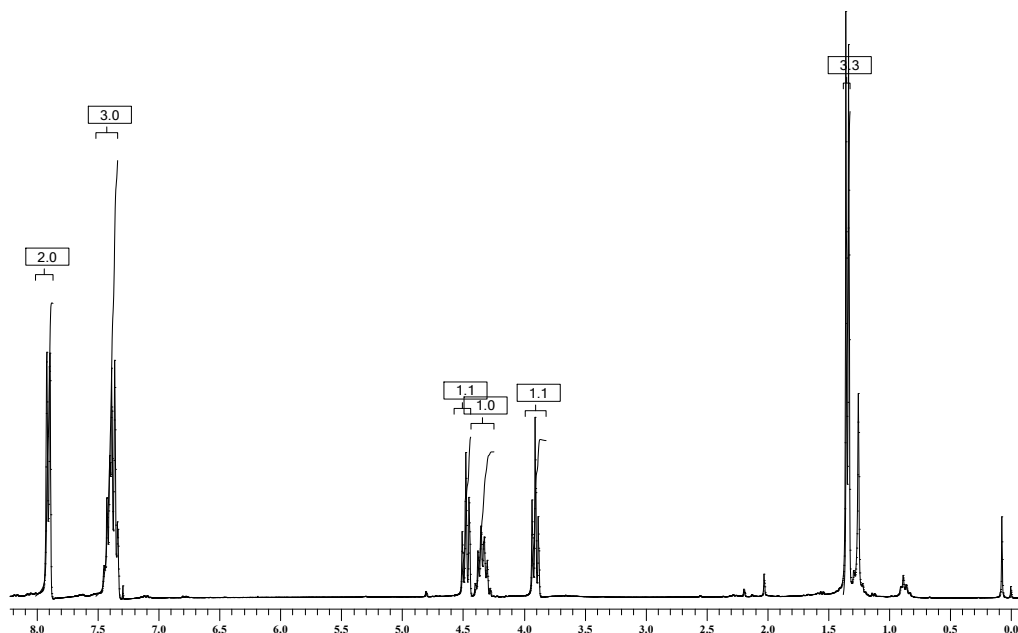


5-methyl-2-phenyl-4,5-dihydrooxazole (3d, Table 2, Entry 4) [3]

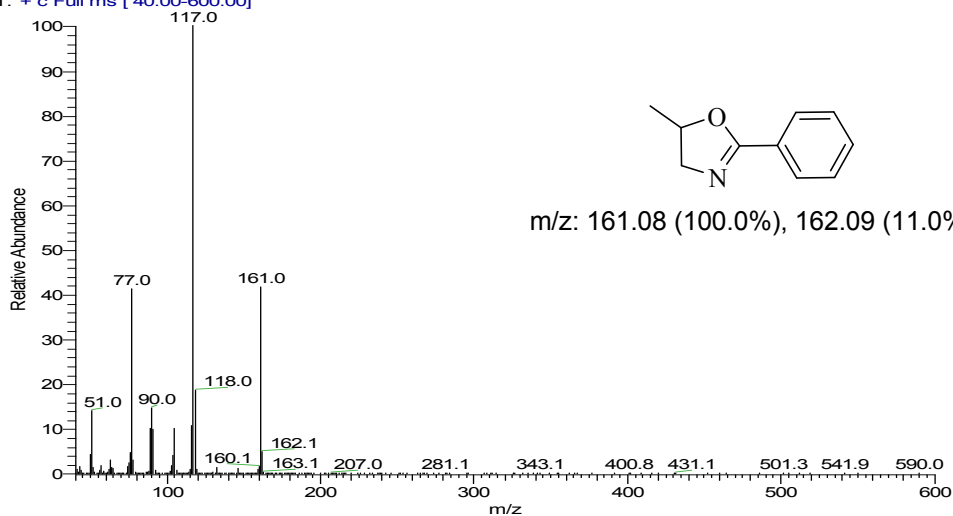


Isolated yield = 83%; $^1\text{H NMR}$ δ (300 MHz, CDCl_3) 7.86 (d, $J = 8.5$ Hz, Ar, 2H), 7.54 (d, $J = 8.2$ Hz, 1H), 7.37 (d, $J = 8.5$ Hz, Ar, 2H), 4.70 – 4.85 (m, 1H), 4.05 (dd, $J = 9.3$ Hz, 9.3 Hz, 1H), 3.52 (dd, $J = 7.4$ Hz, 7.4 Hz), 1.37

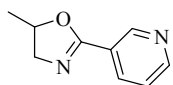
(d, $J = 6.1$ Hz, $-\text{CH}_3$, 3H). **GC-MS** m/z 161.0, 117.0, 90.0, 77.0, 51.0 .



UMA-ASOX-131-8H #427 RT: 11.11 AV: 1 NL: 4.24E7
T: + c Full ms [40.00-600.00]



5-methyl-2-(pyridin-3-yl)-4,5-dihydrooxazole (3e, Table 2, Entry 5)



Isolated yield = 61%; yellow liquid; IR (KBr) cm^{-1} : 2924, 2854, 1730,

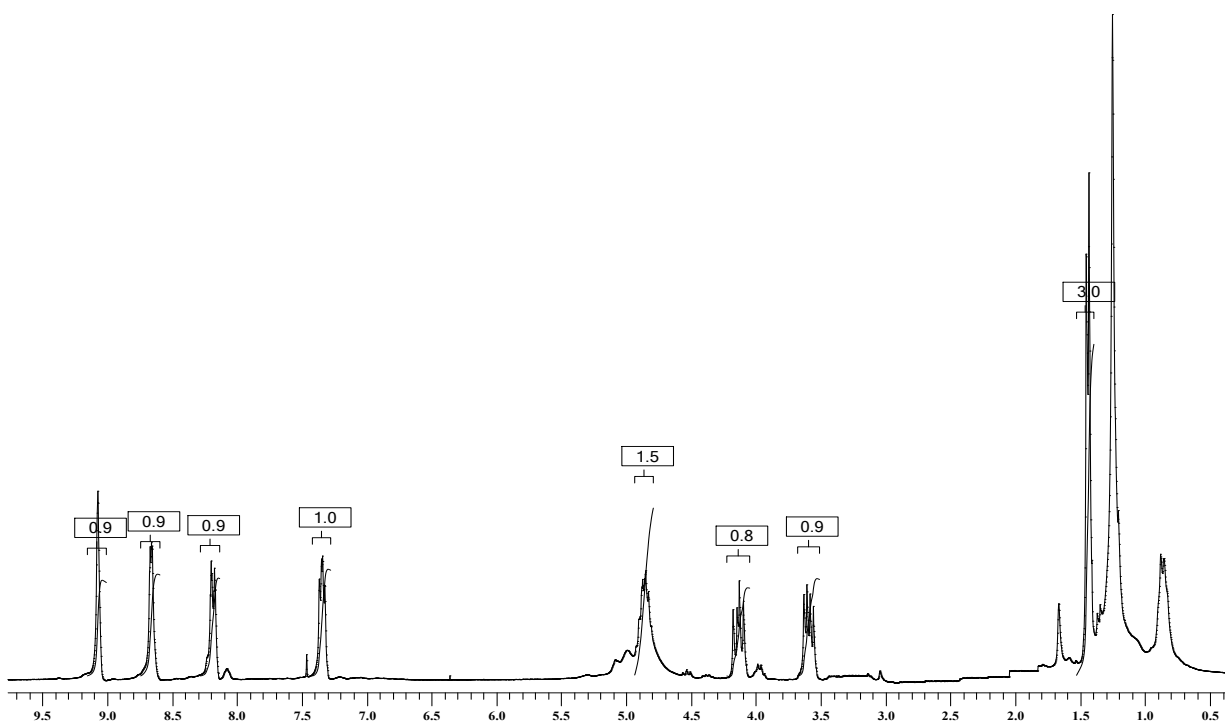
1652, 1458, 1377, 1278, 1114, 965, 811. ^1H NMR δ (300 MHz, CDCl_3) 9.1

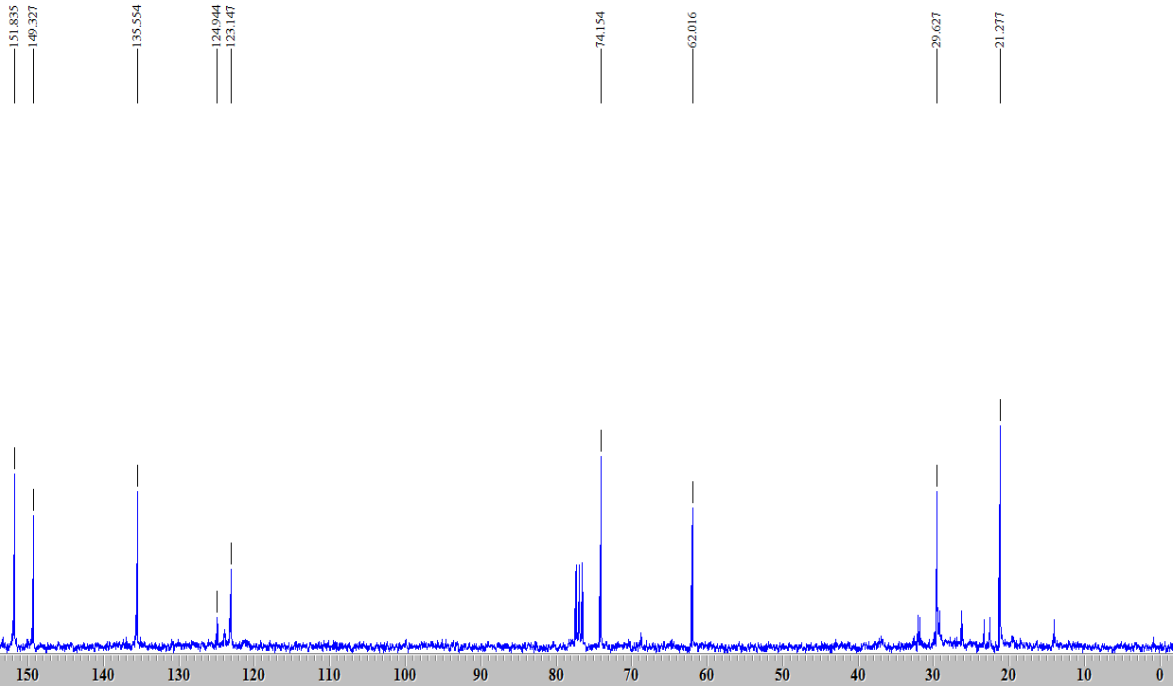
(s, Ar, 1H), 8.69 (s, Ar, 1H), 8.27 (d, $J = 8.1$ Hz, Ar, 1H), 7.32 (d, $J = 8.1$, Ar, 1H), 4.90 – 4.72

(m, 1H), 4.11 (dd, $J = 9.4$ Hz, 7.4 Hz, 1H), 3.58 (dd, $J = 7.5$ Hz, 9.5 Hz), 1.39 (d, $J = 6.3$ Hz, -

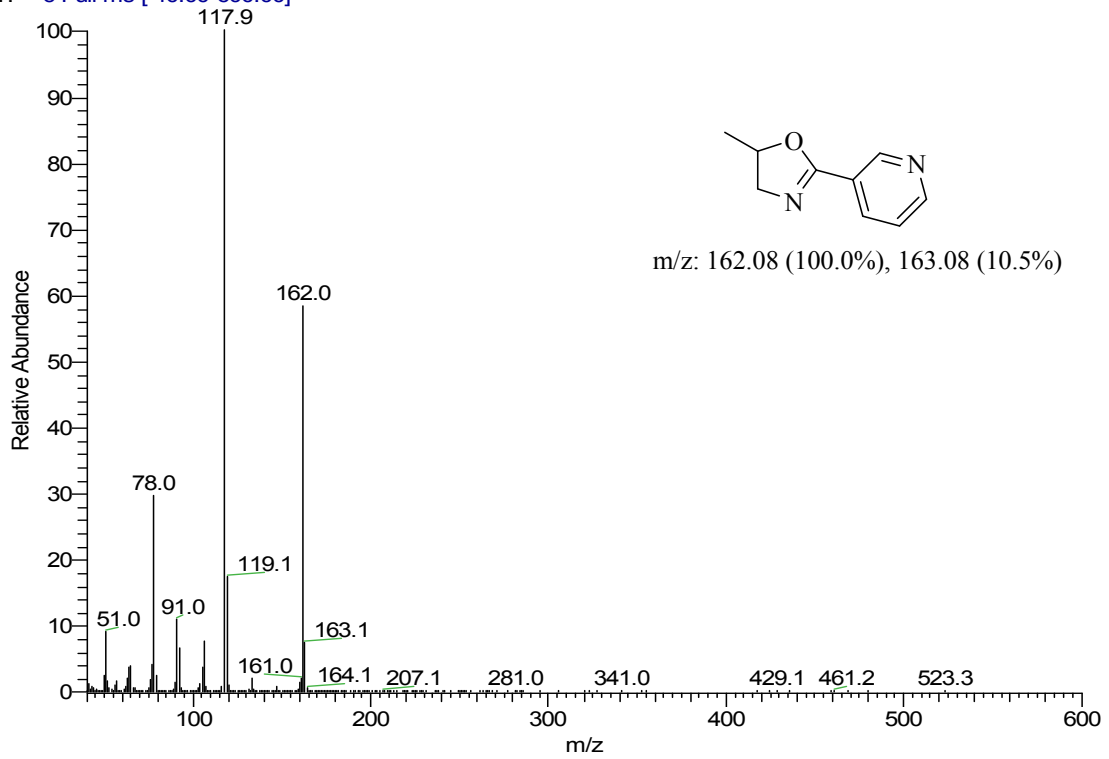
CH_3 , 3H). ^{13}C NMR (75 MHz, CDCl_3): 151.8, 149.3, 135.6, 124.9, 123.1, 74.2, 62.0, 29.6,

21.3. GC-MS m/z 162.0, 117.9, 91.0, 78.0, 51.0 .

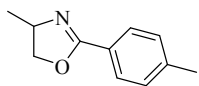




uma-asox-139-10h #467 RT: 11.87 AV: 1 NL: 4.25E7
 T: + c Full ms [40.00-600.00]



4-methyl-2-p-tolyl-4,5-dihydrooxazole (3f, Table 2, Entry 6)



Isolated yield = 77%; yellow liquid; IR (KBr) cm^{-1} : 2956, 2923, 2855, 1724,

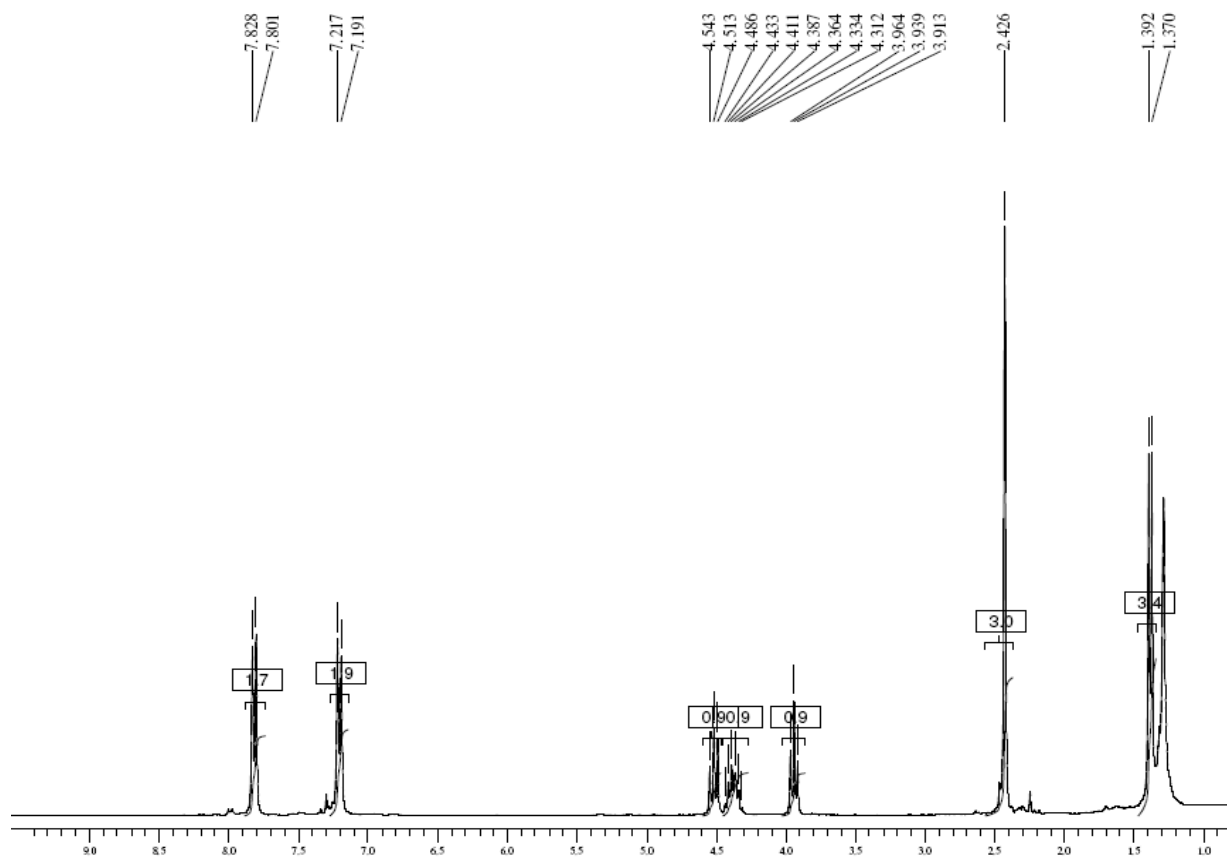
1647, 1457, 1350, 1264, 1178, 1108, 1062, 1021, 972, 829, 728. ^1H NMR

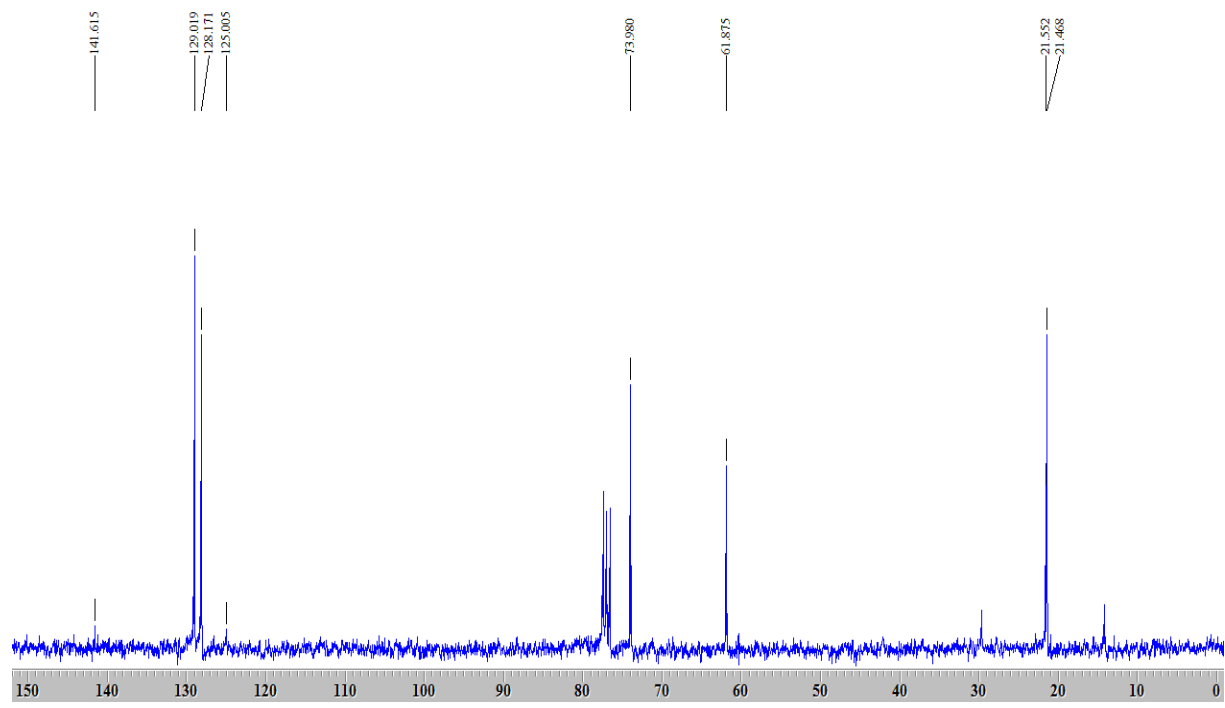
δ (300 MHz, CDCl_3) 7.81 (d, $J = 7.9$ Hz, Ar, 2H), 7.20 (d, $J = 7.9$ Hz, Ar, 2H), 4.51 (t, $J = 9.0$ Hz,

8.1 Hz, 1H), 4.31- 4.43 (m, 1H), 3.94 (t, $J = 7.5$ Hz, 7.7 Hz, 1H), 2.42(s, $-\text{CH}_3$, 3H), 1.38 (d, $J =$

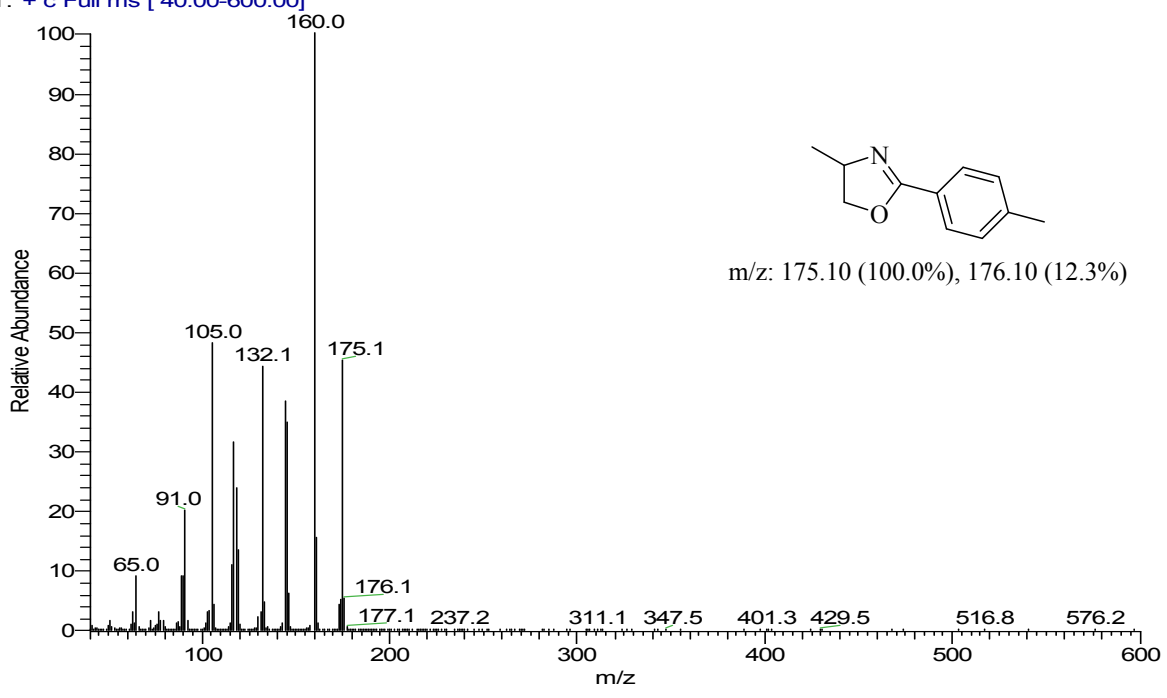
6.6 $-\text{CH}_3$, 3H). ^{13}C NMR (75 MHz, CDCl_3): 151.8, 149.3, 135.6, 124.9, 123.1, 74.2, 62.0, 29.6,

21.3. GC-MS m/z 175.1 (M^+ peak), 160.0, 132.1, 105.0, 91.0, 65.0 .



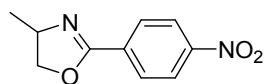


UMA-ASOX-124-1D #501 RT: 12.52 AV: 1 NL: 3.79E7
 T: + c Full ms [40.00-600.00]



4-methyl-2-(4-nitrophenyl)-4,5-dihydrooxazole (3g, Table 2, Entry 7)

Isolated yield = 63%; pale yellow solid; mp 89-91 °C; IR (KBr) cm^{-1} : 2923,



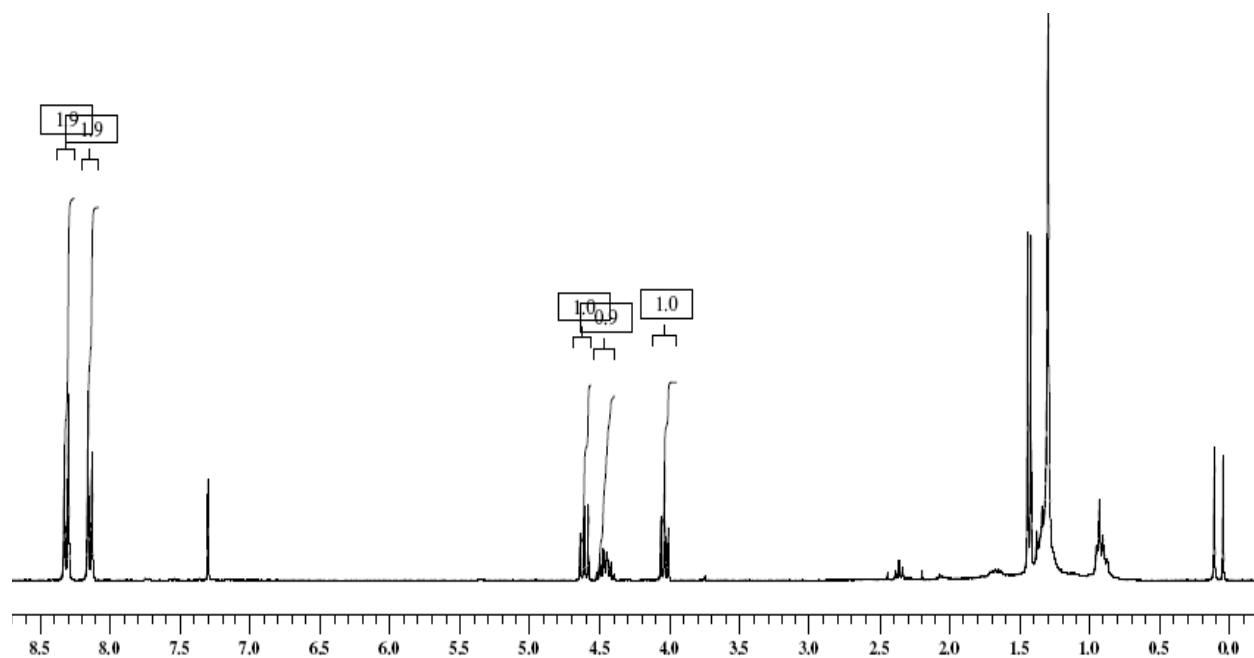
2855, 1645, 1522, 1344, 1069, 963, 856, 706. ^1H NMR δ (300 MHz, CDCl_3)

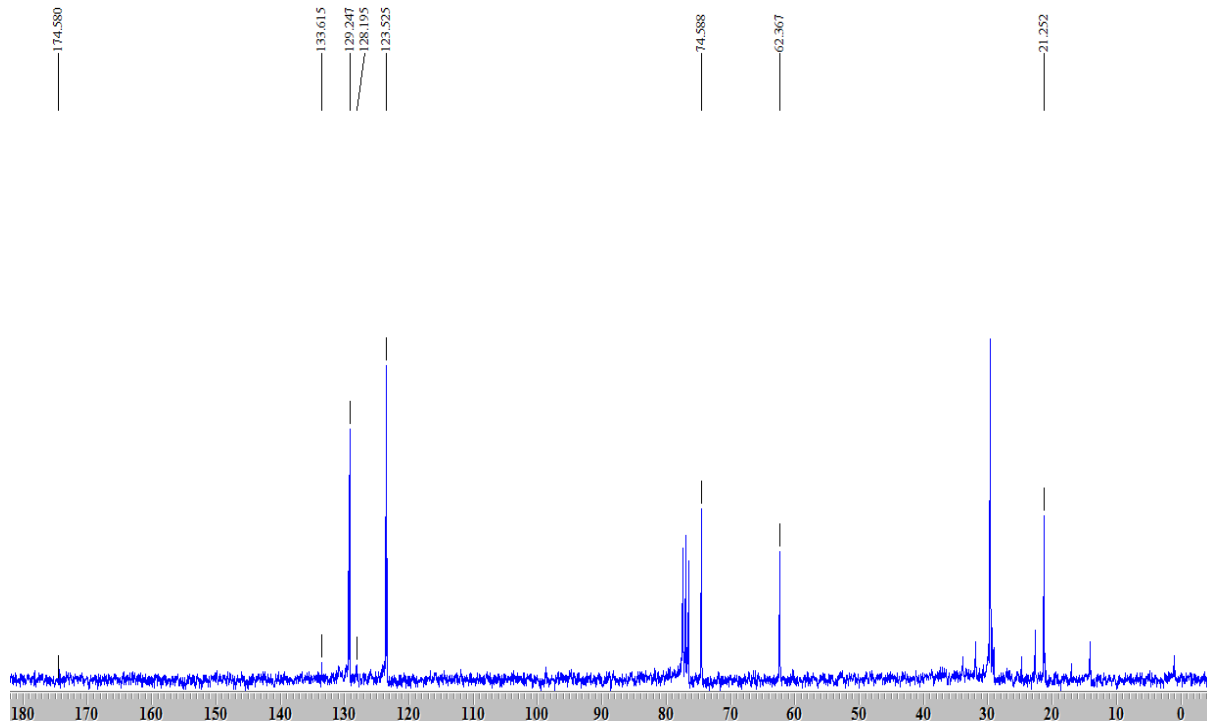
8.21 (d, $J = 8.8$ Hz, Ar, 2H), 8.10 (d, $J = 8.1$ Hz, Ar, 2H), 4.53 (t, $J = 8.1$ Hz, 1H), 4.31- 4.37 (m,

1H), 3.94 (t, $J = 7.8$ Hz, 1H), 1.42 (d, $J = 6.7$, $-\text{CH}_3$, 3H). ^{13}C NMR (75 MHz, CDCl_3): 162.6,

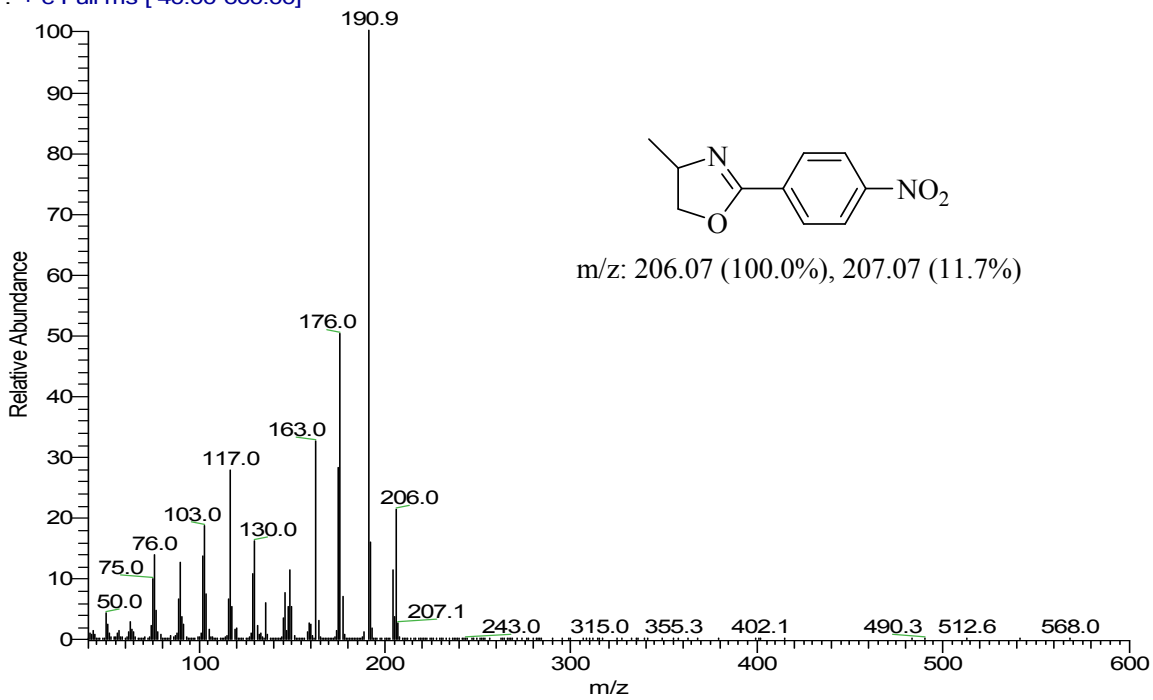
137.4, 129.5, 128.6, 126.3, 74.2, 62.0, 21.4. GC-MS m/z 206.0 (M^+ peak), 190.9, 176.0, 163.0,

130.0, 117.0, 103.0, 76.0.

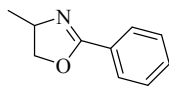




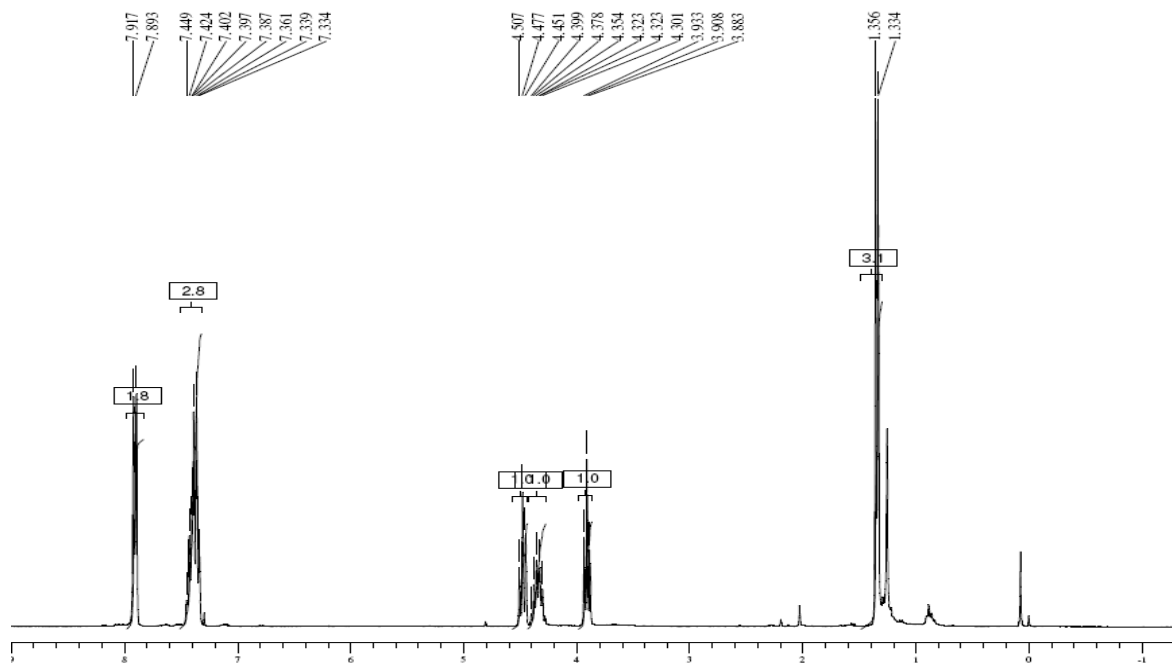
UMA-ASOX-121-6H #670 RT: 15.74 AV: 1 NL: 3.89E7
 T: + c Full ms [40.00-600.00]



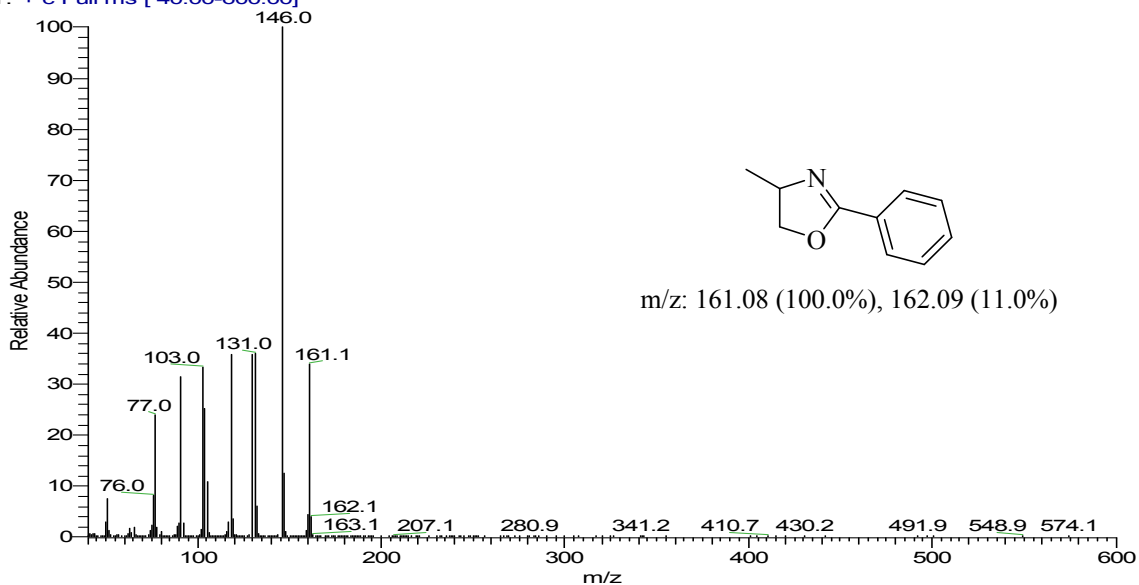
4-methyl-2-phenyl-4,5-dihydrooxazole (3h, table 2, Entry 8) [4]



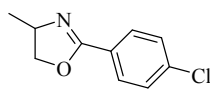
Isolated yield = 78%; ¹H NMR δ(300 MHz, CDCl₃) 7.90 (d, J = 7.5 Hz, Ar, 2H), 7.33 – 7.44 (m, Ar, 3H), 4.47 (t, J = 9.0 Hz, 7.5 Hz, 1H), 4.30- 4.40 (m, 1H), 3.91 (t, J = 7.5 Hz, 7.5 Hz, 1H), 1.34 (d, J = 6.8 -CH₃, 3H). GC-MS m/z 161.1 (M⁺ peak), 146.0, 131.0, 103.0, 77.0.



UMA-ASOX-126-8H #416 RT: 10.90 AV: 1 NL: 3.19E7
T: + c Full ms [40.00-600.00]



2-(4-chlorophenyl)-4-methyl-4,5-dihydrooxazole (3i, Table 2, Entry 9)

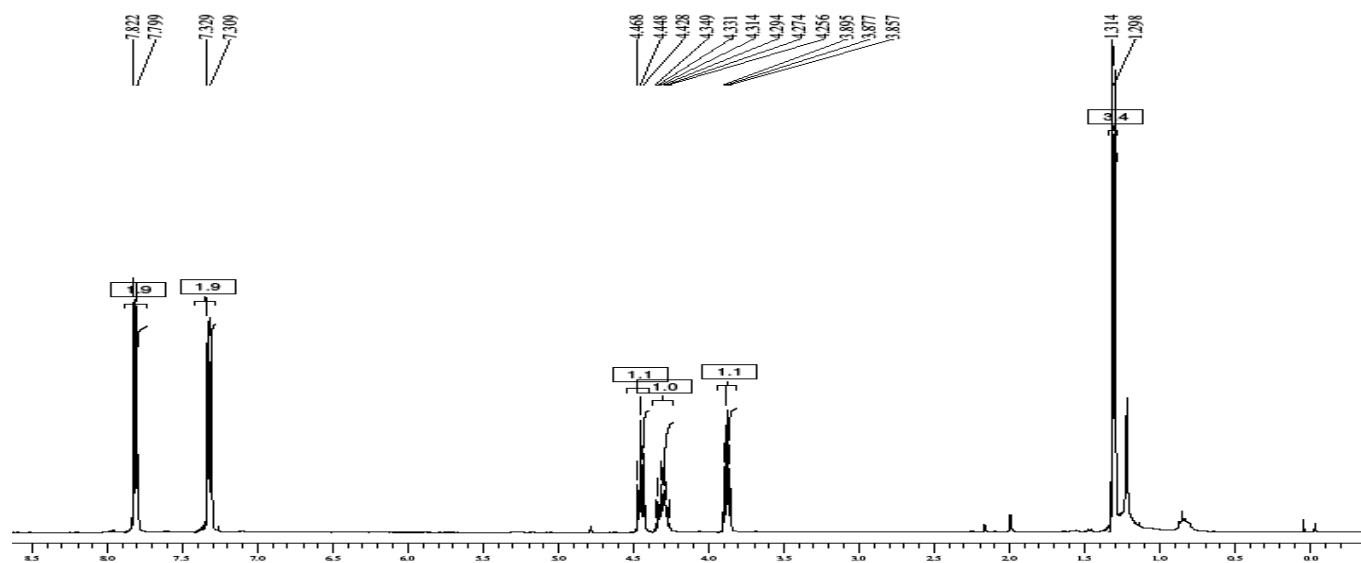


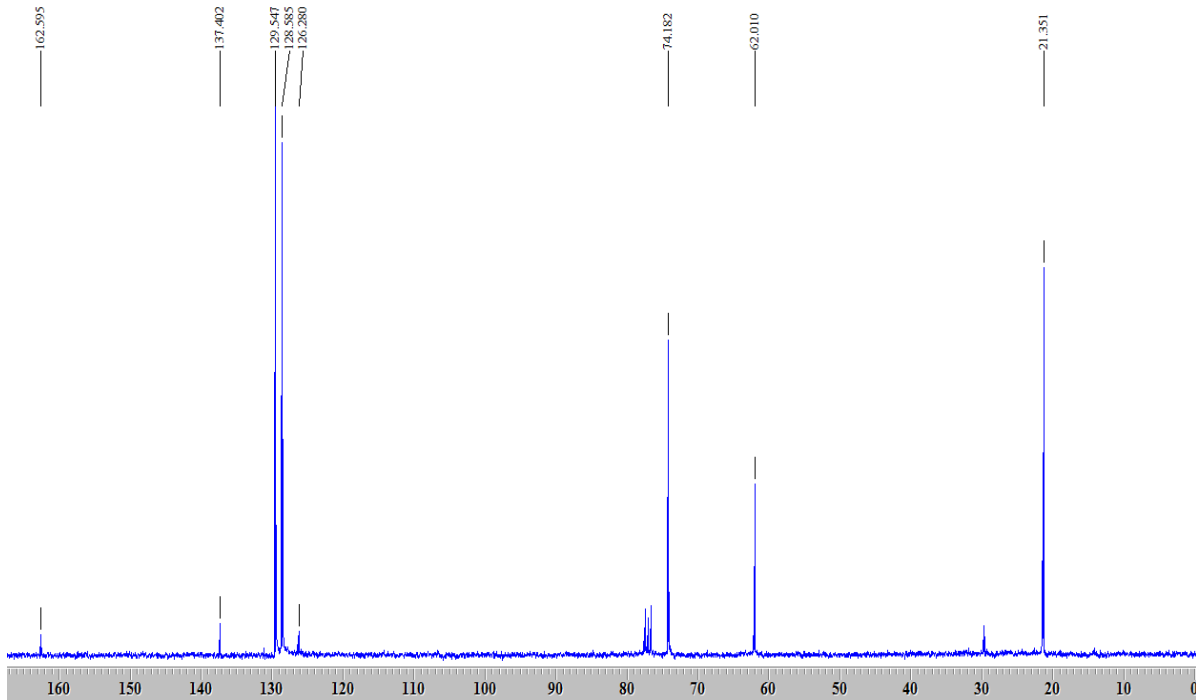
Isolated yield = 65%; yellow liquid; IR (KBr) cm^{-1} : 3064, 3030, 2966, 2924,

1720, 1648, 1491, 1451, 1356, 1269, 1170, 1060, 1021, 969, 841, 701. ^1H

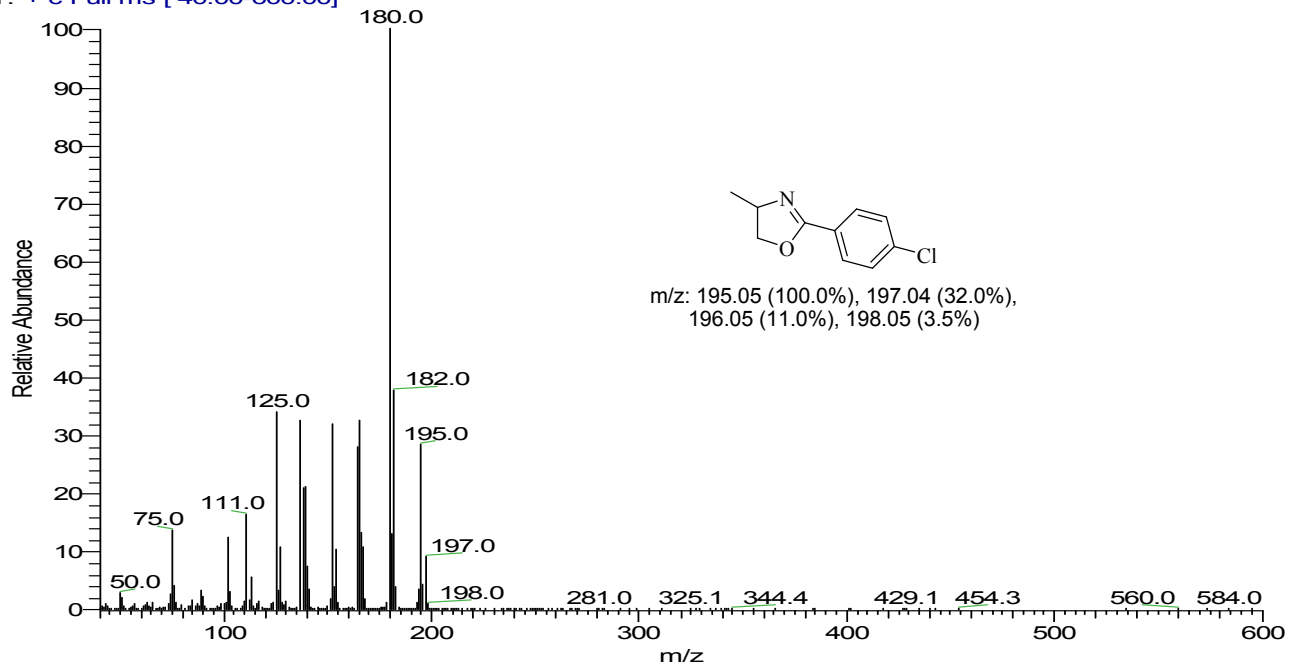
NMR δ (300 MHz, CDCl_3) 7.81 (d, $J = 8.8$ Hz, Ar, 2H), 7.32 (d, $J = 8.1$ Hz, Ar, 2H), 4.45 (t, $J = 8.1$ Hz, 1H), 4.26- 4.35 (m, 1H), 3.88 (t, $J = 7.3$ Hz, 8.1 Hz, 1H), 2.42(s, $-\text{CH}_3$, 3H), 1.38 (d, $J = 6.6$ Hz, $-\text{CH}_3$, 3H). ^{13}C NMR (75 MHz, CDCl_3): 162.6, 137.4, 129.5, 128.6, 126.3, 74.2, 62.0, 21.4.

GC-MS m/z 195.0 (M^+ peak), 180.0, 125.0, 111.0, 75.0 .

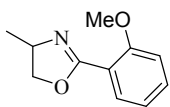




UMA-ASOX-125-6H #533 RT: 13.13 AV: 1 NL: 2.70E7
 T: + c Full ms [40.00-600.00]



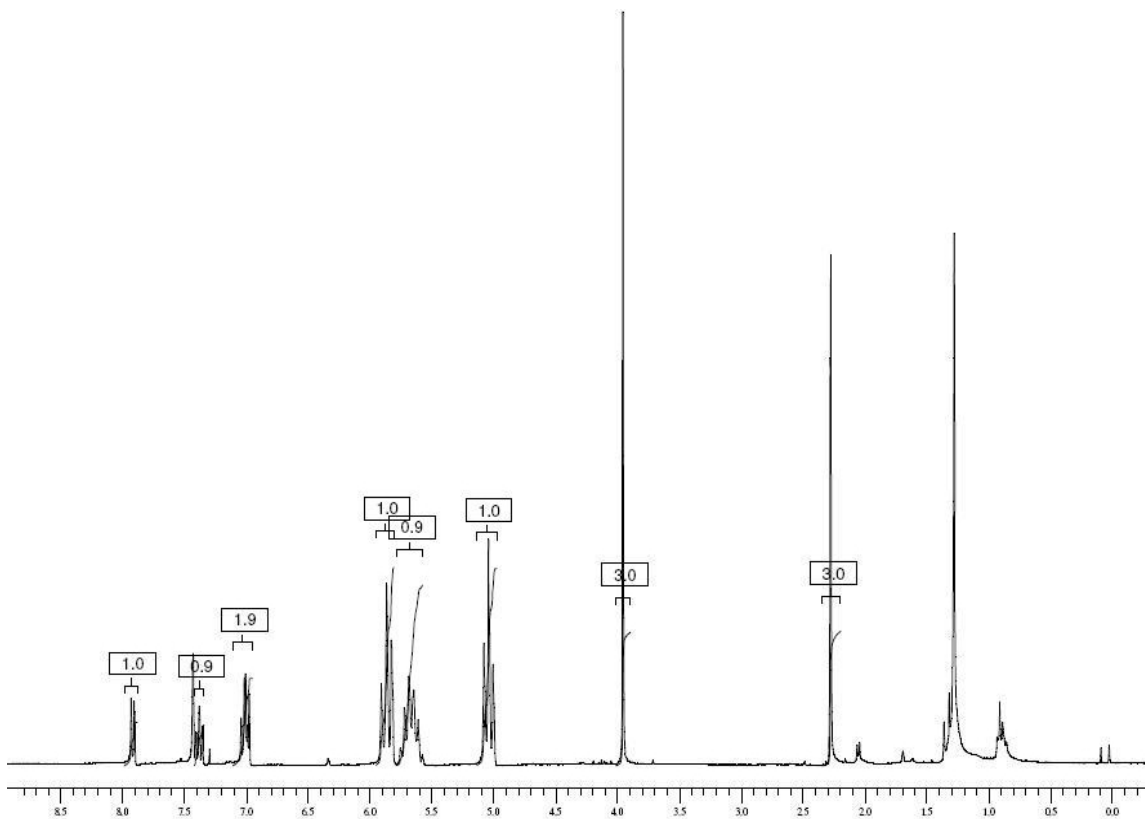
4-methyl-2-p-tolyl-4,5-dihydrooxazole (3j, Table 2, Entry 10)

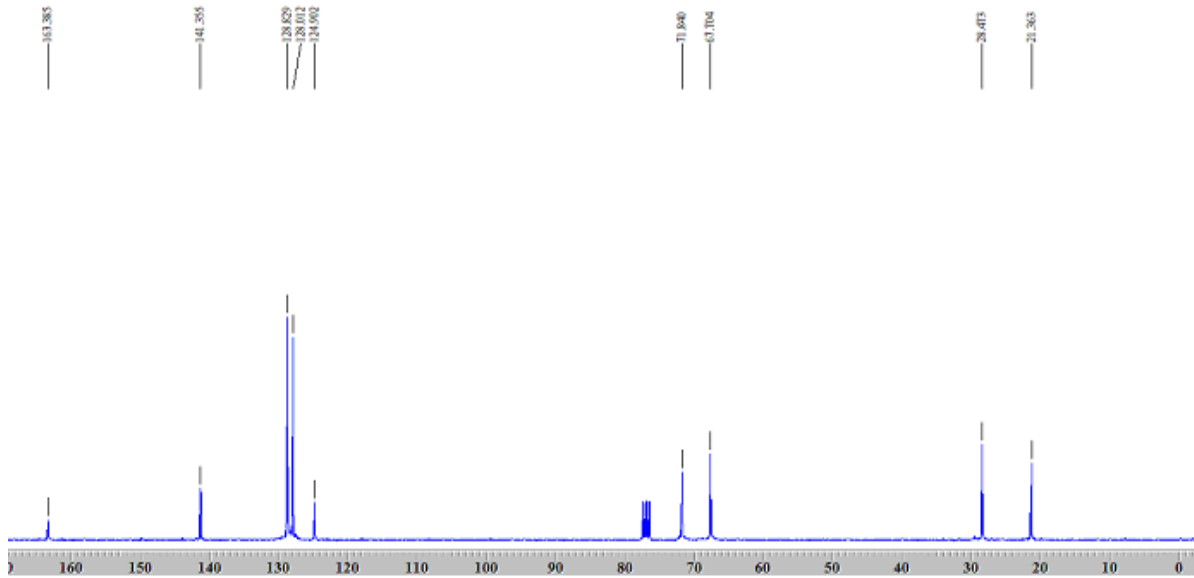


Isolated yield = 83%; pale yellow solid; IR (KBr) cm^{-1} : 2928, 2846, 1719,

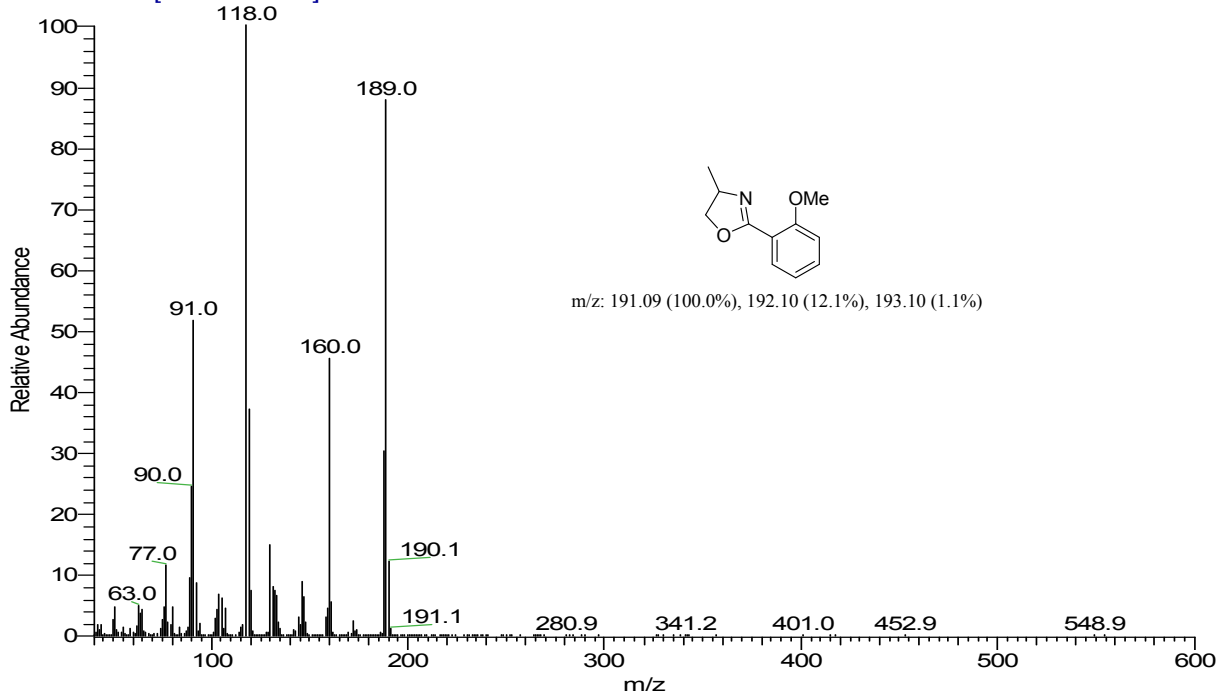
1598, 1466, 1380, 1253, 1175, 1102, 1023, 936, 752; ^1H NMR δ (300

MHz, CDCl_3) 7.95 (d, $J = 8.1$ Hz, Ar, 1H), 7.30-7.50 (m, Ar, 1H), 6.97-7.04 (m, Ar, 1H), 5.90 (t, $J = 9.0$ Hz, 8.1 Hz, 1H), 5.60-5.80 (m, 1H), 5.15 (t, $J = 9.0$ Hz, 8.1 Hz, 1H), 3.95 (s, $-\text{CH}_3$, 3H), 2.27 (d, $J = 5.3$, $-\text{CH}_3$, 3H). ^{13}C NMR (75 MHz, CDCl_3): 163.4, 141.4, 128.8, 128.0, 124.9, 71.8, 67.7, 28.5, 21.4. **GC-MS** m/z 190.1 (M^+ peak), 189.0, 160.0, 118.0, 91.0, 77.0, 63.0. .

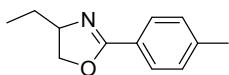




UMA-ASOX-127-8H #560 RT: 13.64 AV: 1 NL: 2.59E7
 T: + c Full ms [40.00-600.00]



4-ethyl-2-p-tolyl-4,5-dihydrooxazole (3k, Table 2, Entry 11)



Isolated yield = 72%; yellow liquid; IR (KBr) cm^{-1} : 2963, 2897, 1648,

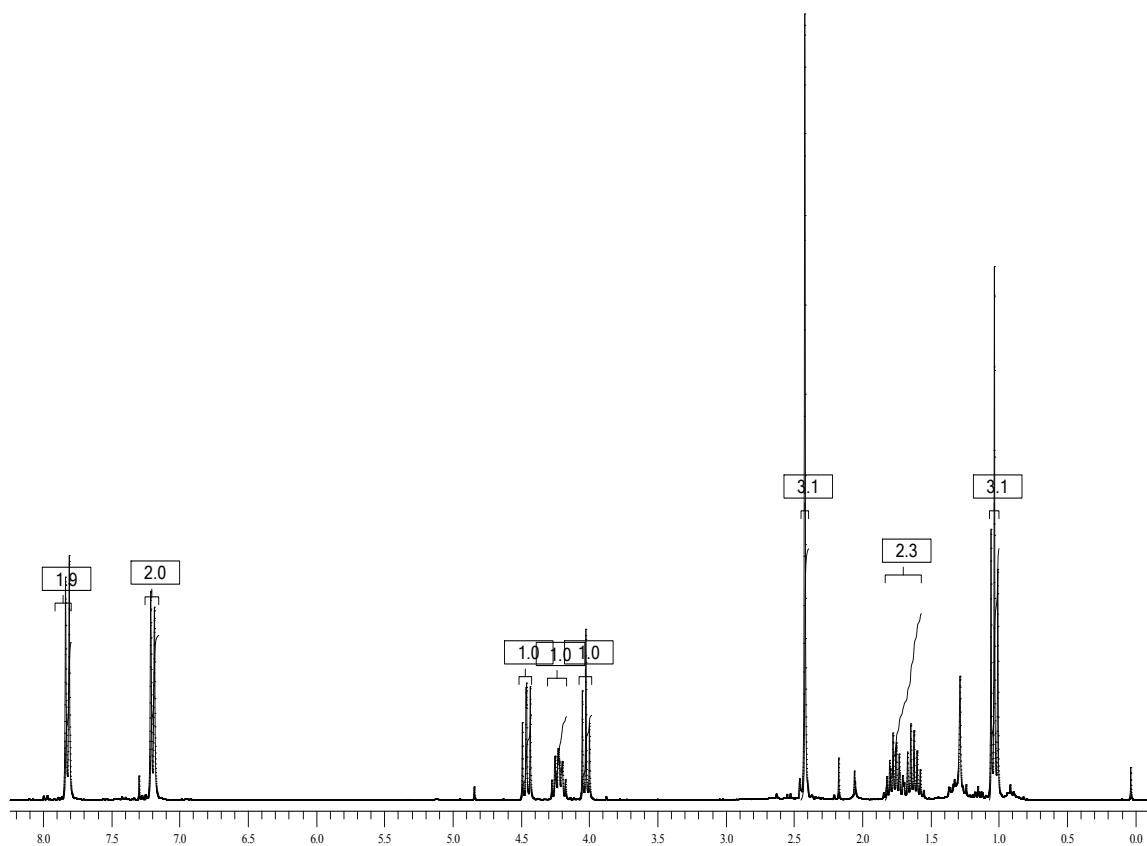
1512, 1461, 1355, 1178, 1065, 963, 828, 728. ^1H NMR δ (300 MHz,

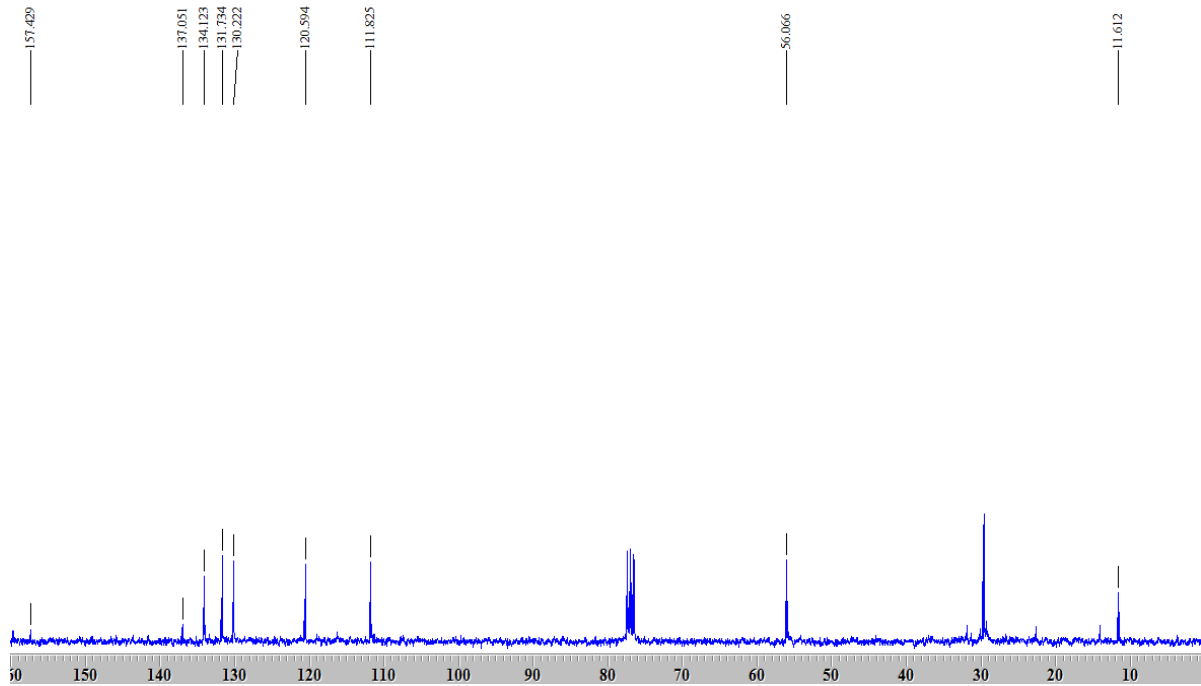
CDCl_3) 7.82 (d, $J = 8.1$ Hz, Ar, 2H), 7.20 (d, $J = 7.9$ Hz, Ar, 2H), 4.46 (t, $J = 7.9$ Hz, 1H), 4.17-

4.43 (m, 1H), 4.02 (t, $J = 7.9$ Hz, 1H), 2.42 (s, $-\text{CH}_3$, 3H), 1.57 – 1.82 (m, 2H), 1.3 (t, $J = 7.4$

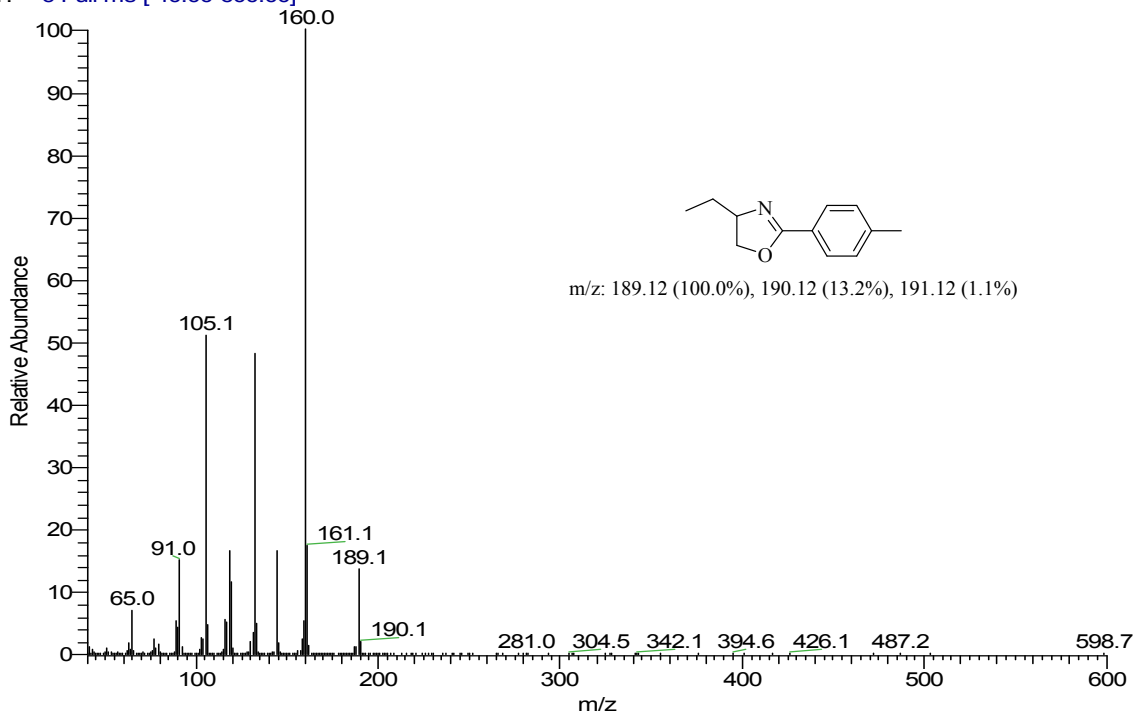
Hz, 7.4 Hz, 3H). ^{13}C NMR (75 MHz, CDCl_3): 157.4, 137.1, 134.1, 131.7, 130.2, 120.6, 111.8,

56.1, 11.6. **GC-MS** m/z 189.1 (M^+ peak), 160.0, 105.1, 91.0, 65.0 .

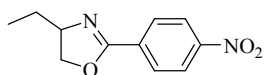




UMA-ASOX-136-10H #563 RT: 13.70 AV: 1 NL: 3.20E7
 T: + c Full ms [40.00-600.00]

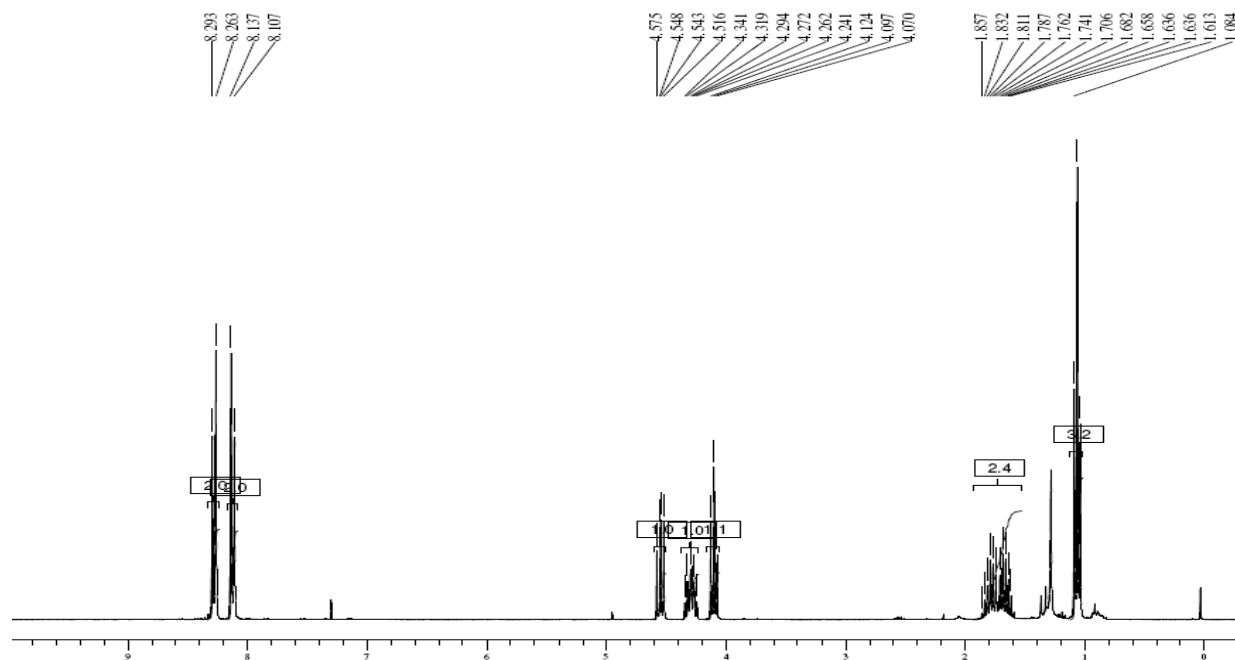


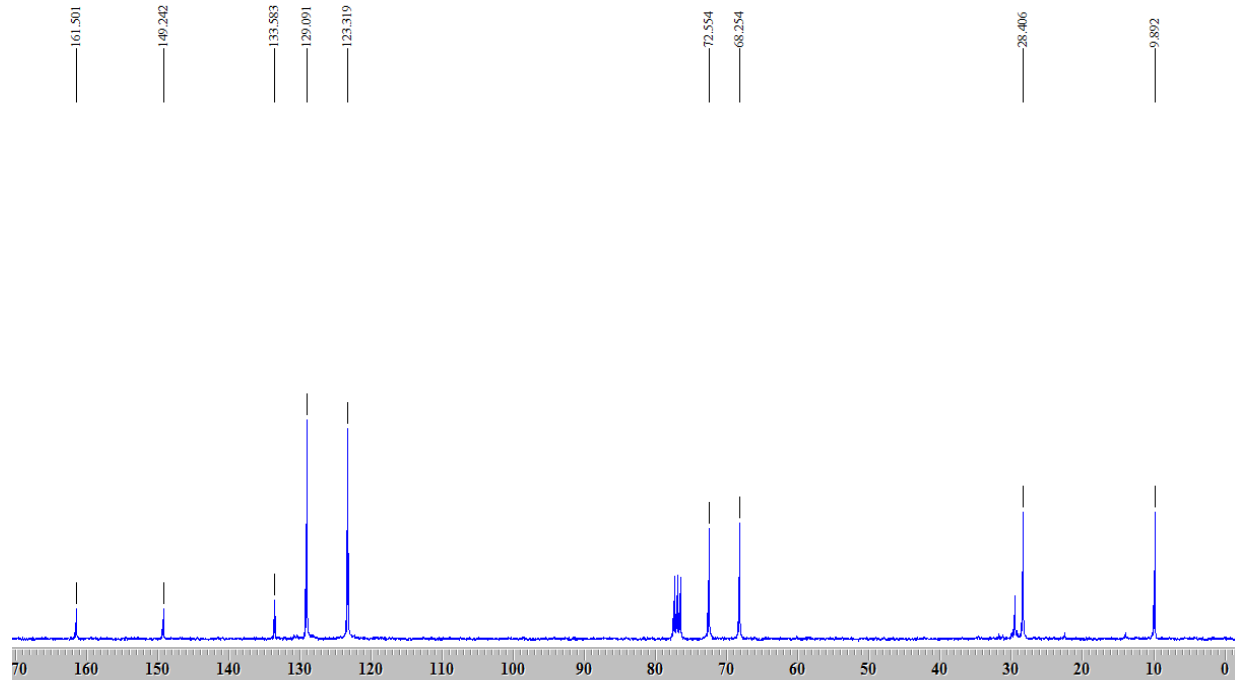
4-ethyl-2-(4-nitrophenyl)-4,5-dihydrooxazole (3I, Table 2, Entry 12) [6]



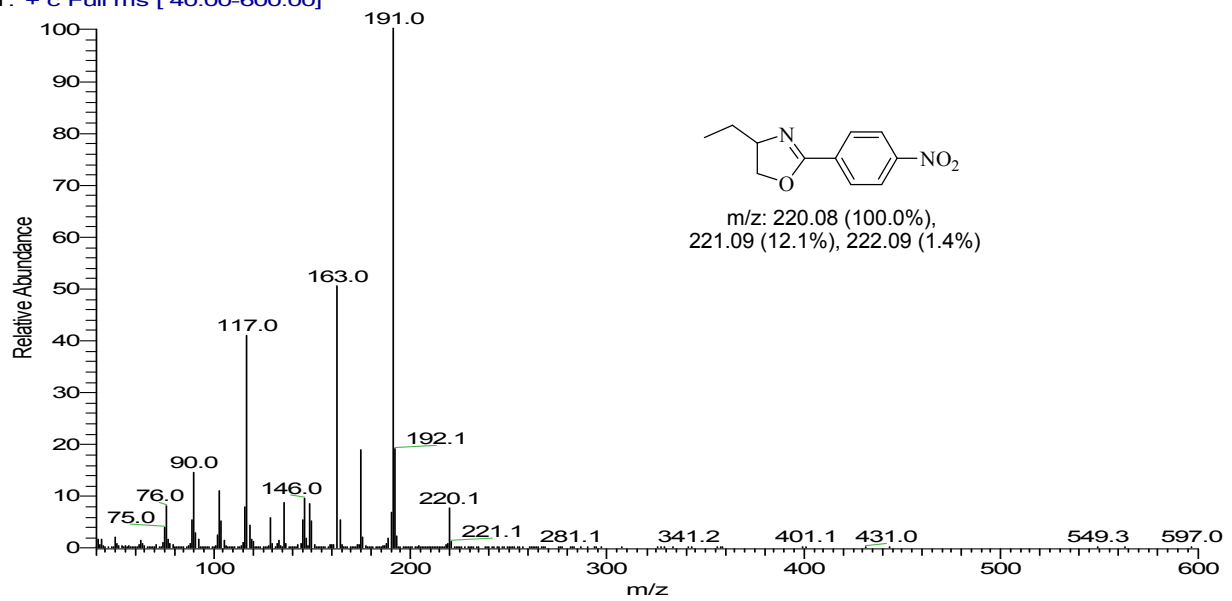
Isolated yield = 81%; $^1\text{H NMR } \delta(300 \text{ MHz, CDCl}_3)$ 8.28 (d, $J = 8.8 \text{ Hz}$, Ar, 2H), 8.12 (d, $J = 8.8 \text{ Hz}$, Ar, 2H), 4.54 (t, $J = 8.1 \text{ Hz}$, 1H), 4.24- 4.34

(m, 1H), 4.10 (t, $J = 7.9 \text{ Hz}$, 1H), 1.61 – 1.86 (m, 2H), 1.06 (d, $J = 7.4 \text{ Hz}$, 3H). **GC-MS** m/z 220 (M^+ peak), 191.0, 163.0, 146.0, 117.0, 90.0, 76.0 .

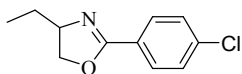




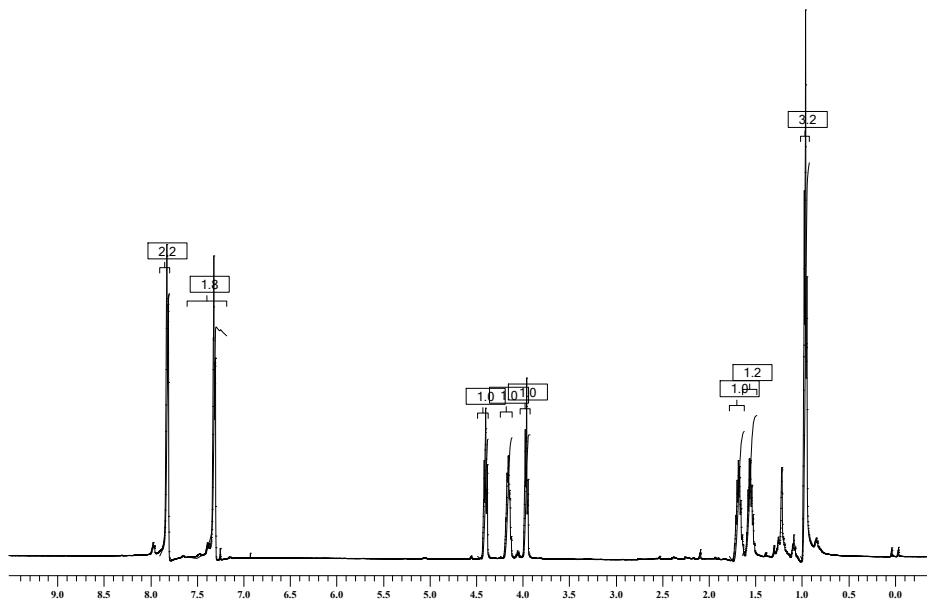
UMA-ASOX-132-5H #731 RT: 16.90 AV: 1 NL: 3.51E7
 T: + c Full ms [40.00-600.00]



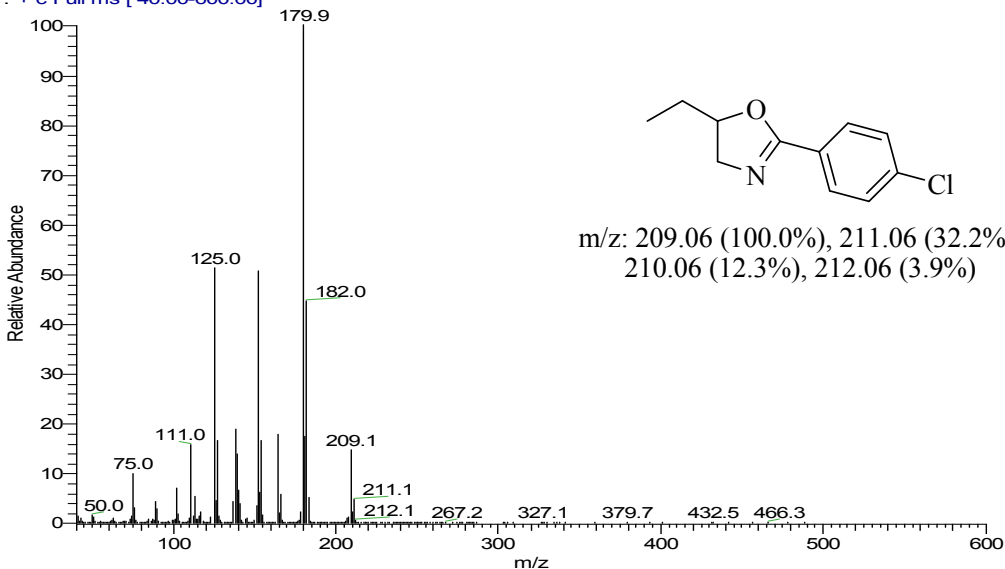
2-(4-chlorophenyl)-4-ethyl-4,5-dihydrooxazole (3m, Table 2, Entry 13) [7]



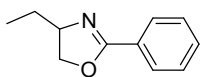
Isolated yield =83%; $^1\text{H NMR}$ δ (300 MHz, CDCl_3) 7.80 (d, $J = 8.6$ Hz, Ar, 2H), 7.32 (d, $J = 8.6$ Hz, Ar, 2H), 4.46 (t, $J = 8.1$ Hz, 1H), 4.21- 4.30 (m, 1H), 3.96 (t, $J = 7.9$ Hz, 1H), 1.72 – 1.80 (m, 2H), 1.04 (d, $J = 7.7$ Hz, 7.6 Hz, 3H). **GC-MS** m/z 209.1 (M^+ peak), 182.0,179.9, 125.0, 111.0, 75.0.



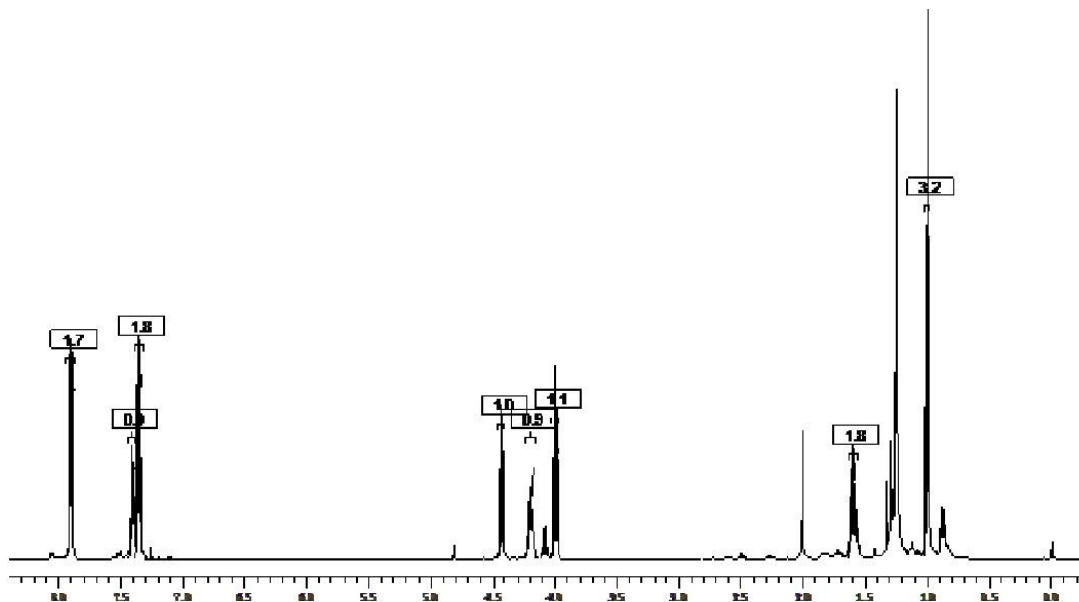
UMA-ASOX-140-1D #599 RT: 14.39 AV: 1 NL: 4.09E7
T: + c Full ms [40.00-600.00]



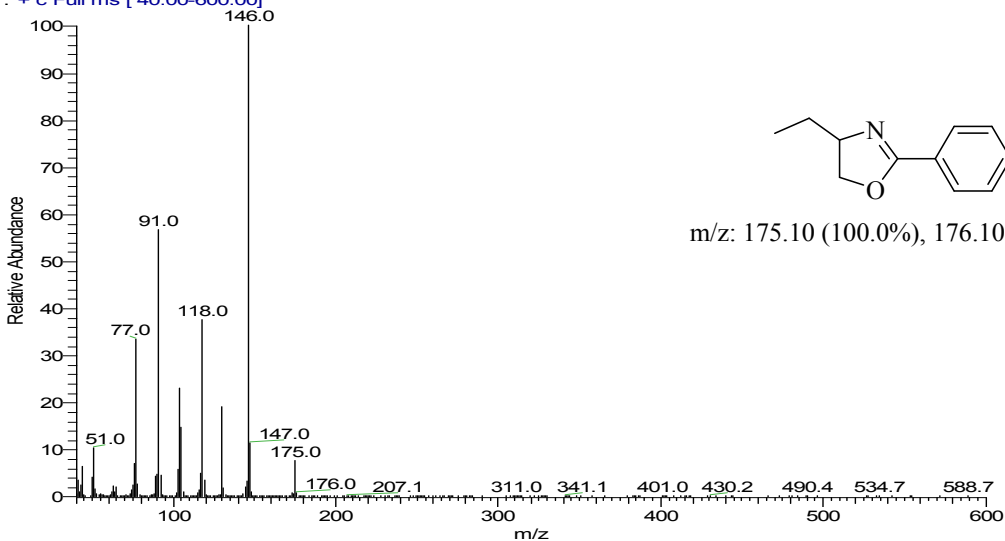
4-ethyl-2-phenyl-4,5-dihydrooxazole (3n, Table 2, Entry 14) [5]



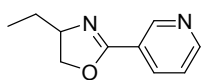
Isolated yield = 84%; $^1\text{H NMR}$ δ (300 MHz, CDCl_3) 7.82 (d, $J = 8.1$ Hz, Ar, 2H), 7.32 (d, $J = 7.9$ Hz, 1H), 7.20 (d, $J = 7.9$ Hz, Ar, 2H), 4.50 (t, $J = 7.9$ Hz, 1H), 4.27- 4.48 (m, 1H), 4.03 (t, $J = 7.9$ Hz, 1H), 1.62 – 1.91 (m, 2H), 1.0 (t, $J = 7.4$ Hz, 3H). **GC-MS** m/z 175.0 (M^+ peak), 146.0, 118.0, 91.0, 77.0.



UMA-ASOX-130-8H #482 RT: 12.16 AV: 1 NL: 4.60E6
T: + c Full ms [40.00-600.00]



4-ethyl-2-(pyridin-3-yl)-4,5-dihydrooxazole (3o, Table 2, Entry15)



Isolated yield = 74%; yellow liquid; IR (KBr) cm^{-1} : 2962, 2926, 2875,

1728, 1653, 1571, 1529, 1416, 1359, 1260, 1077, 1022, 947, 797. ^1H NMR

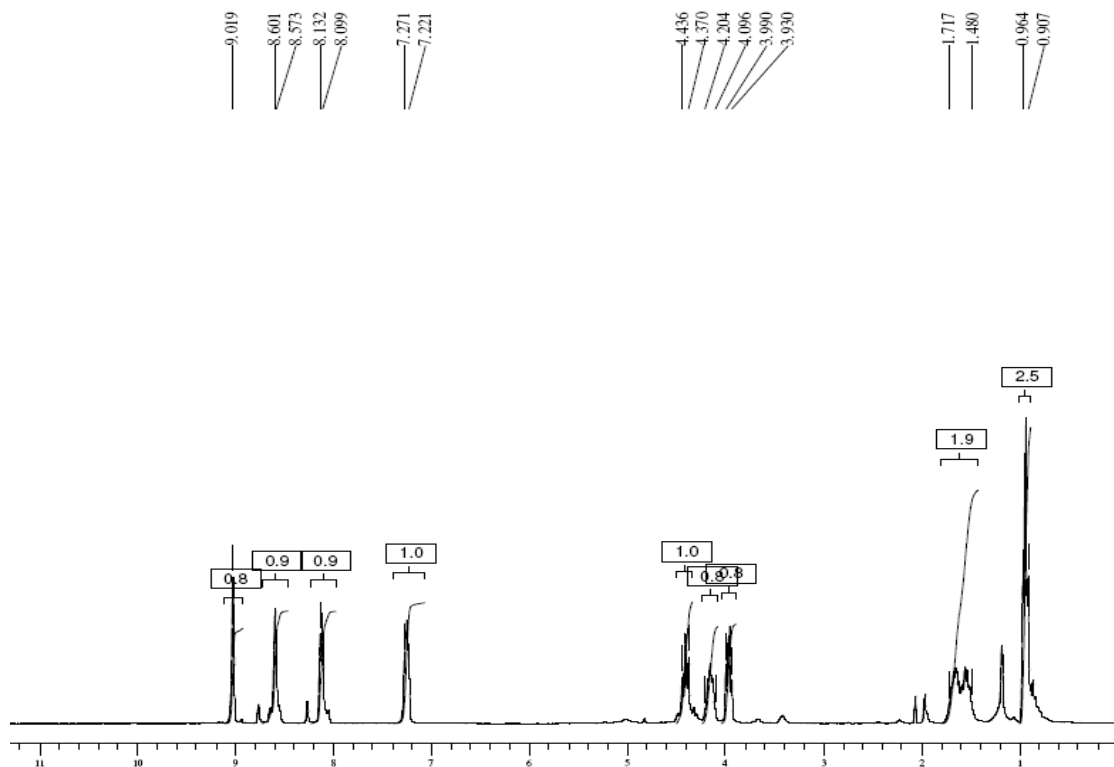
δ (300 MHz, CDCl_3) 9.02(s, Ar, 1H), 8.58 (d, $J = 8.1$ Hz, Ar, 1H), 8.11 (d, $J = 8.1$ Hz, 1H), 7.22

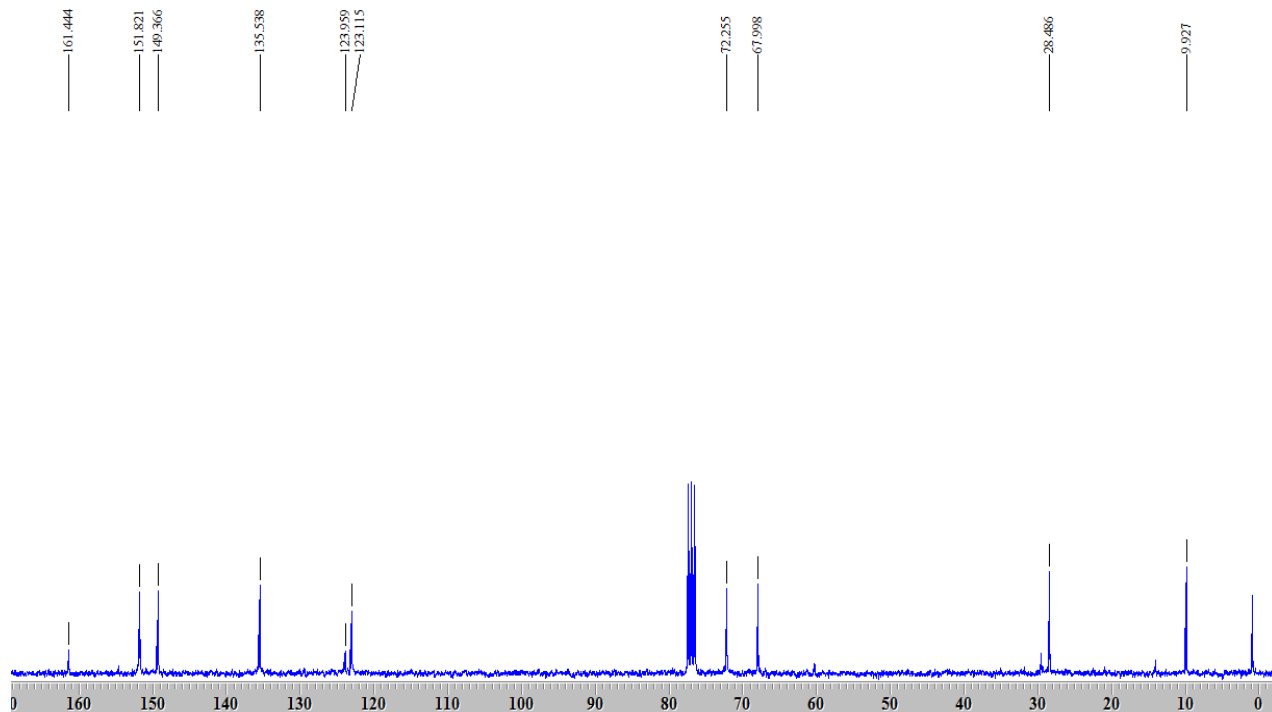
– 7.27 (m, Ar, 1H), 4.37 (t, $J = 8.1$ Hz, 1H), 4.17- 4.43 (m, 1H), 4.09 – 4.20 (m, 1H), 3.93 – 3.99

(m, 1H), 1.48 – 1.71 (m, 2H), 0.91 -0.96 (m, 3H). ^{13}C NMR (75 MHz, CDCl_3): 161.4, 151.8,

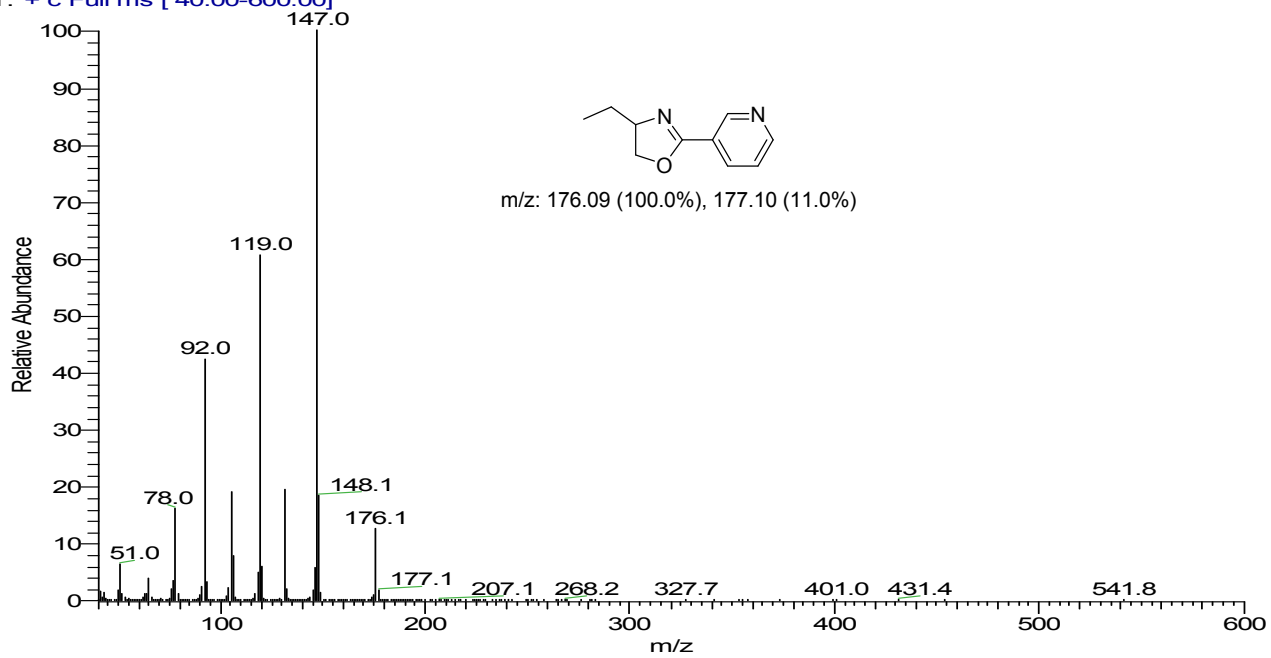
149.4, 135.5, 123.9, 123.1, 72.3, 67.9, 28.5, 9.9. **GC-MS** m/z 176.1 (M^+ peak), 147.0, 119.0,

92.0, 78.0.



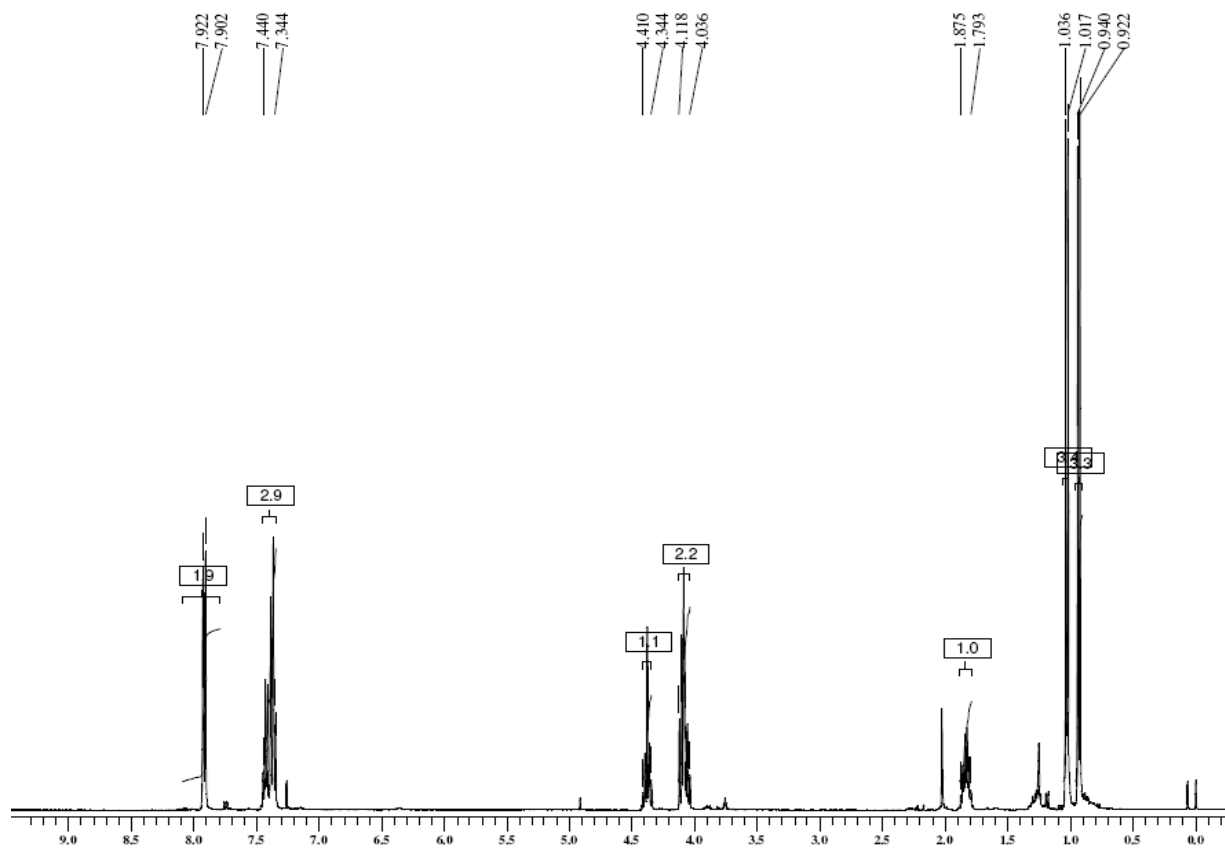


UMA-ASOX-138-4H #524 RT: 12.96 AV: 1 NL: 3.32E7
 T: + c Full ms [40.00-600.00]

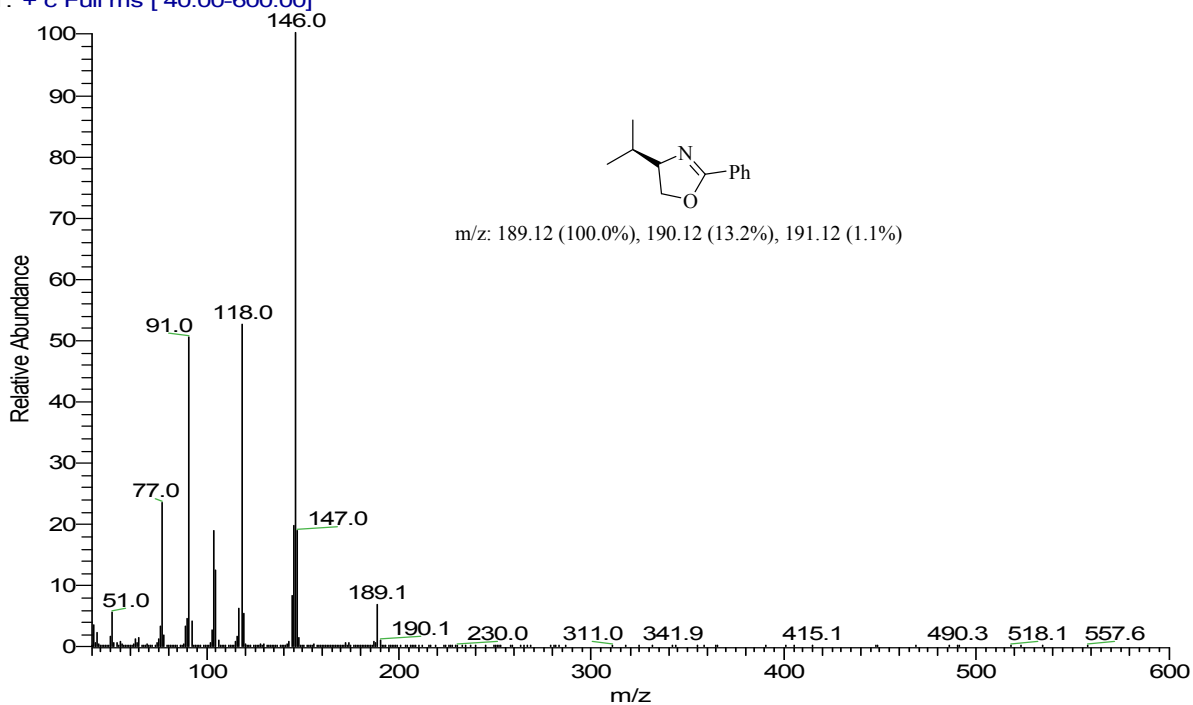


(S)-4-isopropyl-2-phenyl-4,5-dihydrooxazole (3p, Table 3, Entry 1) [6]

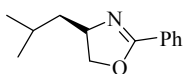
Isolated yield = 81%; $^1\text{H NMR } \delta(300 \text{ MHz, CDCl}_3)$ 7.91 (d, $J = 8.1 \text{ Hz, Ar, 2H}$), 7.34-7.44 (m, Ar, 3H), 4.34-4.41 (m, -CH, 1H), 4.04- 4.12 (m, -CH₂, 2H), 1.79-1.88 (m, -CH, 1H), 1.02 (d, $J = 7.3 \text{ Hz, -CH}_3$, 3H), 0.93 (d, $J = 7.3 \text{ Hz, -CH}_3$, 3H). **GC-MS** m/z 189.1, 146.0, 118.0, 91.0, 77.0, 51.0. Optical rotation: $[\alpha]_{\text{D}}^{22} = -76.4$, ($c = 0.63$ in CHCl_3) {Lit.⁵ $[\alpha]_{\text{D}}^{22} -83$ ($c = 0.63$, CHCl_3)}



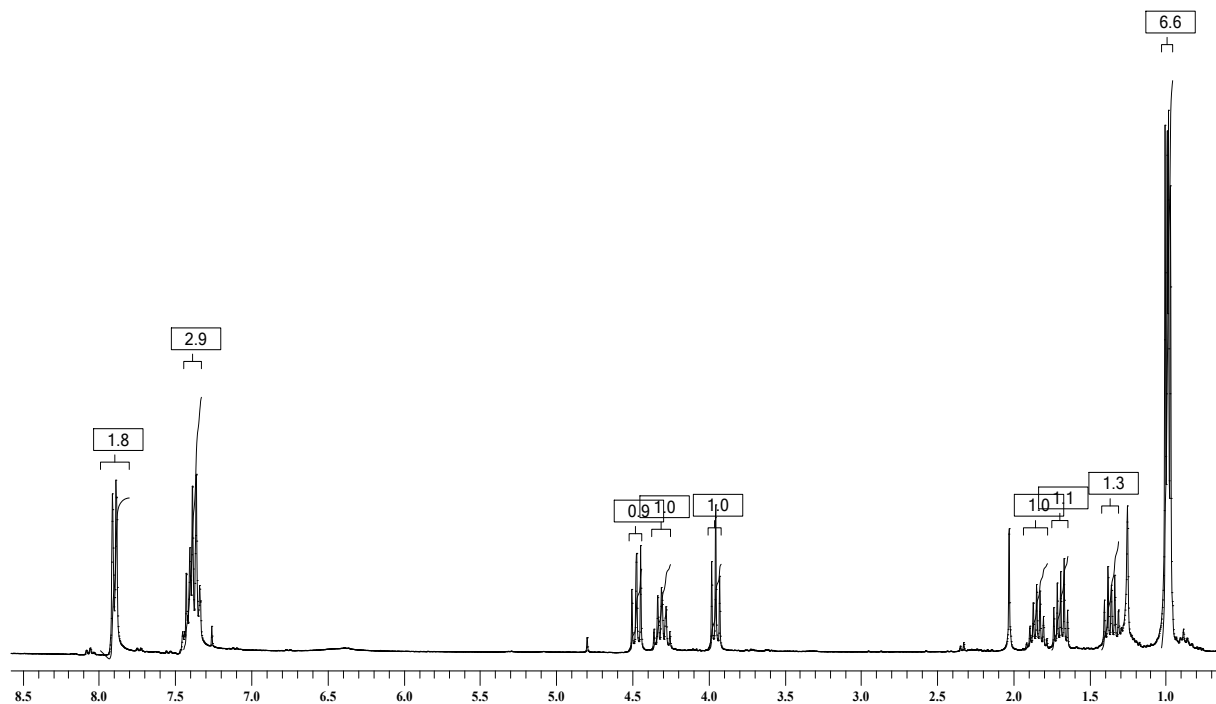
UMA-ASOX-115-20H #523 RT: 12.94 AV: 1 NL: 3.75E7
T: + c Full ms [40.00-600.00]



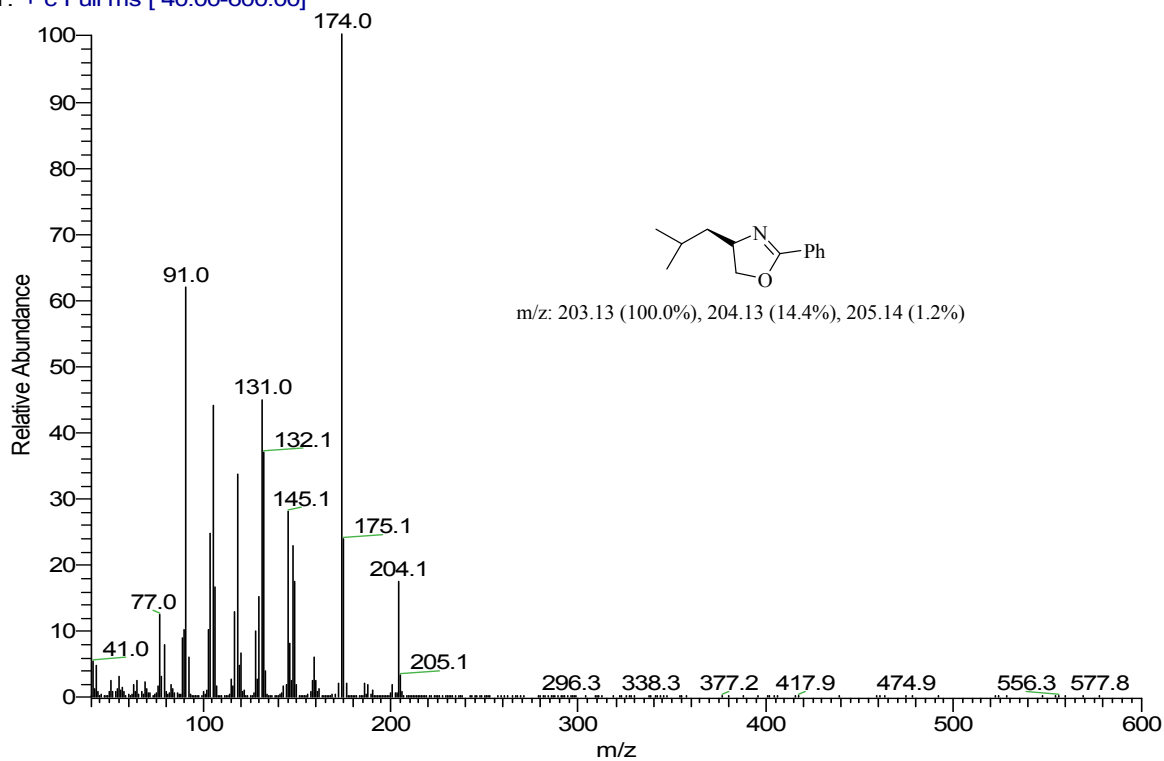
(S)-4-isobutyl-2-phenyl-4,5-dihydrooxazole (3q, Table 3, Entry 2) [8]



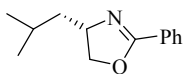
Isolated yield = 86%; $^1\text{H NMR}$ δ (300 MHz, CDCl_3) 7.89 (d, $J = 8.3$ Hz, Ar, 2H), 7.33- 7.45 (m, Ar, 3H), 4.47 (t, $J = 9.8$ Hz, 7.5 Hz, 1H), 4.25- 4.36 (m, H), 3.95 (t, 7.5 Hz, 1H), 1.78 - 1.91 (m, 1H), 1.64 – 1.74 (m, 1H), 1.31 – 1.40 (m, 1H), 0.97 - 1.00 (m, $-(\text{CH}_3)_2$, 6H). **GC-MS** m/z 204.1 (M+1), 174.0, 145.1, 132.1, 131.0, 91.0, 77.0. Optical rotation: $[\alpha] = -66.1$, $c = 1.0$ in CHCl_3 . {Lit.⁷ $[\alpha]_{\text{D}}^{21} = -76.9$ ($c = 1$, CHCl_3)}



UMA-AOX-110-6H #610 RT: 14.59 AV: 1 NL: 4.00E7
 T: + c Full ms [40.00-600.00]



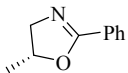
(R)-4-isobutyl-2-phenyl-4,5-dihydrooxazole (3r, Table 3, Entry 3) [8]



Optical rotation: $[\alpha] = +71.5$, $c = 1.0$ in CHCl_3 . {Lit. [8] $[\alpha]_{\text{D}}^{21} = -76.9$ for

(S)-4-isobutyl-2-phenyl-4,5-dihydrooxazole ($c = 1.0$, CHCl_3)}

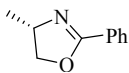
(R)-5-methyl-2-phenyl-4,5-dihydrooxazole (3s, Table 3, Entry 4) [9]



Optical rotation: $[\alpha] = -4.5$, $c = 1.0$ in MeOH . {Lit. [9] $[\alpha]_{\text{D}}^{25} = -5.1$ ($c = 1.0$,

MeOH)}

(S)-4-methyl-2-phenyl-4,5-dihydrooxazole (3t, Table 3, Entry 5) [9]



Optical rotation: $[\alpha] = -74.2$, $c = 1.0$ in EtOH . {Lit.[9] $[\alpha]_{\text{D}}^{25} = -79.8$ ($c = 1.0$,

EtOH)}

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