

Supporting Information

Pd NPs decorated on crosslinked sodium alginate modified Iron-based metal-organic framework Fe(BTC) as a green multifunctional catalyst for the oxidative amidation

Samaneh Koosha^a, Ramin Ghorbani-Vaghei^{a*}, Sedigheh Alavinia^a, Rahman Karimi-Nami^b, Idris Karakaya^c

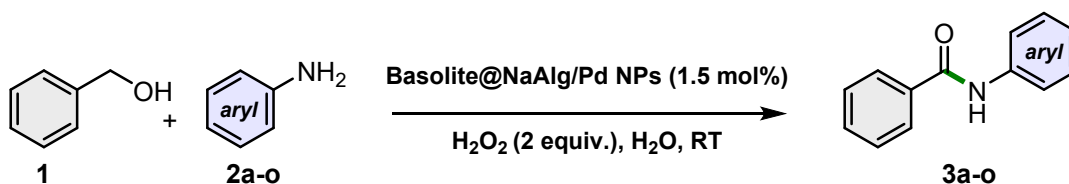
^aDepartment of Organic Chemistry, Faculty of Chemistry and Petroleum Sciences, Bu-Ali Sina University, 6517838683, Hamadan, Iran.

^bDepartment of Chemistry, Faculty of Science, University of Maragheh, Maragheh, Iran.

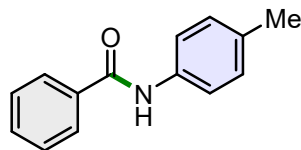
^cDepartment of Chemistry, College of Basic Sciences, Gebze Technical University, 41400 Gebze, Turkey.

Corresponding Author: rgvaghei@yahoo.com & ghorbani@basu.ac.ir

General procedure for the one-pot synthesis of secondary amides

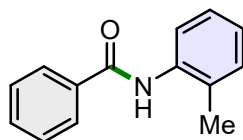


In a standard experimental procedure, the following components were combined in a 25 mL round-bottomed flask: 1.0 mmol of benzyl alcohol, 2.0 mmol of aqueous H₂O₂ (30%), and 1.5 mol% of basolite@SA/ED /Pd NP catalyst in 3 mL of H₂O. The mixture was then stirred at room temperature for 2 hours. After this initial reaction period, 1.0 mmol of aniline was introduced into the reaction mixture. Upon completion of the reaction, the catalyst was separated by centrifugation, and the organic residue was subsequently extracted with ethyl acetate. The solvent was then removed under reduced pressure. Finally, the resulting product was subjected to purification through column chromatography.



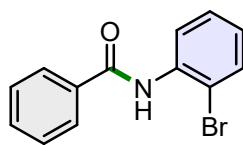
3b

***N*-(*p*-tolyl)benzamide (3b):** ^1H NMR (500 MHz, $\text{DMSO-}d_6$, TMS) δ 7.94 (s, 1H), 7.84 (d, J = 7.5 Hz, 2H), 7.55 – 7.48 (m, 3H), 7.44 (t, J = 7.6 Hz, 2H), 7.14 (d, J = 8.2 Hz, 2H), 2.33 (s, 3H). ^{13}C NMR (126 MHz, $\text{DMSO-}d_6$, TMS) δ 166.03, 135.54, 135.45, 135.03, 134.22, 131.68, 130.16, 129.53, 129.39, 128.66, 128.45, 127.14, 120.60, 21.08. FTIR (cm^{-1}) 3310.34 (NH), 1646.90 (C=O).



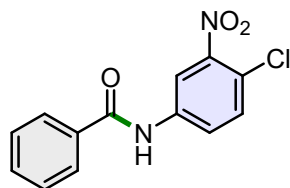
3c

***N*-(*o*-tolyl)benzamide (3c):** ^1H NMR (500 MHz, CDCl_3 , TMS) δ 8.00 – 7.84 (m, 3H), 7.76 (s, 1H), 7.55 (dd, J = 28.5, 5.7 Hz, 3H), 7.32 – 7.21 (m, 2H), 7.15 (d, J = 6.1 Hz, 1H), ^1H NMR (500 MHz, CDCl_3) δ 7.94 (s, 1H), 7.84 (d, J = 7.5 Hz, 2H), 7.56 – 7.48 (m, 3H), 7.44 (t, J = 7.6 Hz, 2H), 7.14 (d, J = 8.2 Hz, 2H), 2.36 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3 , TMS) δ 165.83, 135.79, 134.97, 131.85, 130.50, 129.67, 128.95, 128.83, 128.41, 127.13, 126.87, 125.49, 123.44, 17.87; FTIR (cm^{-1}) 3246.88 (NH), 1649.86 (C=O).



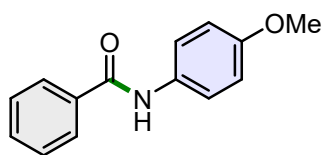
3g

***N*-(2-bromophenyl)benzamide (3g):** ^1H NMR (500 MHz, CDCl_3 , TMS) δ 8.56 (d, J = 8.1 Hz, 1H), 8.47 (s, 1H), 7.94 (d, J = 7.5 Hz, 2H), 7.55 (dd, J = 24.2, 7.4 Hz, 4H), 7.38 (t, J = 7.7 Hz, 1H), 7.02 (t, J = 7.6 Hz, 1H); FTIR (cm^{-1}) 3219.29 (NH), 1651.60 (C=O).



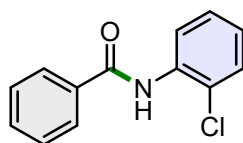
3k

***N*-(4-chloro-3-nitrophenyl)benzamide (3k):** ^1H NMR (500 MHz, CDCl_3) δ 13.98 (s, 1H), 8.31 (s, 1H), 8.07 (s, 1H), 7.98 – 7.75 (m, 3H), 7.57 (dd, $J = 29.3, 10.4$ Hz, 3H); ^{13}C NMR (126 MHz, $\text{DMSO-}d_6$, TMS) δ 165.89, 147.96, 137.58, 133.69, 132.65, 132.28, 129.06, 127.12, 124.29, 121.85, 116.80; FTIR (cm^{-1}) 3406.99 (NH), 1683.95 (C=O).



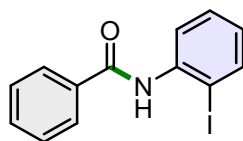
3d

***N*-(4-methoxyphenyl)benzamide (3d):** ^1H NMR (500 MHz, CDCl_3 , TMS) δ 8.12 (s, 1H), 7.85 (s, 2H), 7.53 (d, $J = 0.8$ Hz, 2H), 7.42 (d, $J = 28.3$ Hz, 3H), 6.82 (d, $J = 4.1$ Hz, 2H), 3.74 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3 , TMS) δ 165.71, 156.54, 135.00, 131.64, 131.22, 128.67, 127.18, 122.25, 114.16, 55.34; FTIR (cm^{-1}) 3330.69 (NH), 1646.99 (C=O).



3h

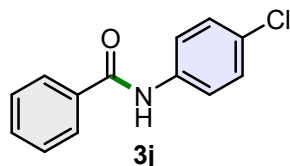
***N*-(2-chlorophenyl)benzamide (3h):** ^1H NMR (500 MHz, CDCl_3 , TMS) δ 8.45 (d, $J = 8.2$ Hz, 1H), 8.36 (s, 1H), 7.82 (d, $J = 7.5$ Hz, 2H), 7.44 (dt, $J = 15.0, 7.3$ Hz, 3H), 7.30 (d, $J = 8.0$ Hz, 1H), 7.22 (t, $J = 7.8$ Hz, 1H), 6.97 (t, $J = 7.7$ Hz, 1H); FTIR (cm^{-1}) 3226.77 (NH), 1653.24 (C=O).



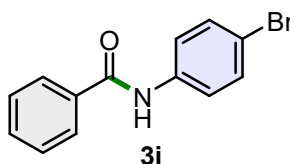
3f

***N*-(2-iodophenyl)benzamide (3f):** ^1H NMR (500 MHz, CDCl_3) δ 8.37 (d, $J = 7.5$ Hz, 1H), 8.22 (s, 1H), 7.89 (d, $J = 7.4$ Hz, 2H), 7.73 (d, $J = 7.9$ Hz, 1H), 7.50 (t, $J = 7.4$ Hz, 1H), 7.44 (t, $J = 7.5$ Hz, 2H), 7.32 (t, $J = 7.7$ Hz, 1H), 6.80 (t, $J = 7.6$ Hz, 1H); ^{13}C NMR (126 MHz, CDCl_3 , TMS) δ

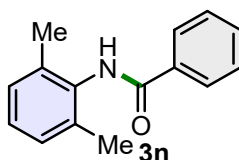
165.38, 138.87, 138.31, 134.48, 132.03, 129.42, 128.99, 127.25, 126.18, 122.05, 90.63; FTIR (cm^{-1}) 3215.80 (NH), 1649.33 (C=O).



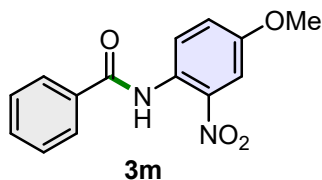
***N*-(4-chlorophenyl)benzamide (3j):** ^1H NMR (500 MHz, CDCl_3 , TMS) δ 7.79 (s, 2H), 7.51 (d, $J = 20.1$ Hz, 1H), 7.43 (m, 3H), 7.27 (d, $J = 3.7$ Hz, 3H), 7.19 (s, 1H); FTIR (cm^{-1}) 3349.44 (NH), 1654.64 (C=O).



***N*-(4-bromophenyl)benzamide (3i):** ^1H NMR (500 MHz, CDCl_3 , TMS) δ 7.79 (d, $J = 7.5$ Hz, 2H), 7.75 (s, 1H), 7.54 – 7.46 (m, 2H), 7.42 (t, $J = 9.1$ Hz, 3H), 7.19 (s, 1H); ^{13}C NMR (126 MHz, $\text{DMSO-}d_6$, TMS) δ 164.62, 135.99, 133.61, 131.06, 130.88, 127.86, 125.98, 125.83, 120.68; FTIR (cm^{-1}) 3333.19 (NH), 1648.04 (C=O).

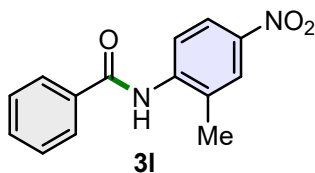


***N*-(2,6-dimethylphenyl)benzamide (3n):** ^1H NMR (500 MHz, CDCl_3 , TMS) δ 7.82 (d, $J = 7.4$ Hz, 2H), 7.47 (t, $J = 7.3$ Hz, 2H), 7.39 (t, $J = 7.6$ Hz, 2H), 7.06 (dd, $J = 8.9, 5.6$ Hz, 1H), 7.03 – 7.01 (m, 2H), 2.18 (s, 6H); ^{13}C NMR (126 MHz, CDCl_3 , TMS) δ 165.98, 135.60, 134.45, 133.91, 131.76, 128.72, 128.25, 127.40, 127.24, 18.45; FTIR (cm^{-1}) 3274.83 (NH), 1644.05 (C=O).

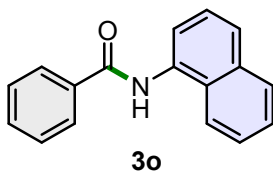


***N*-(4-chloro-3-nitrophenyl)benzamide (3m):** ^1H NMR (500 MHz, CDCl_3 , TMS) δ 11.01 (s, 1H), 8.81 (d, $J = 9.3$ Hz, 1H), 7.90 (d, $J = 7.5$ Hz, 2H), 7.64 (d, $J = 2.6$ Hz, 1H), 7.51 (t, $J = 7.2$ Hz,

1H), 7.45 (t, $J = 7.5$ Hz, 2H), 7.21 (dd, $J = 13.9, 7.3$ Hz, 1H), 3.80 (s, 3H); ^{13}C NMR(126 MHz, CDCl_3 , TMS) δ 165.54, 155.02, 137.07, 134.16, 132.48, 129.05, 129.01, 127.29, 123.68, 108.69, 17.93; FTIR (cm^{-1}) 3335.72 (NH), 1679.45 (C=O).



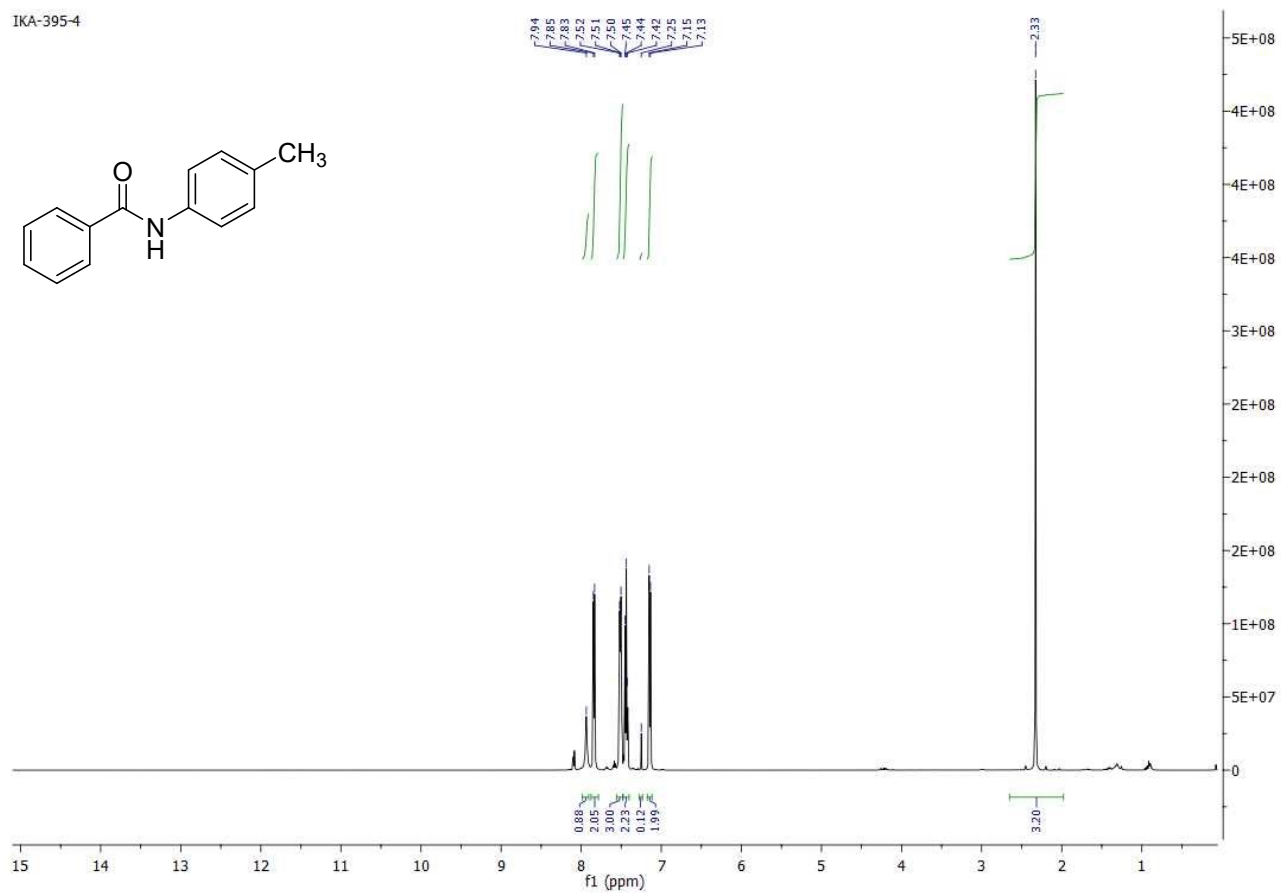
***N*-(2-methyl-4-nitrophenyl)benzamide (31):** ^1H NMR (500 MHz, CDCl_3 , TMS) δ 8.40 (d, $J = 8.9$ Hz, 1H), 8.07 (dd, $J = 13.8, 4.8$ Hz, 2H), 7.82 (d, $J = 7.4$ Hz, 2H), 7.54 (t, $J = 7.4$ Hz, 1H), 7.46 (t, $J = 7.6$ Hz, 2H), 2.39 (s, 3H); ^{13}C NMR (126 MHz, CDCl_3 , TMS) δ 165.51, 143.61, 134.21, 132.65, 129.15, 128.78, 127.09, 125.73, 123.03, 120.97, 113.14, 17.90; FTIR (cm^{-1}) 3309.58 (NH), 1651.80 (C=O).

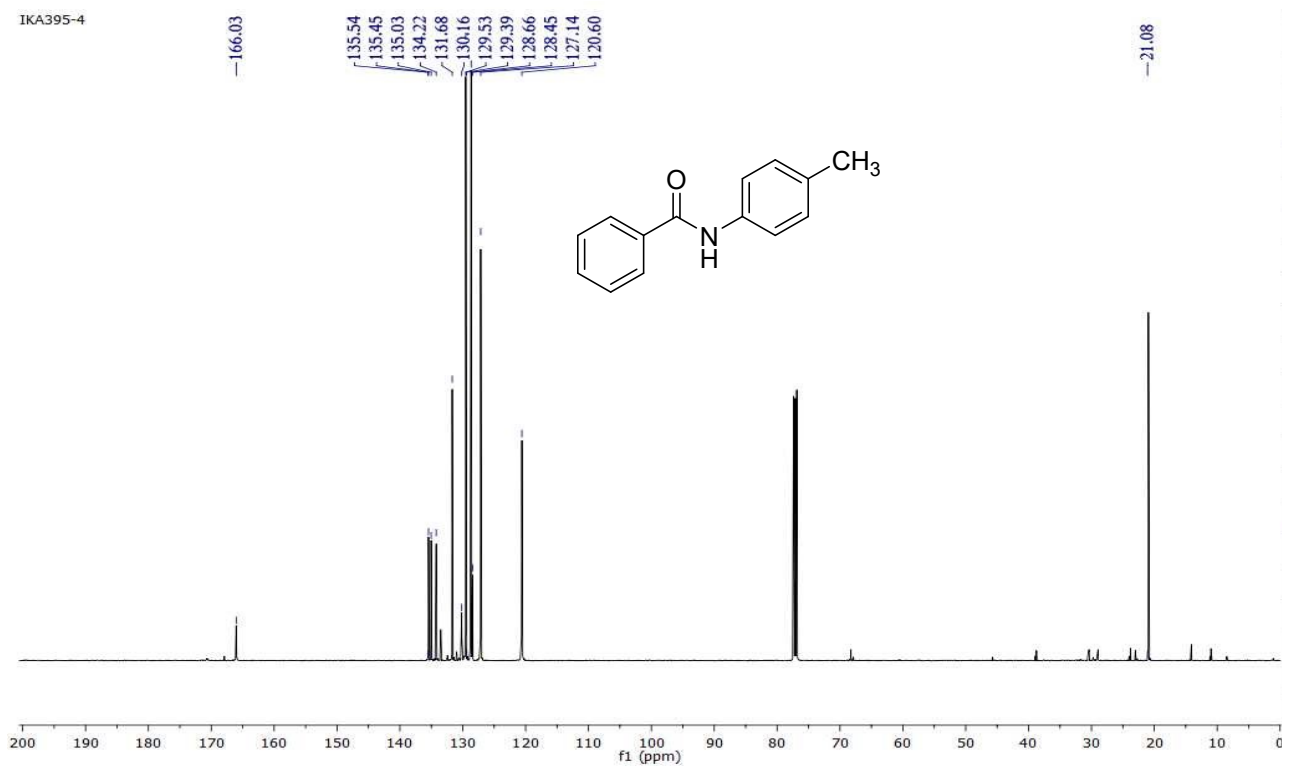


***N*-(naphthalen-1-yl)benzamide (30):** ^1H NMR (500 MHz, CDCl_3 , TMS) δ 8.20 (s, 1H), 7.88 (d, $J = 7.2$ Hz, 3H), 7.84 – 7.77 (m, 2H), 7.65 (d, $J = 8.2$ Hz, 1H), 7.49 (t, $J = 7.3$ Hz, 1H), 7.46 – 7.36 (m, 4H), 7.17 (s, 1H); ^{13}C NMR (126 MHz, CDCl_3 , TMS) δ 166.34, 134.85, 134.19, 131.97, 128.88, 128.85, 127.24, 126.43, 126.17, 126.08, 125.79, 121.38, 120.79; FTIR (cm^{-1}) 3235.26 (NH), 1647.10 (C=O).

Spectral Data

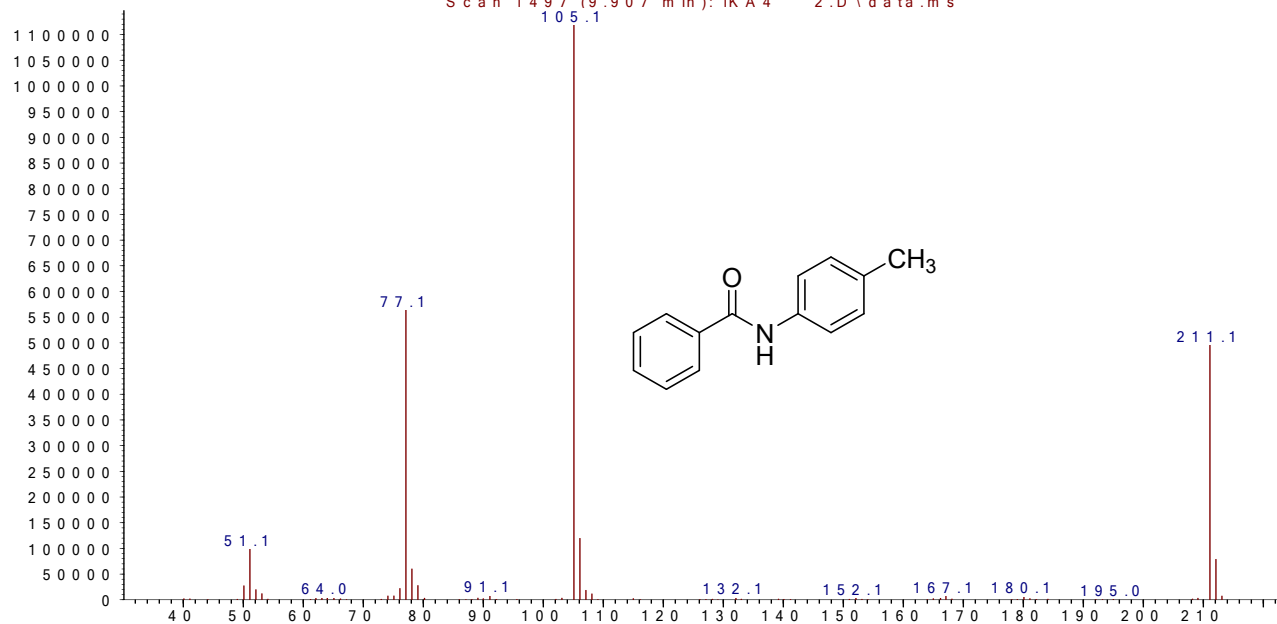
^1H NMR, ^{13}C NMR, mass and IR spectrum of *N*-(*o*-tolyl)benzamide (3b)





Abundance

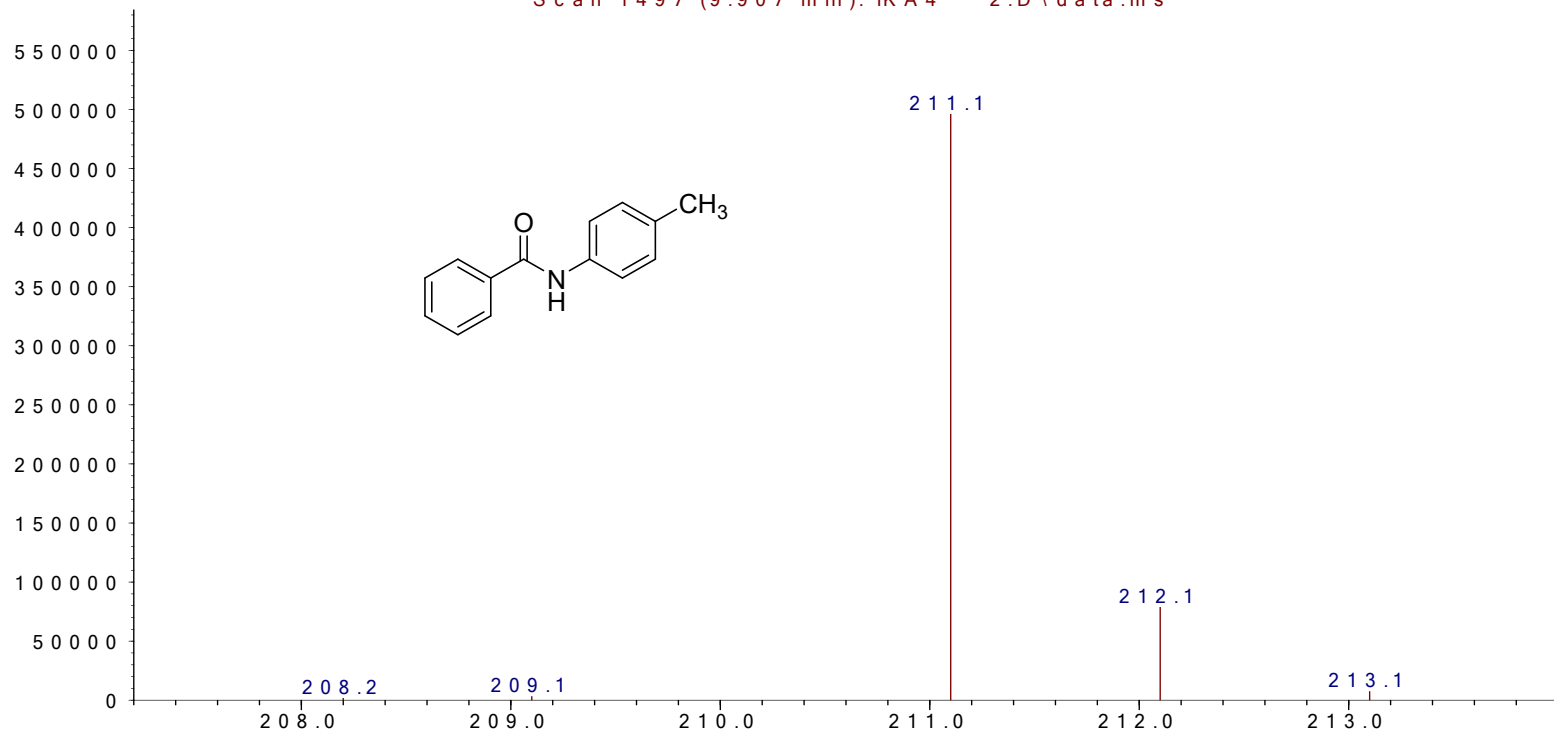
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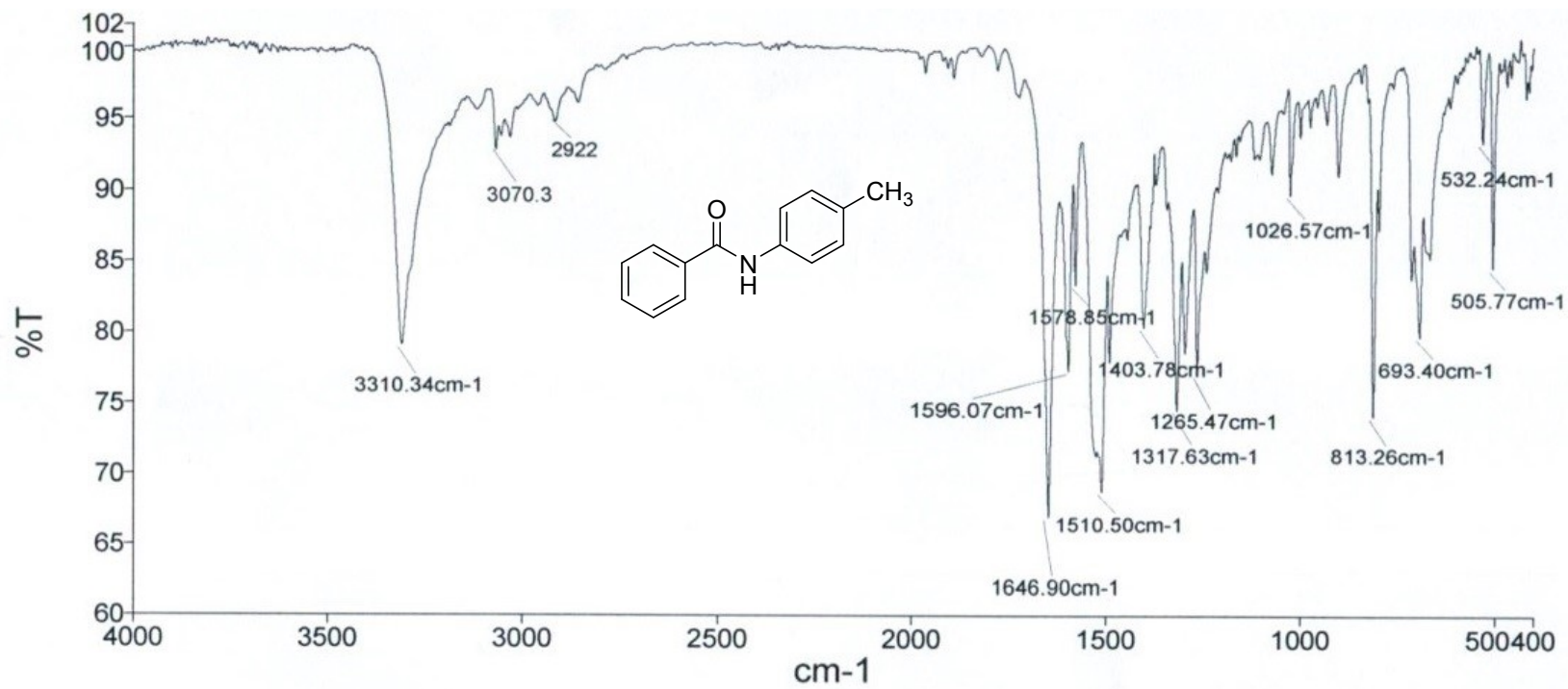
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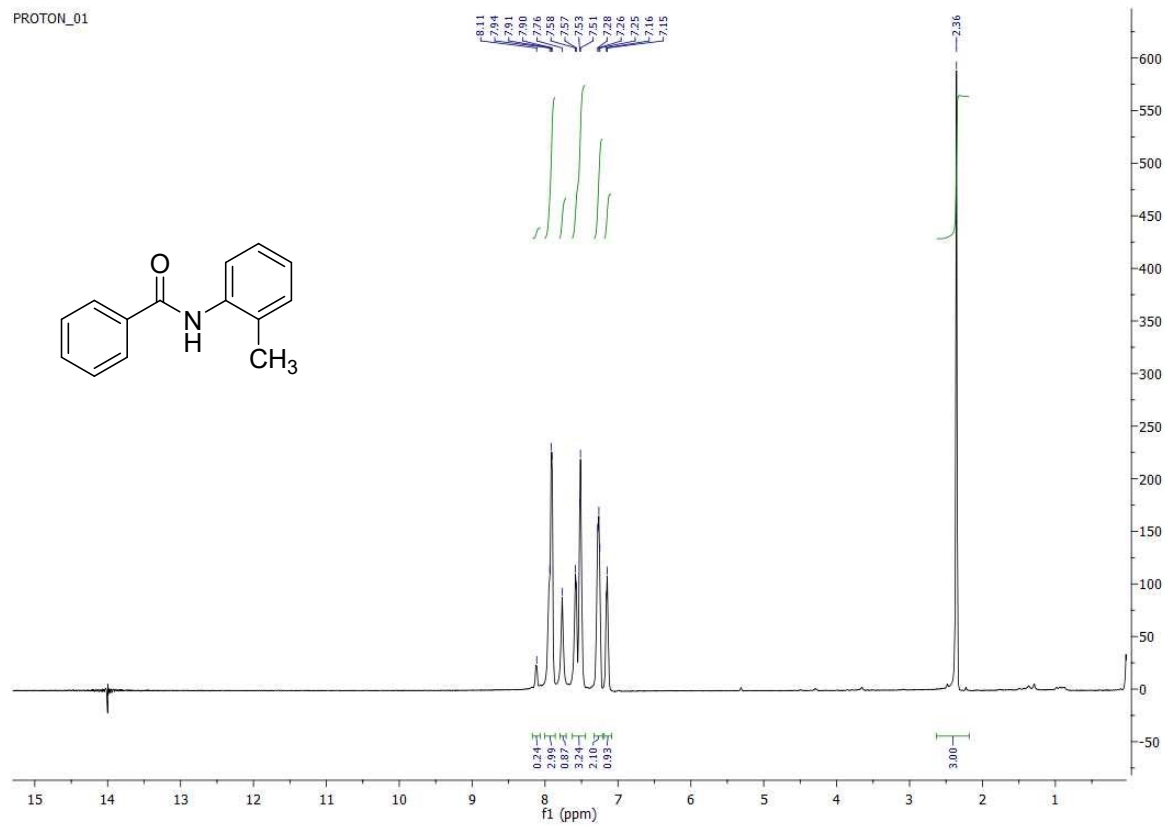
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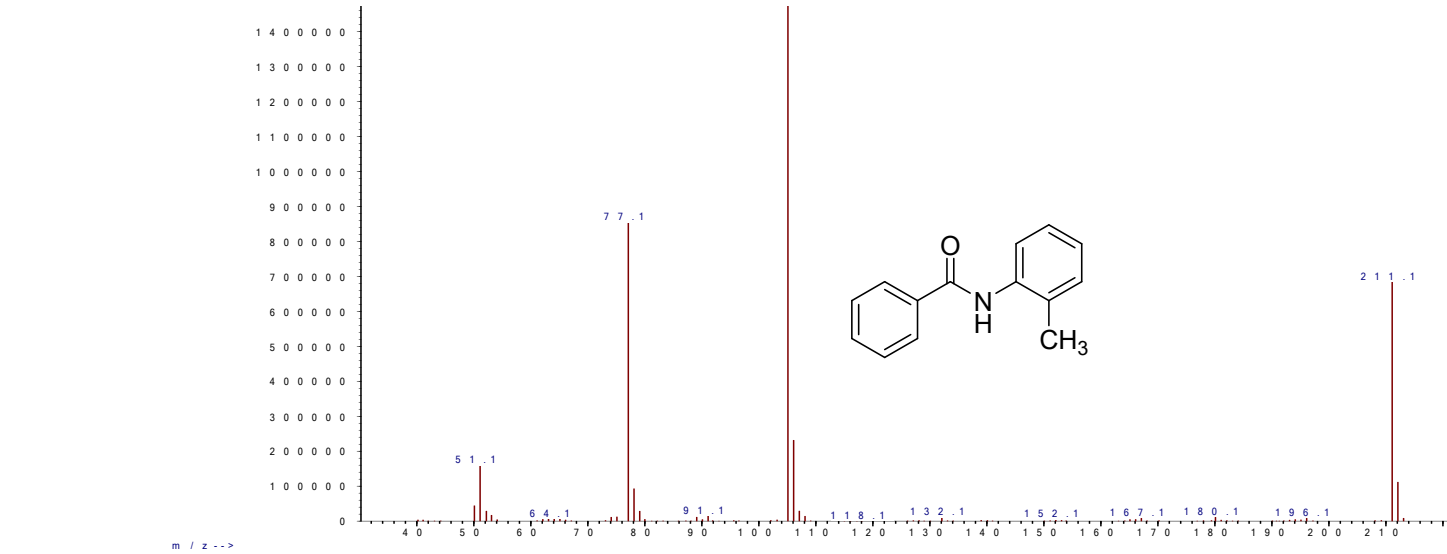
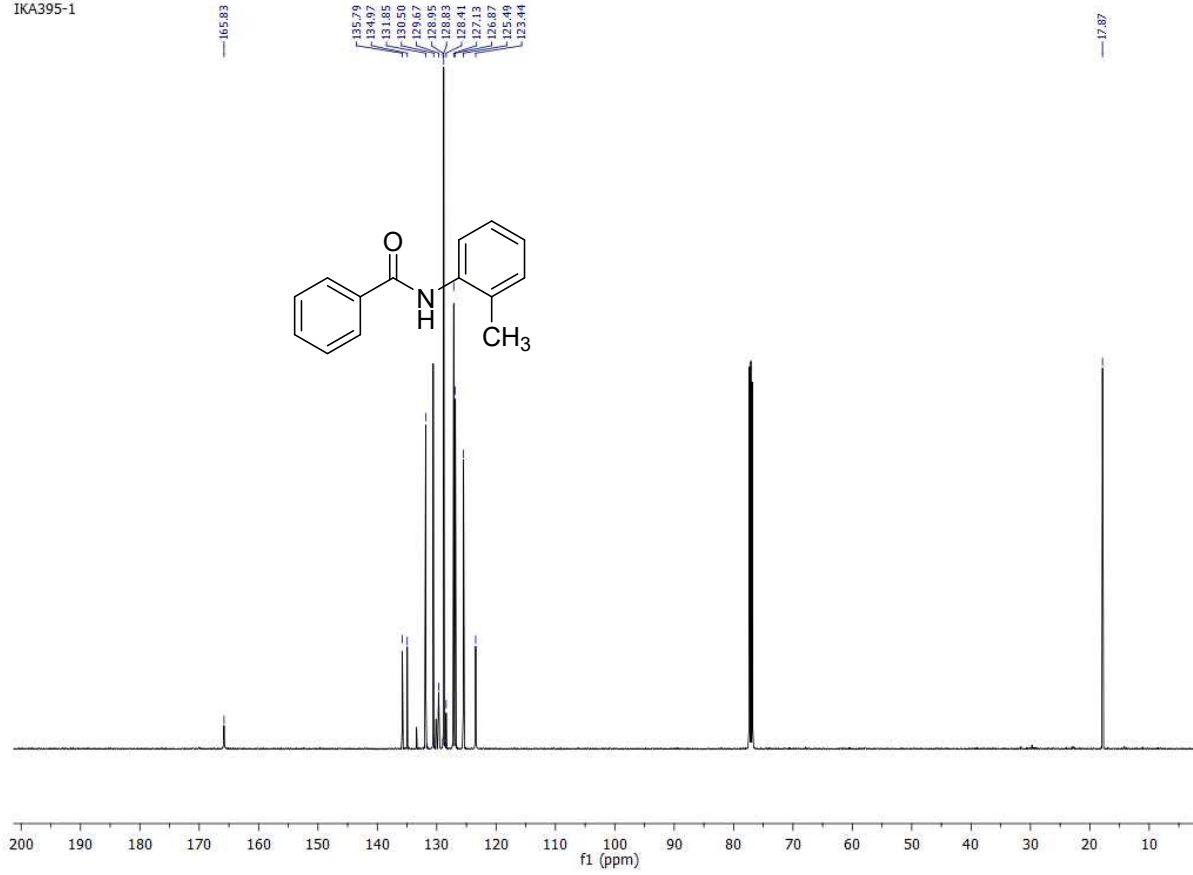
m / z-->



^1H NMR, ^{13}C NMR, mass and IR spectrum of *N*-(*o*-tolyl)benzamide (3c)

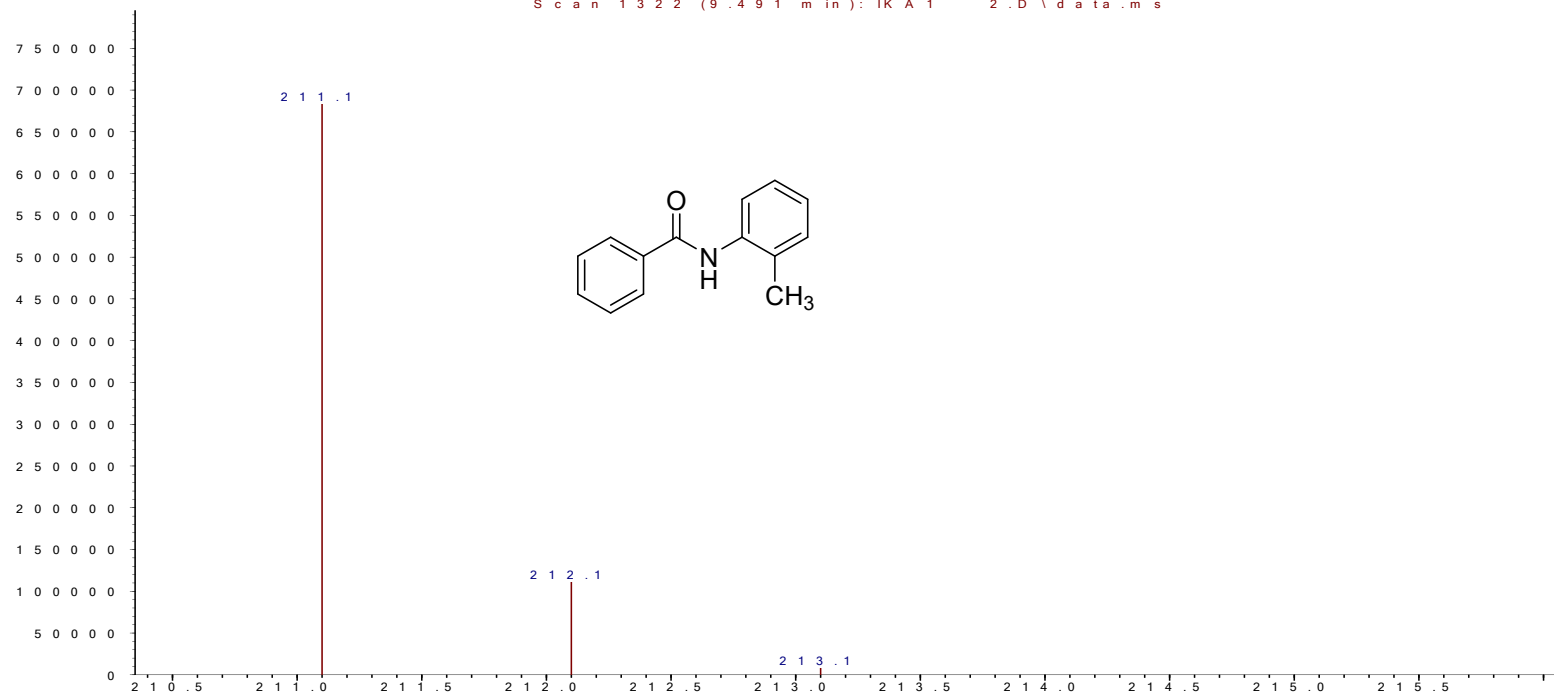


IKA395-1

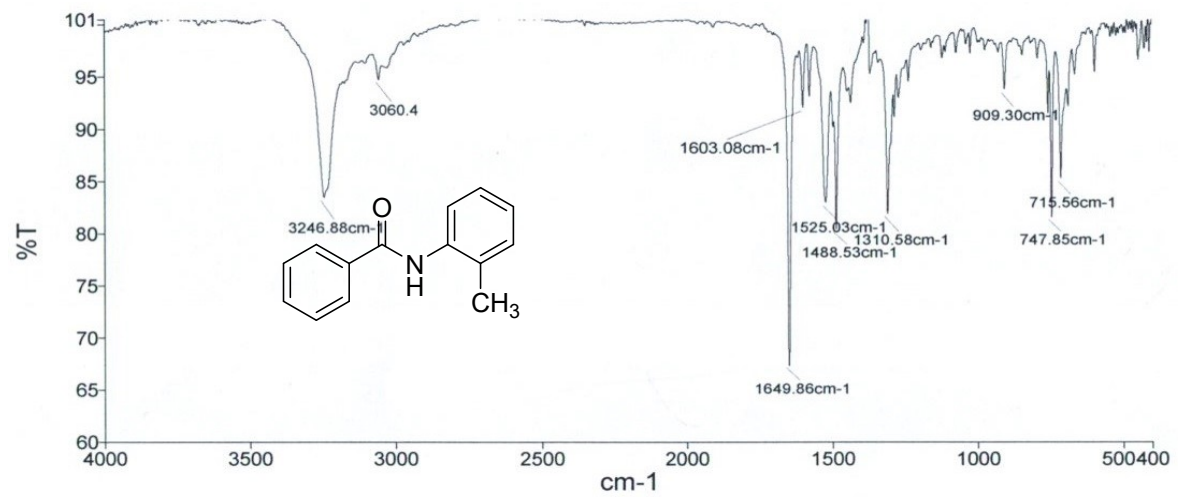


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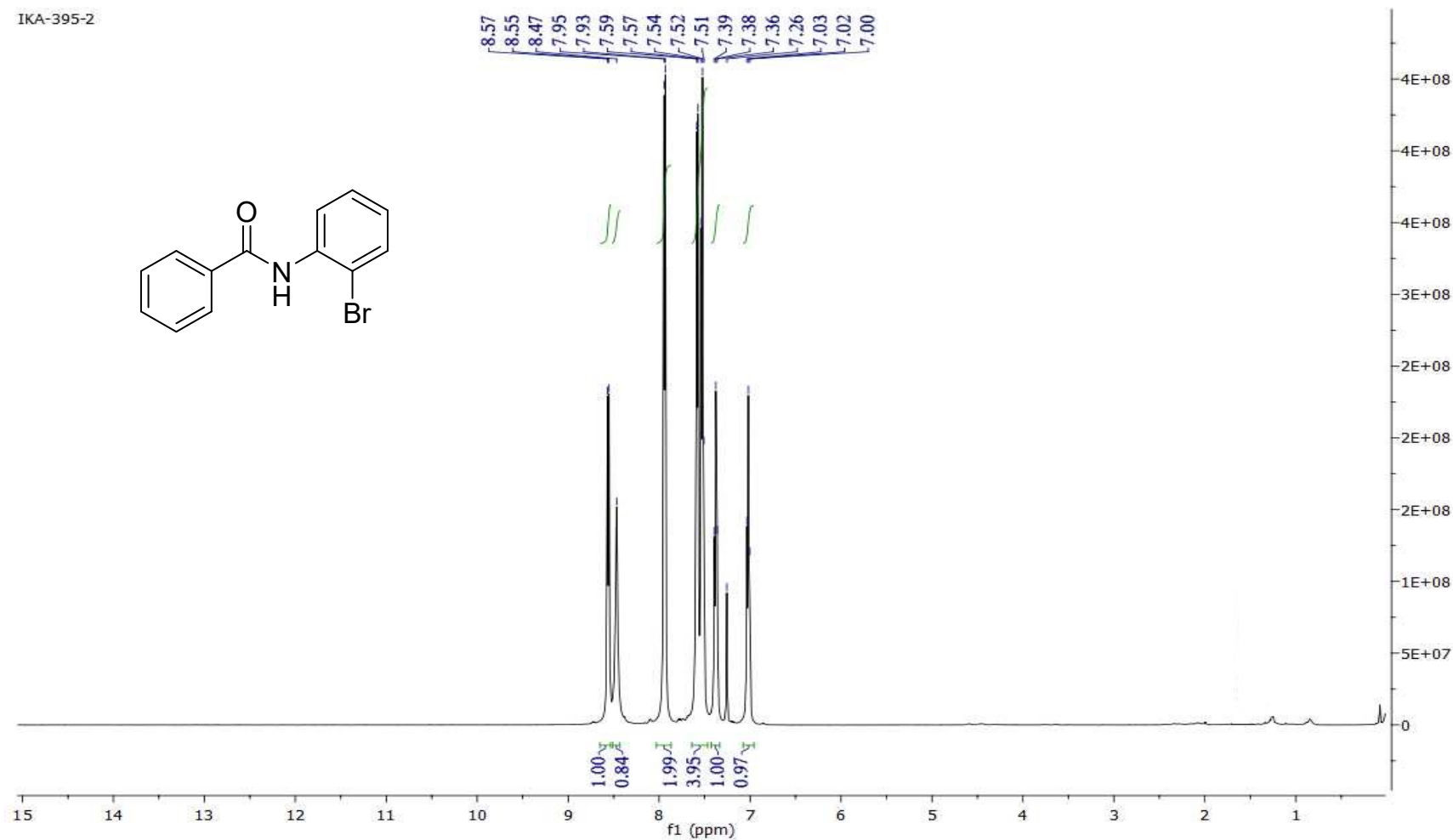
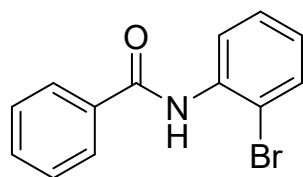


m / z-->



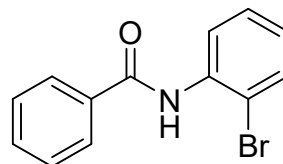
¹H NMR, mass and IR spectrum of *N*-(2-bromophenyl)benzamide (3g)

IKA-395-2

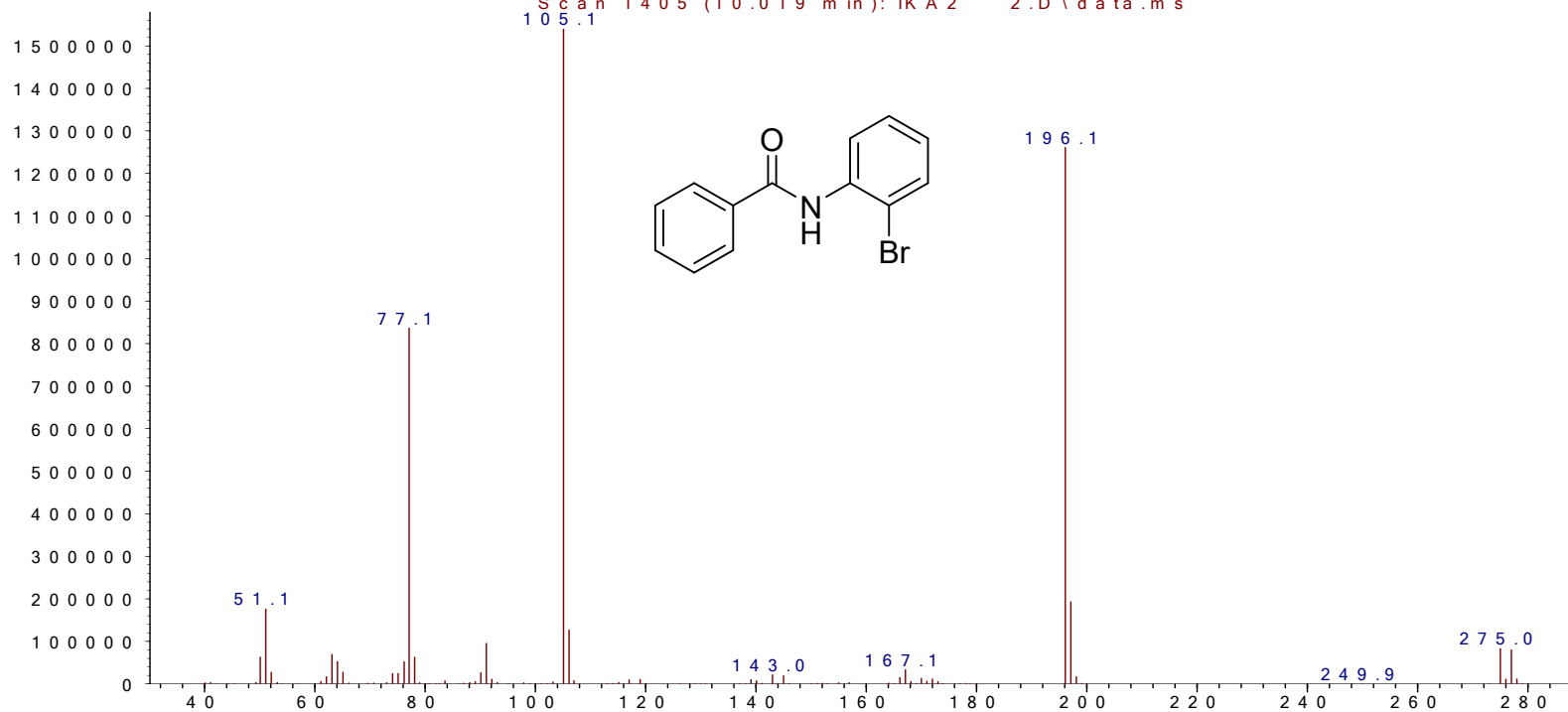


Abundance

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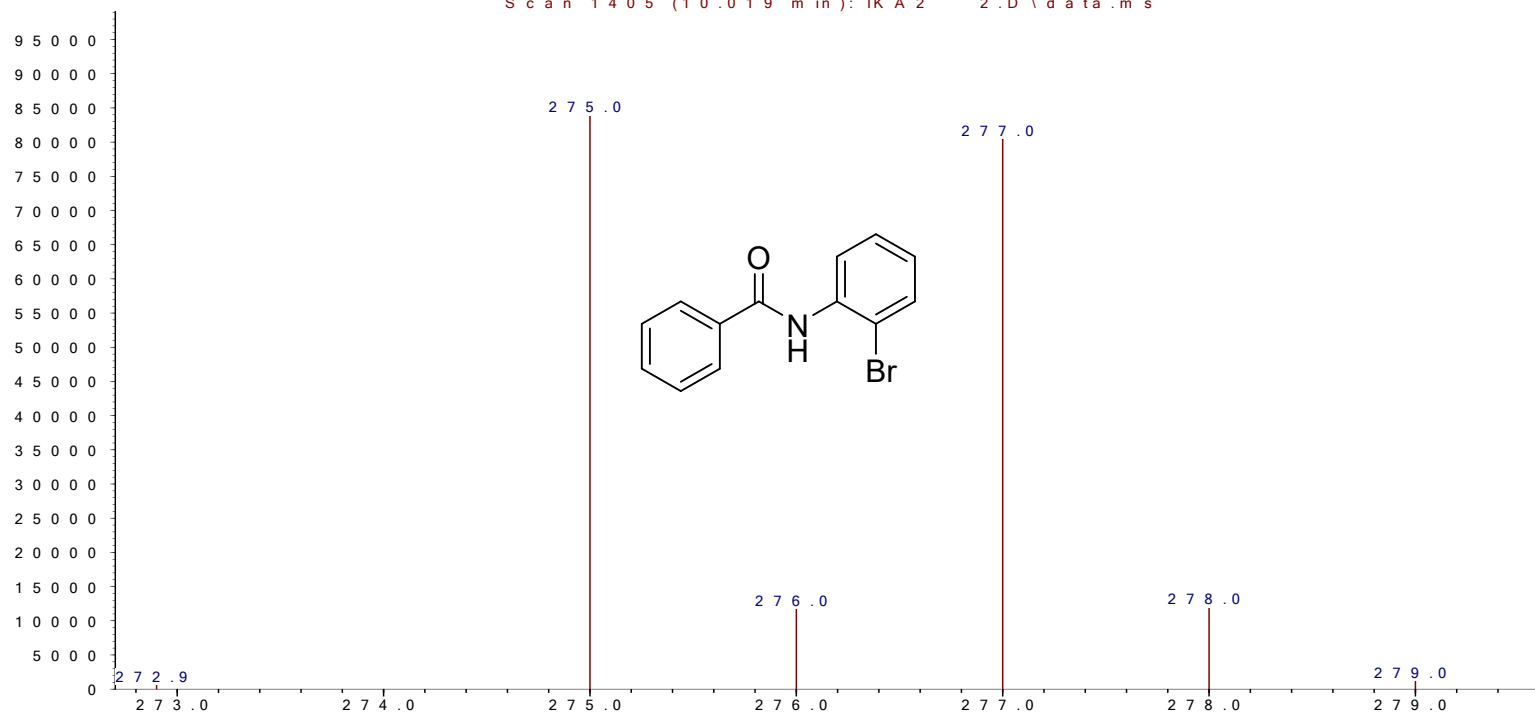


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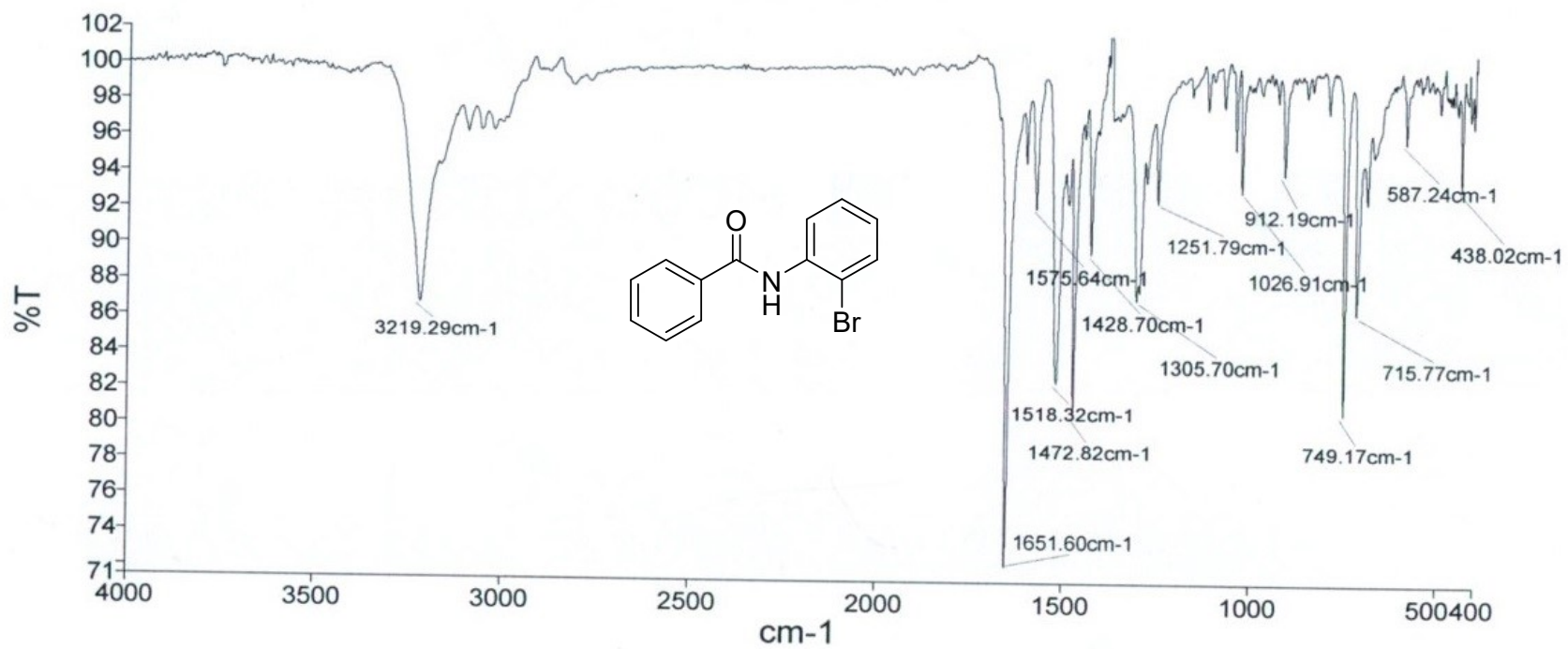


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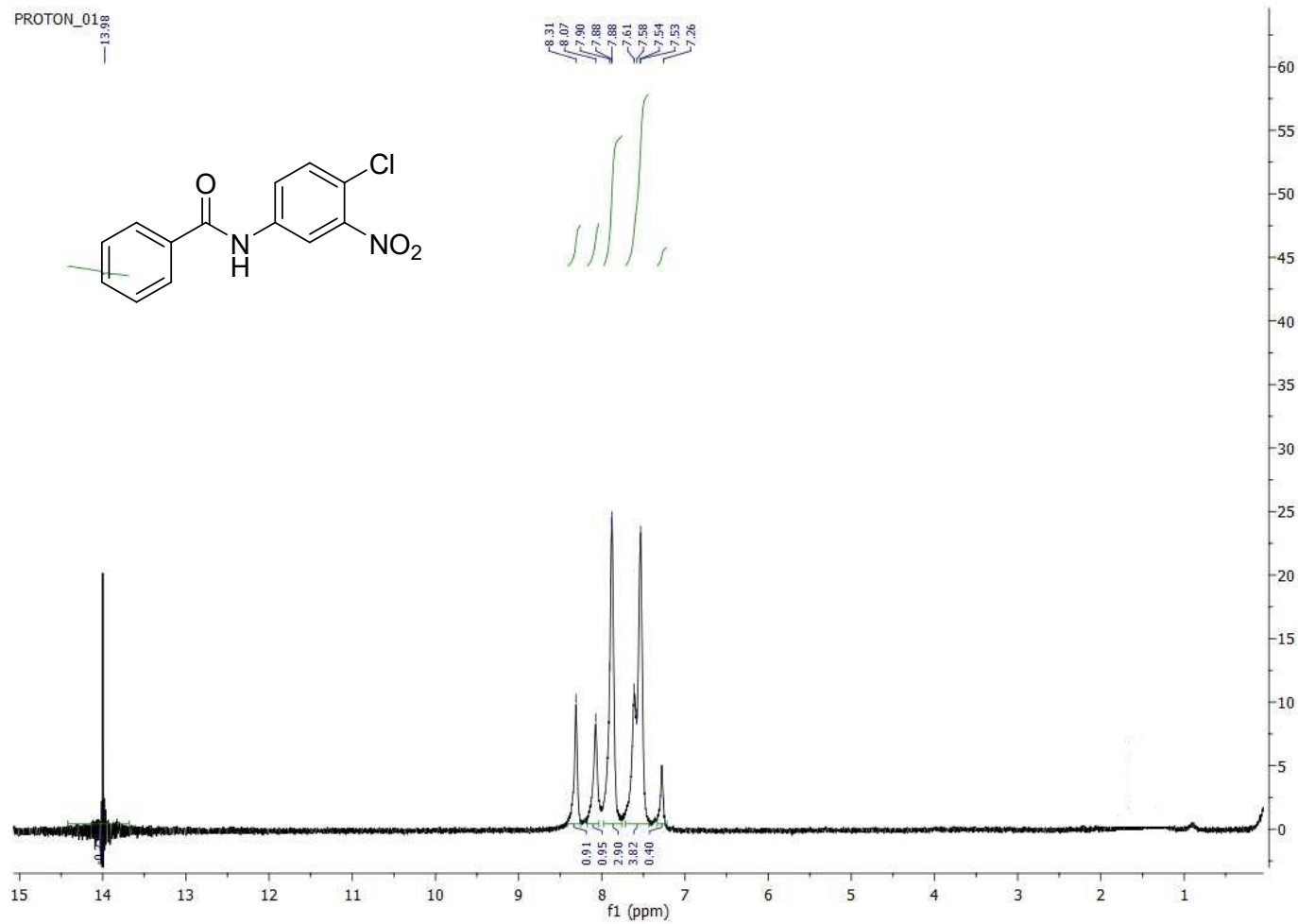
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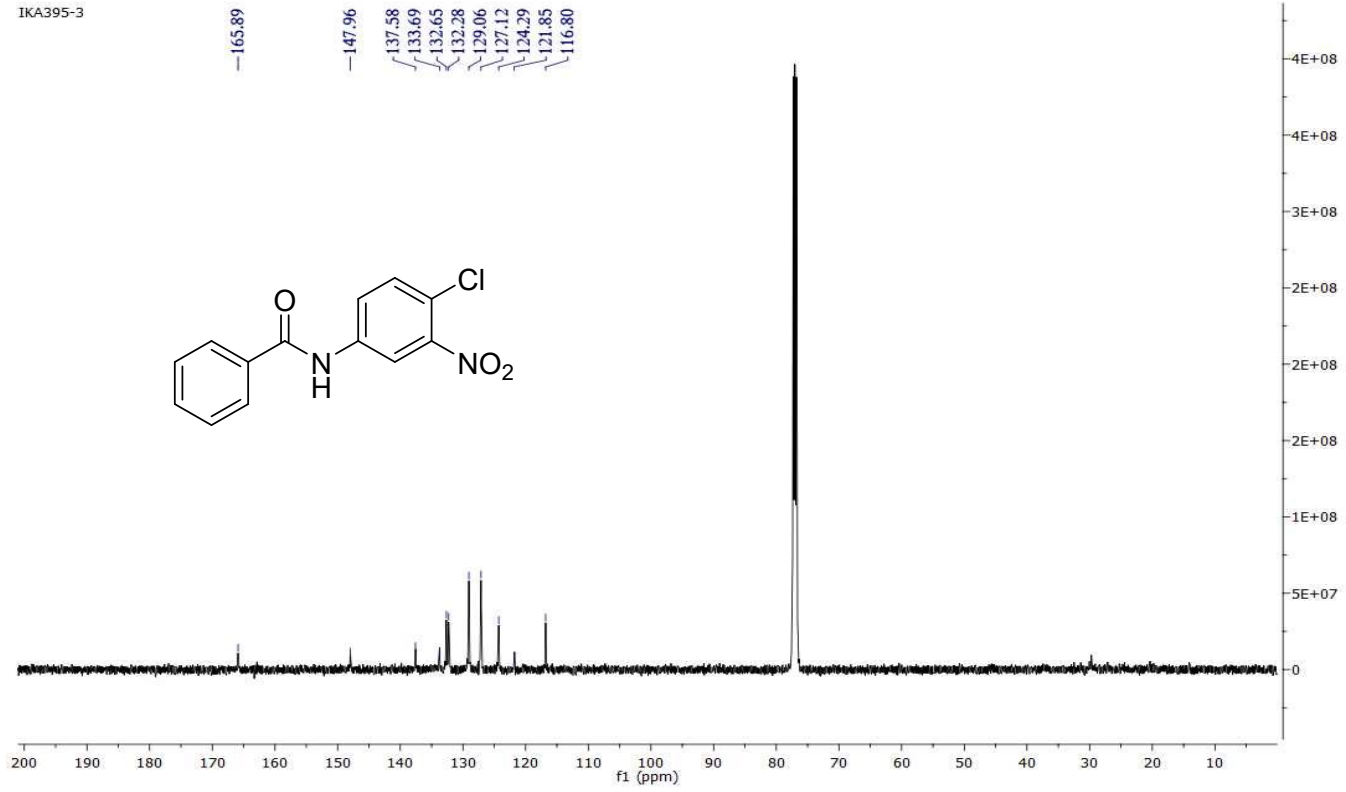
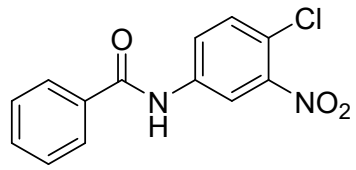
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^1H NMR, ^{13}C NMR, mass and IR spectrum of *N*-(4-chloro-3-nitrophenyl)benzamide (3k)

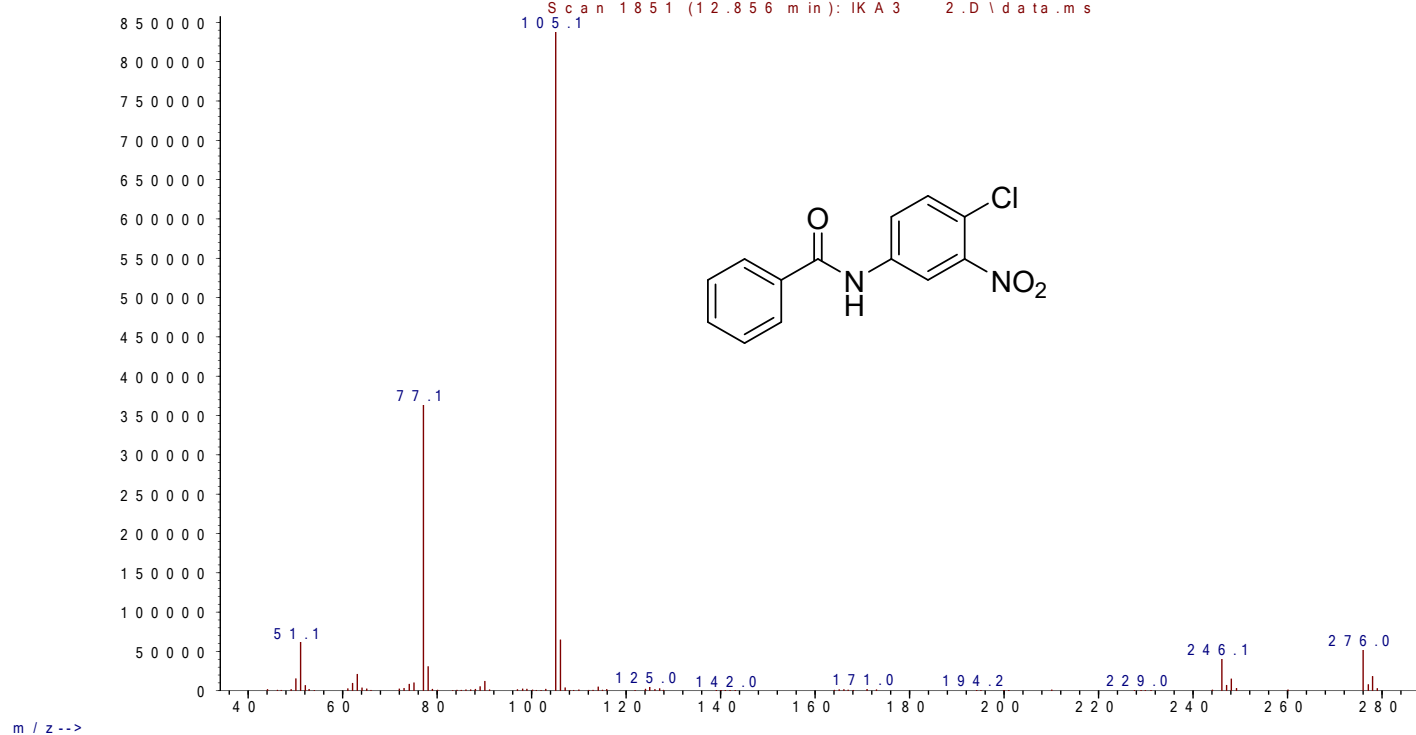
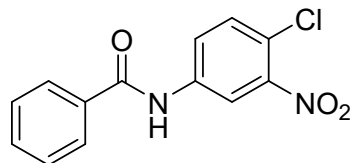


IKA395-3



Abundance

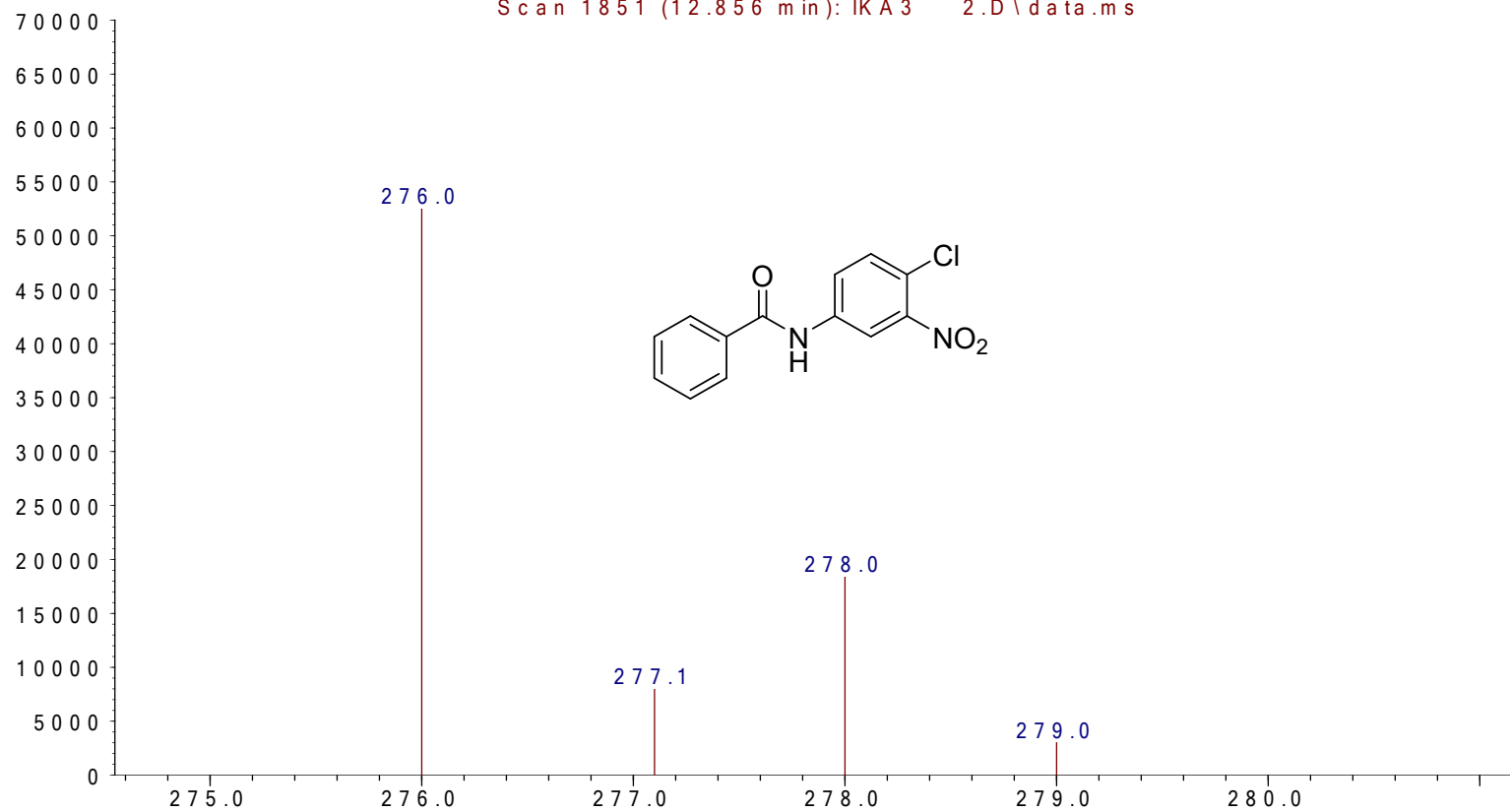
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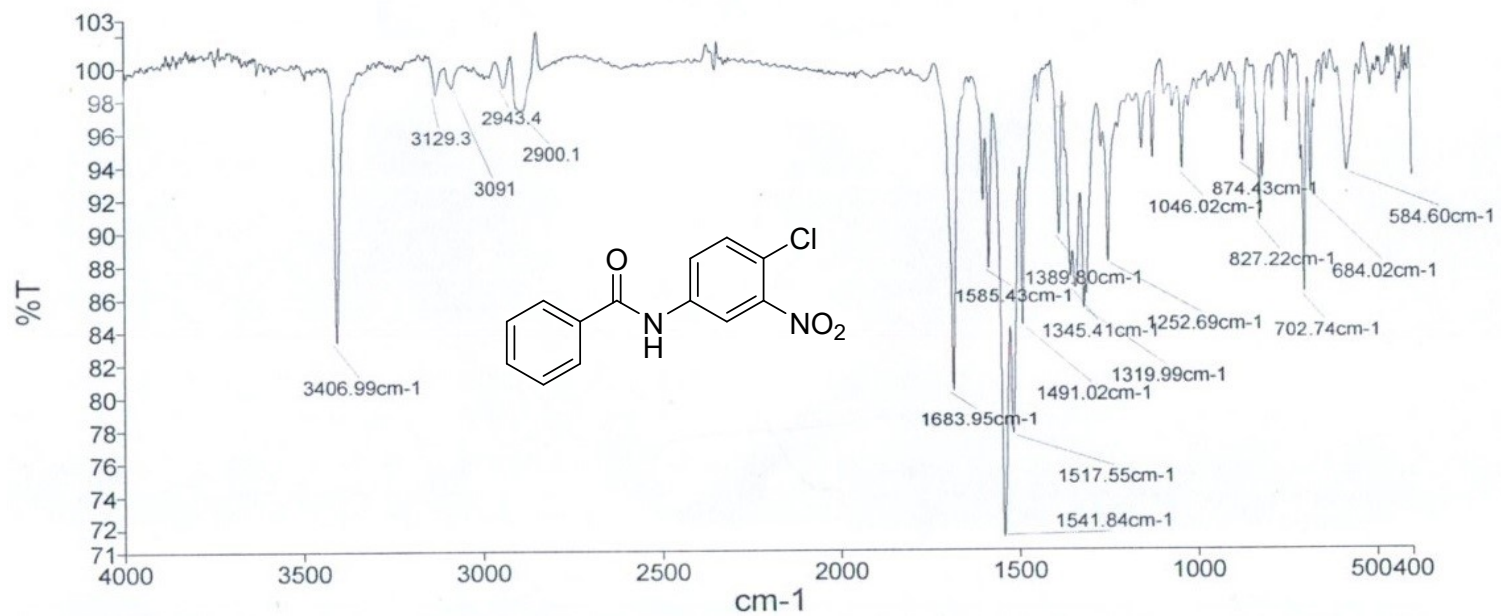
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Abundance

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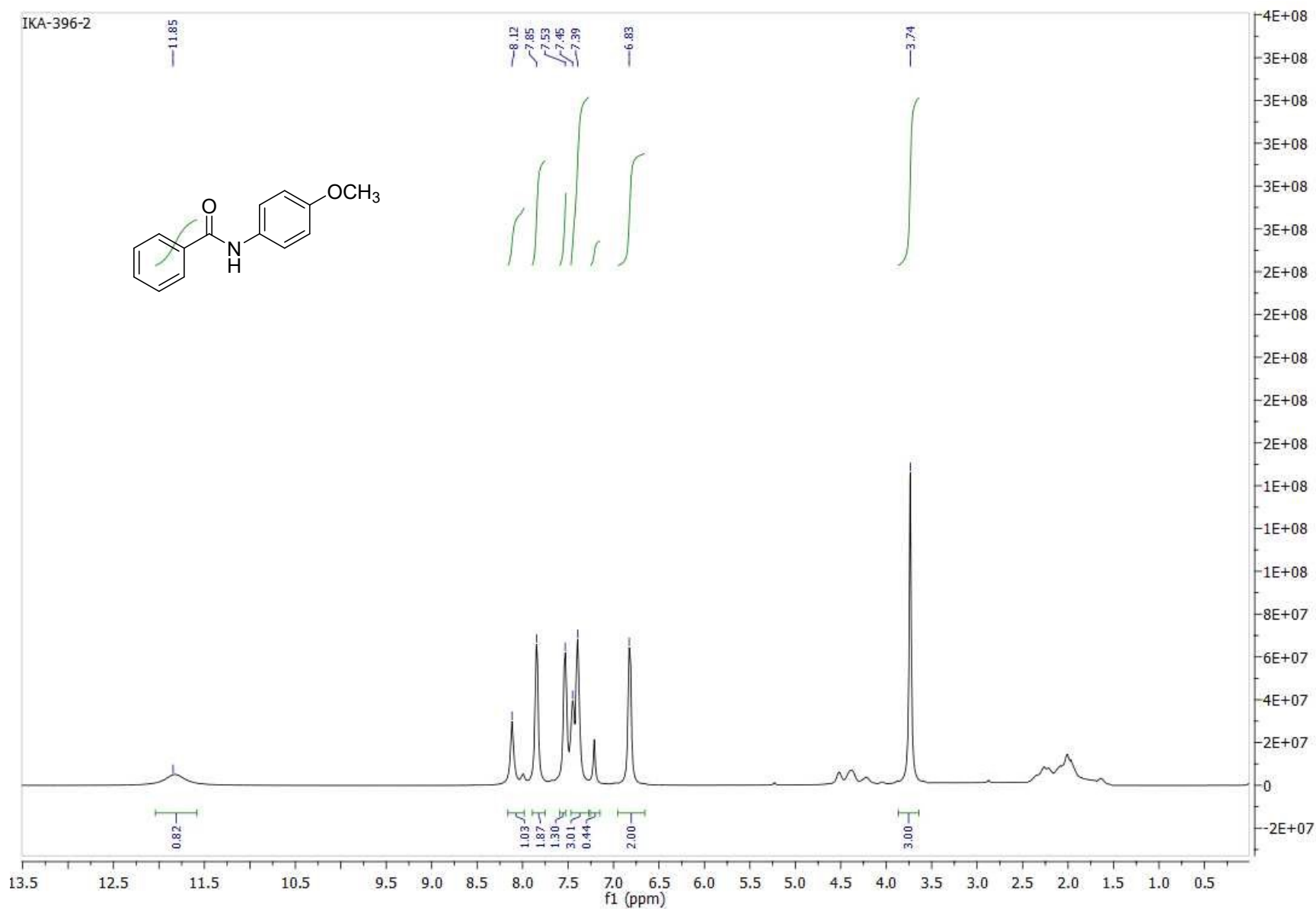
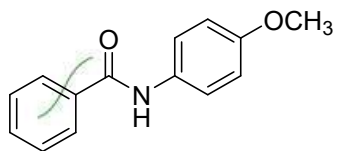


m / z-->

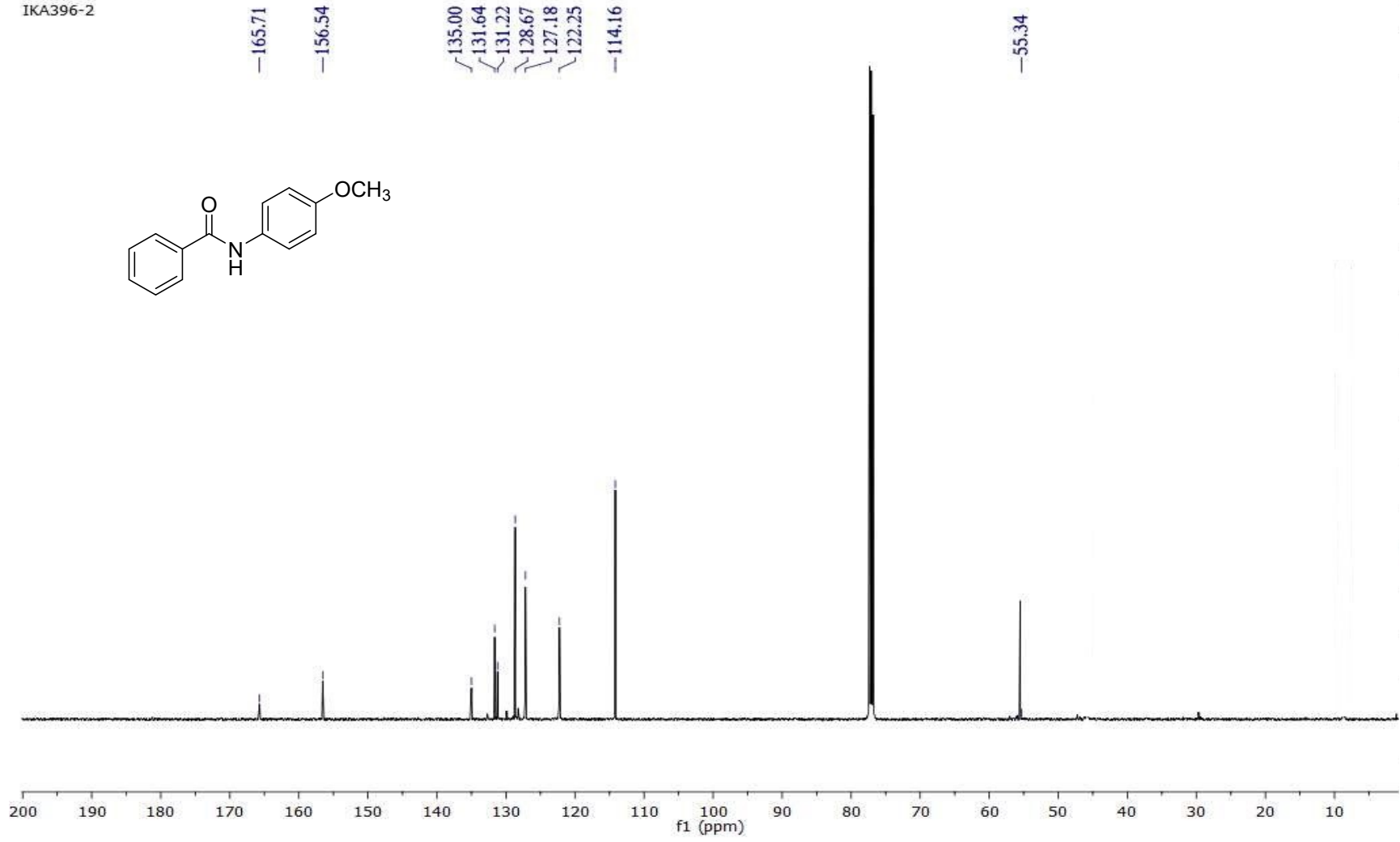
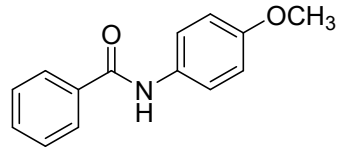


¹H NMR, ¹³C NMR, mass and IR spectrum of *N*-(4-methoxyphenyl)benzamide (3d)

IKA-396-2

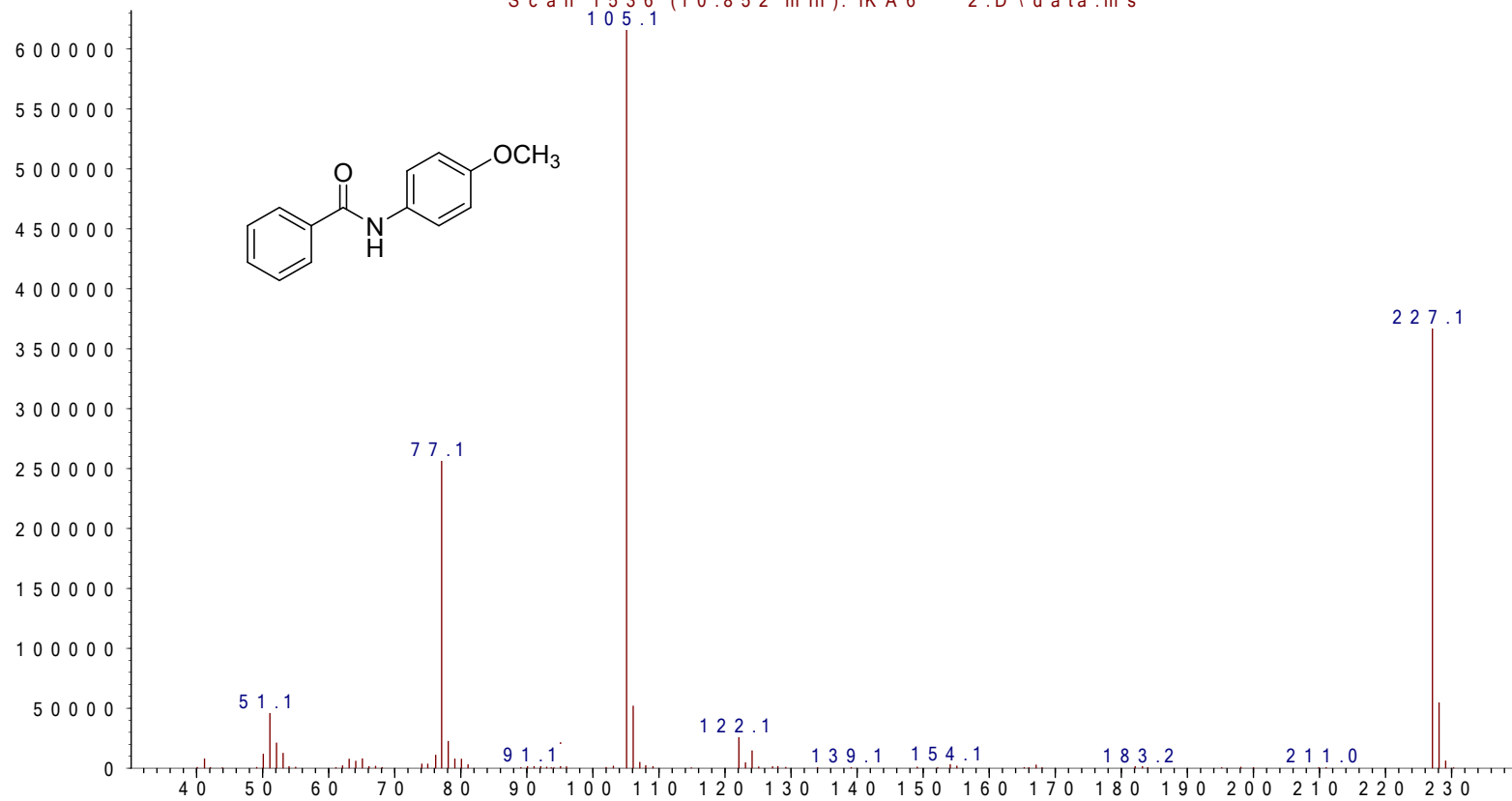
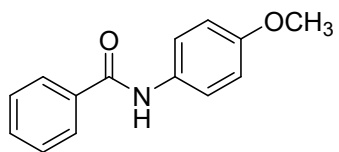


IKA396-2



Abundance

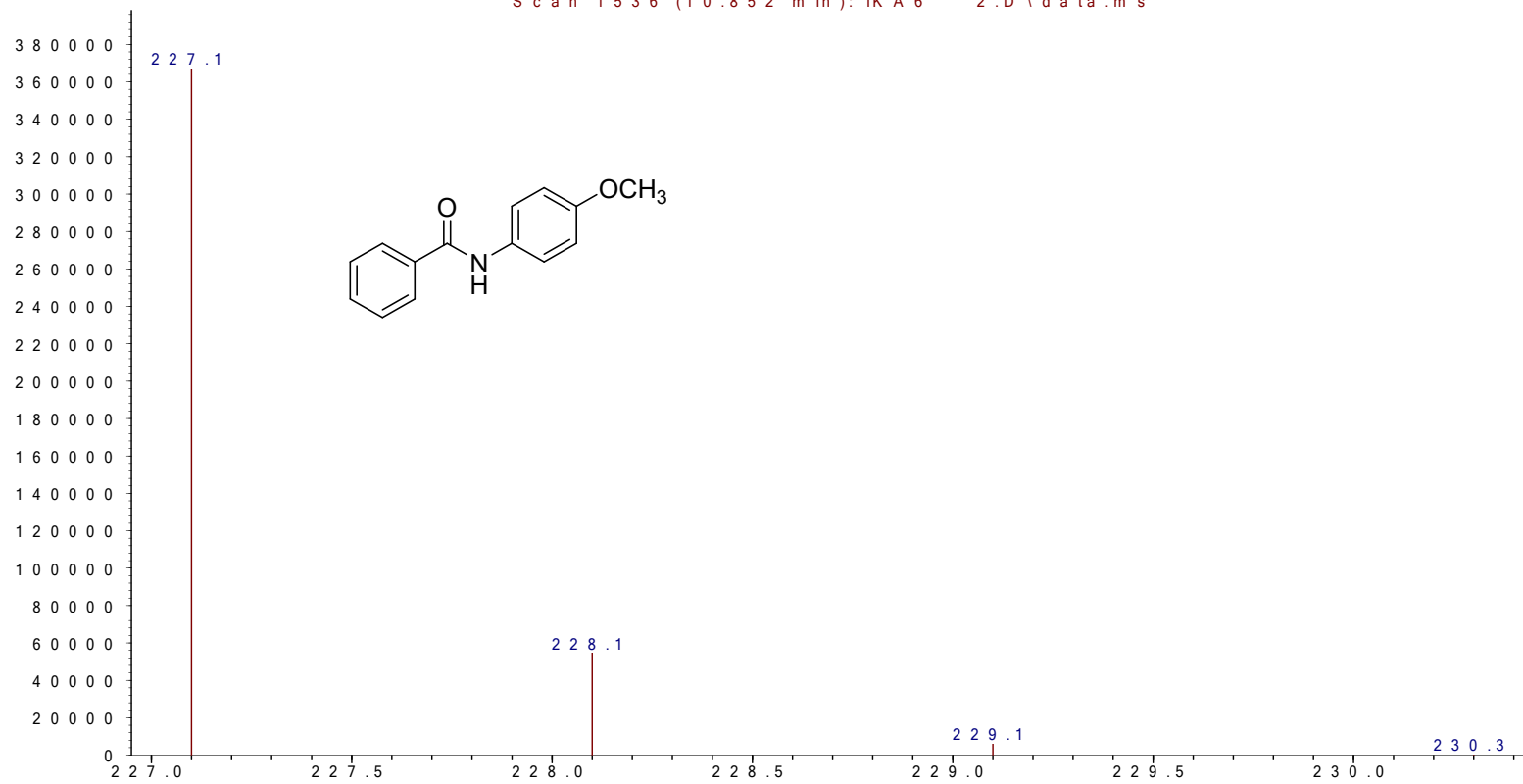
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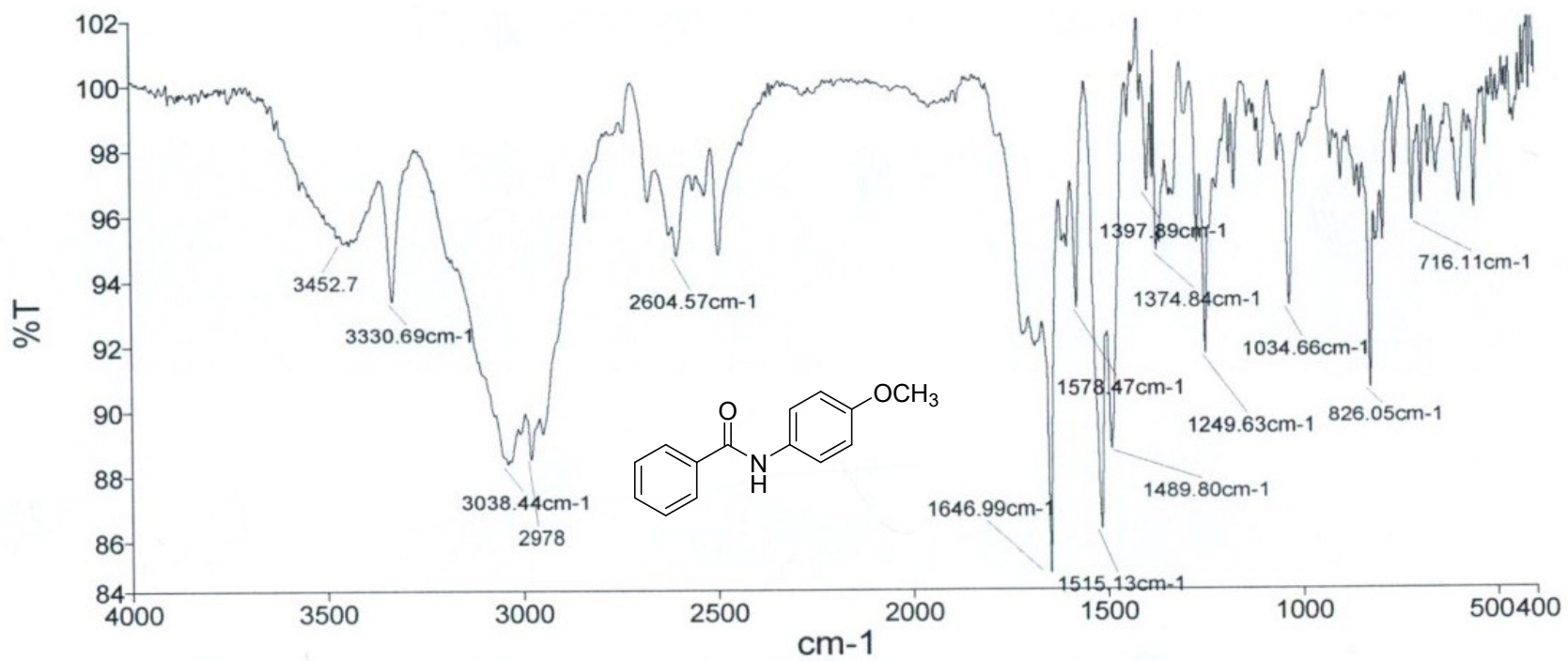
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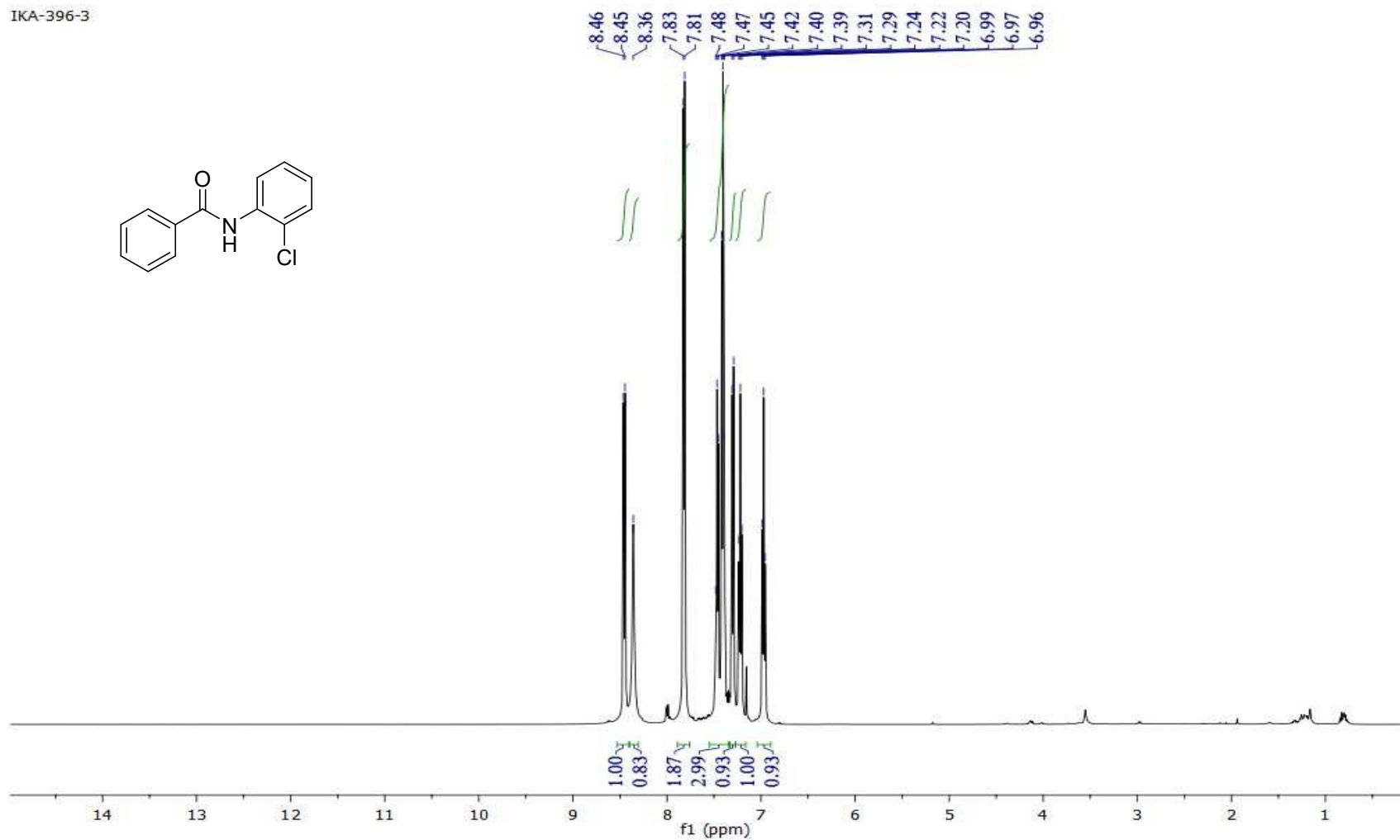
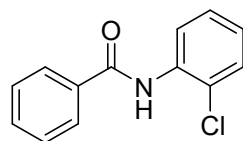


m / z-->

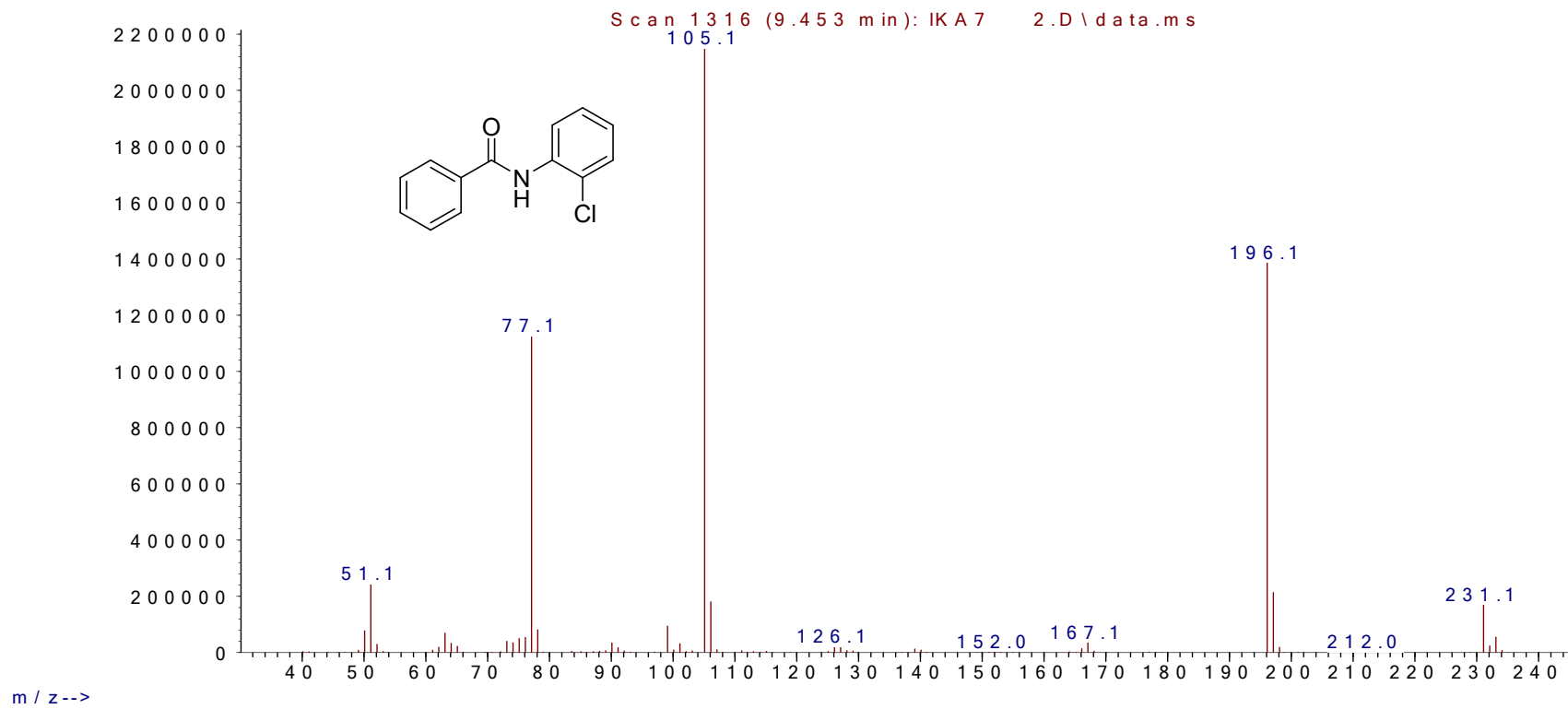


¹H NMR, mass and IR spectrum of *N*-(2-chlorophenyl)benzamide (3h)

IKA-396-3

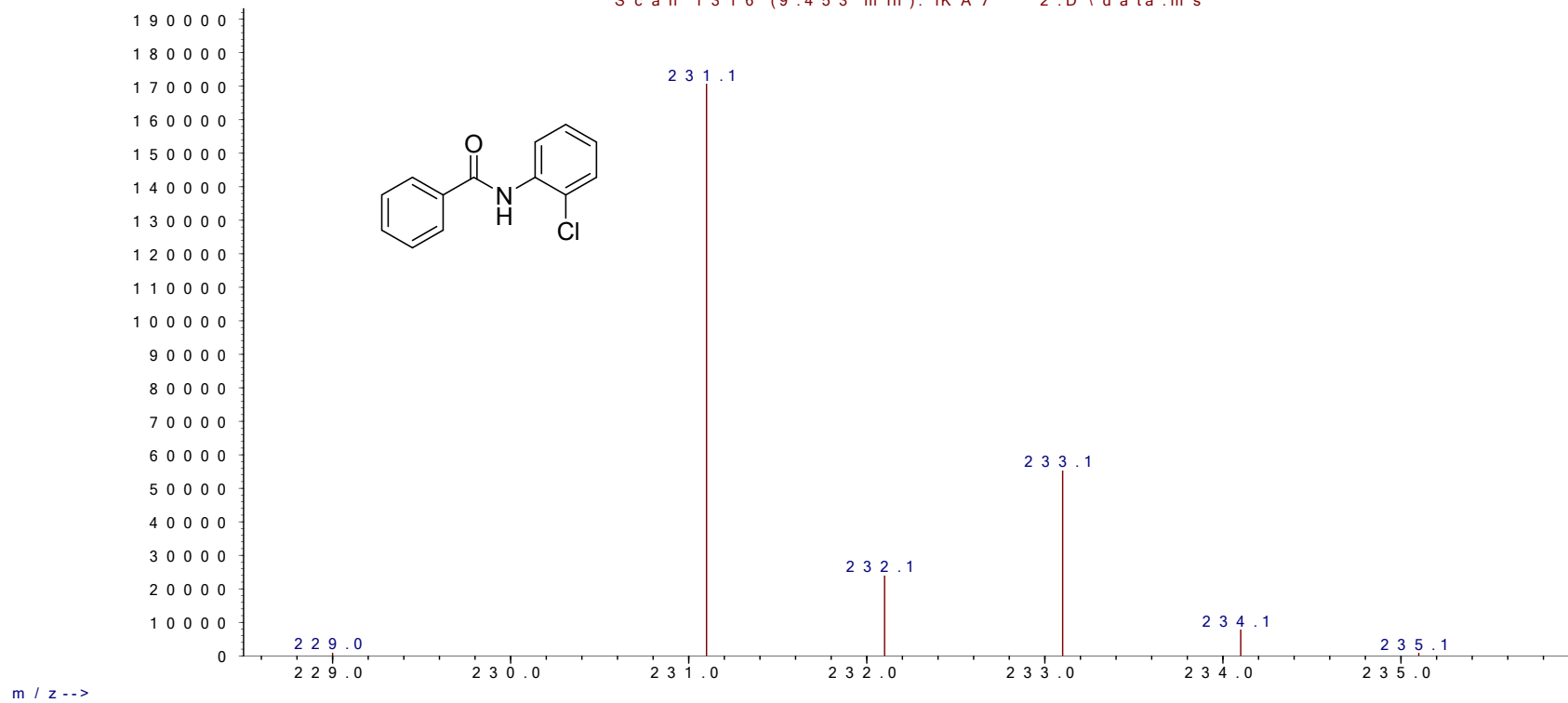
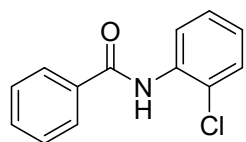


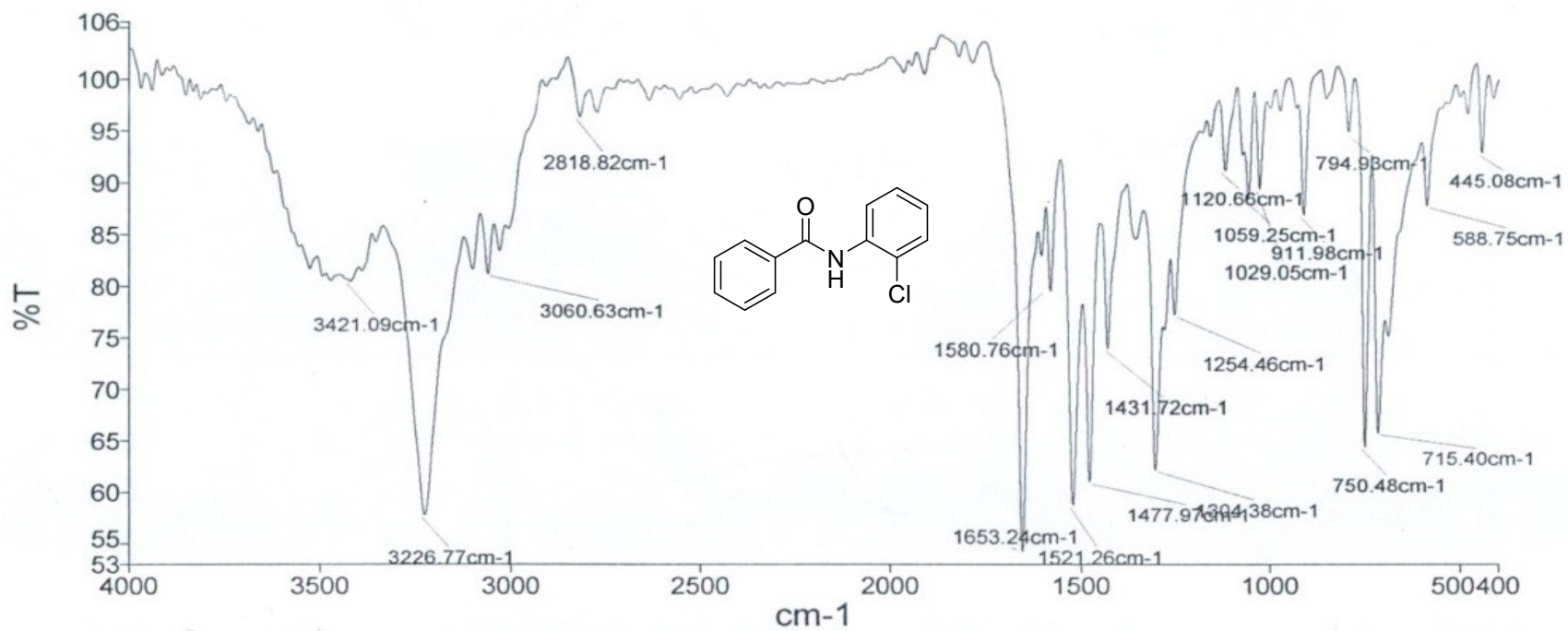
Abundance



Abundance

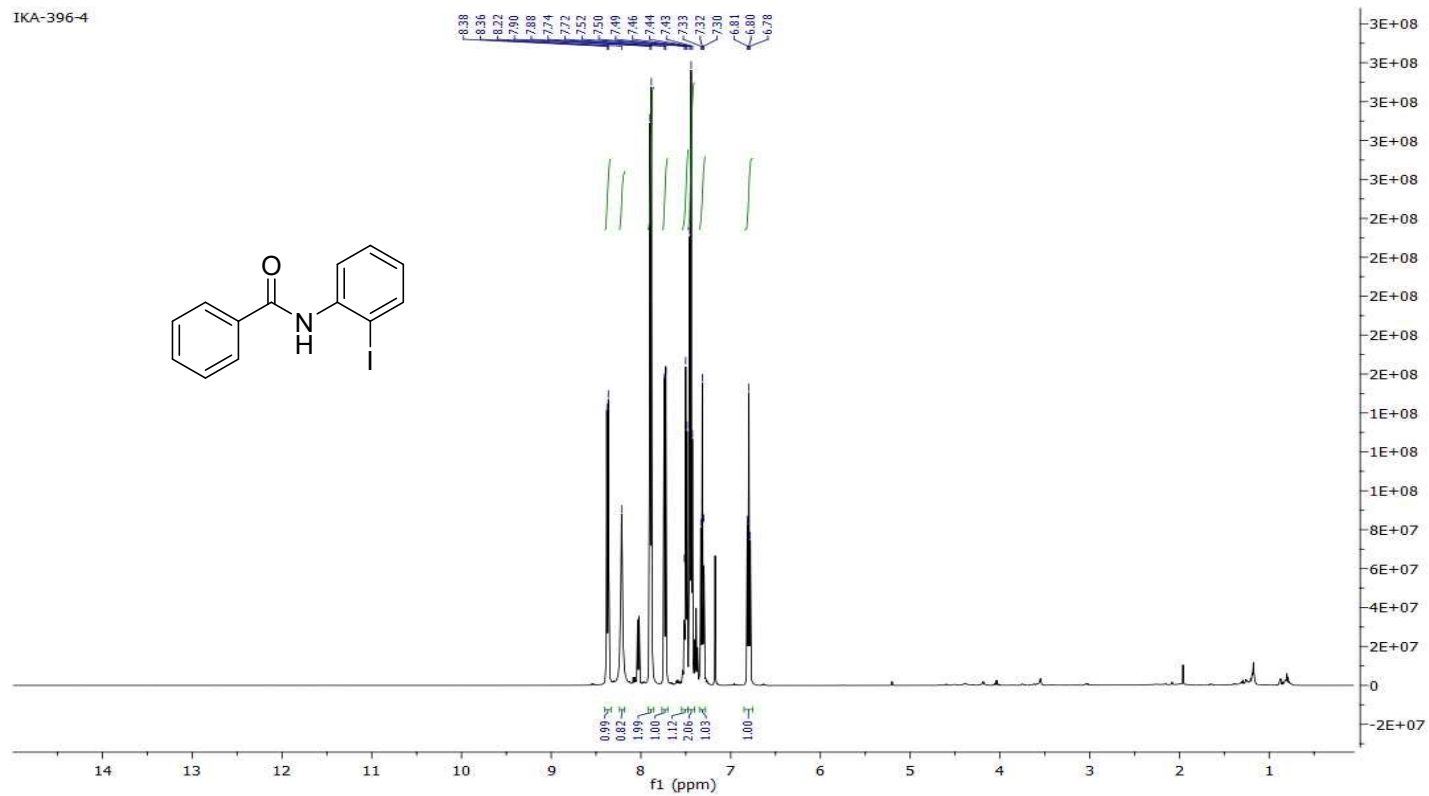
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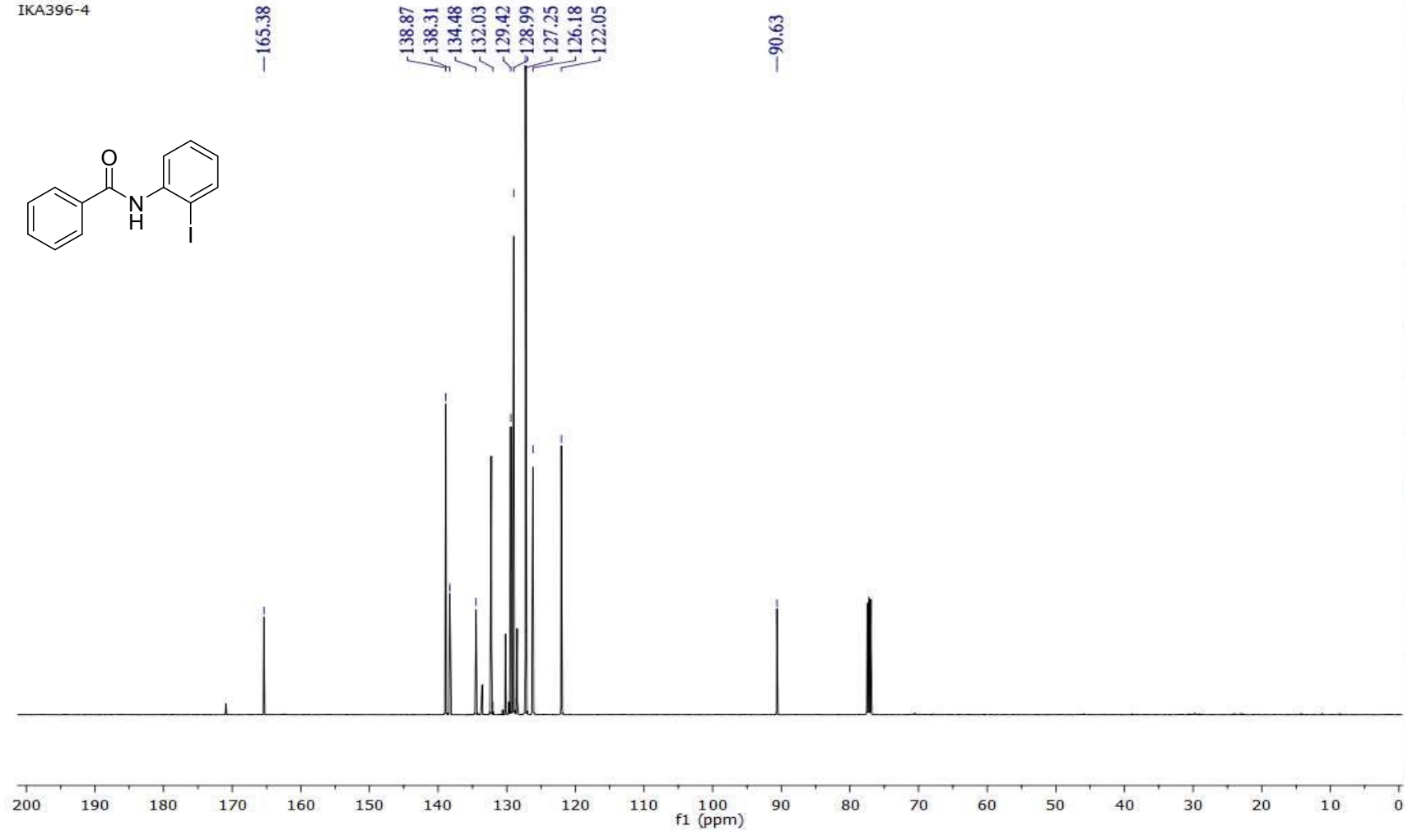
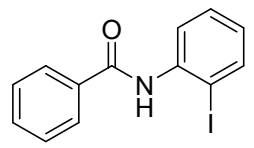


^1H NMR, ^{13}C NMR, mass and IR spectrum of *N*-(2-iodophenyl)benzamide (3f)

IKA-396-4

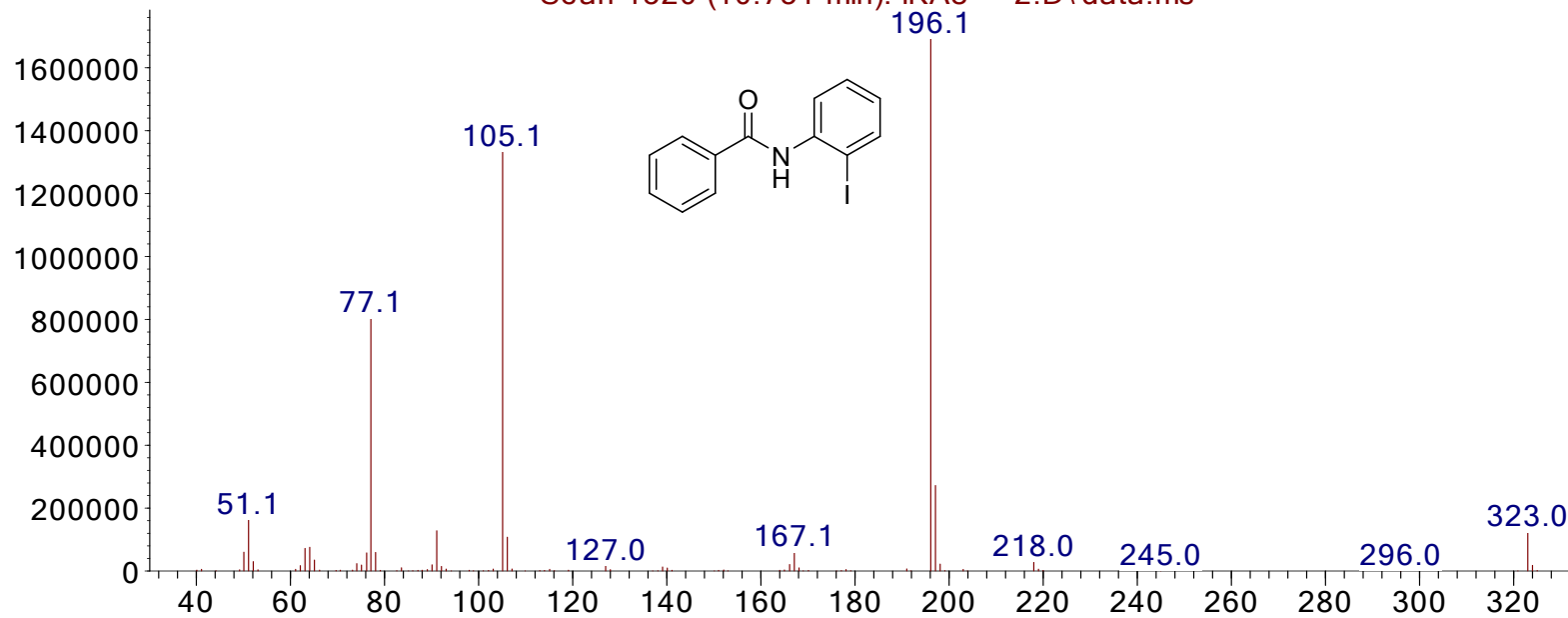


IKA396-4



Abundance

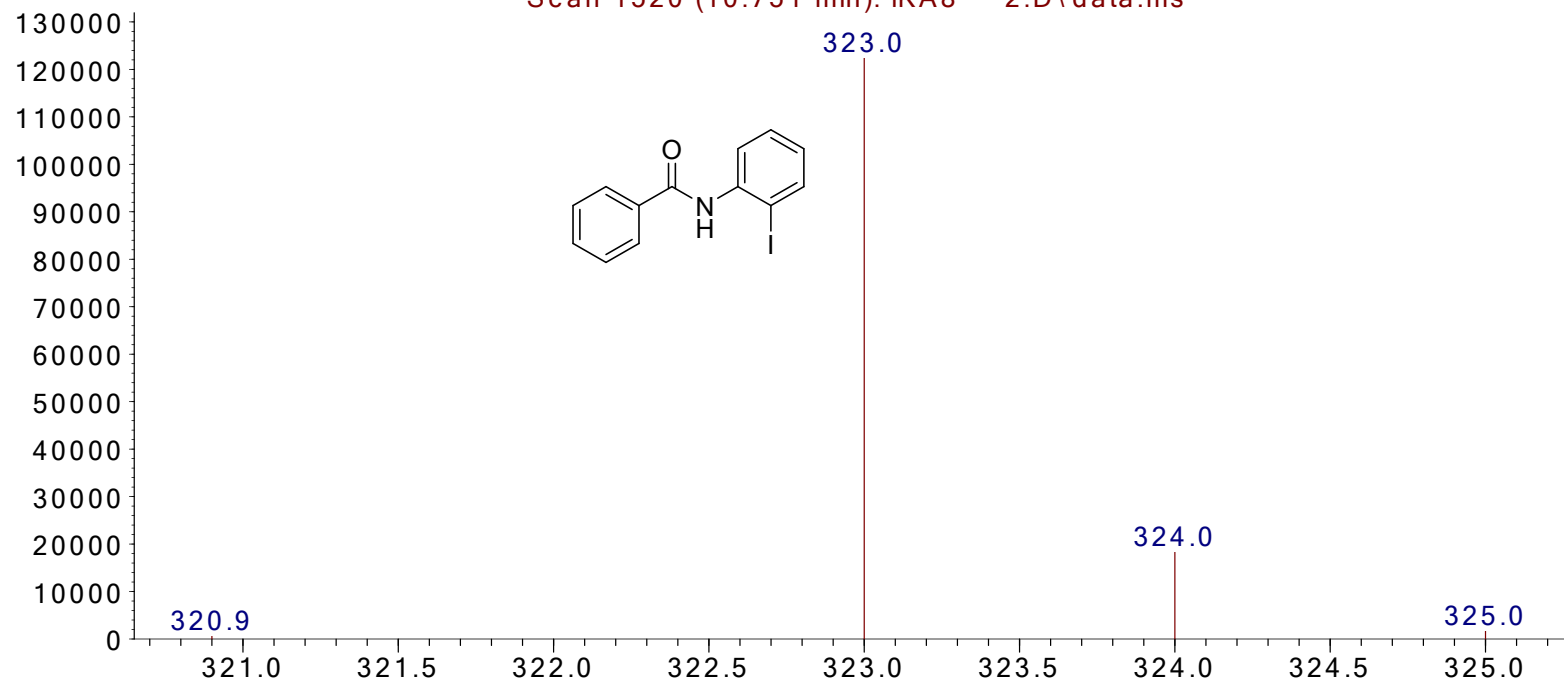
Scan 1520 (10.751 min): IKA8 2.D\data.ms



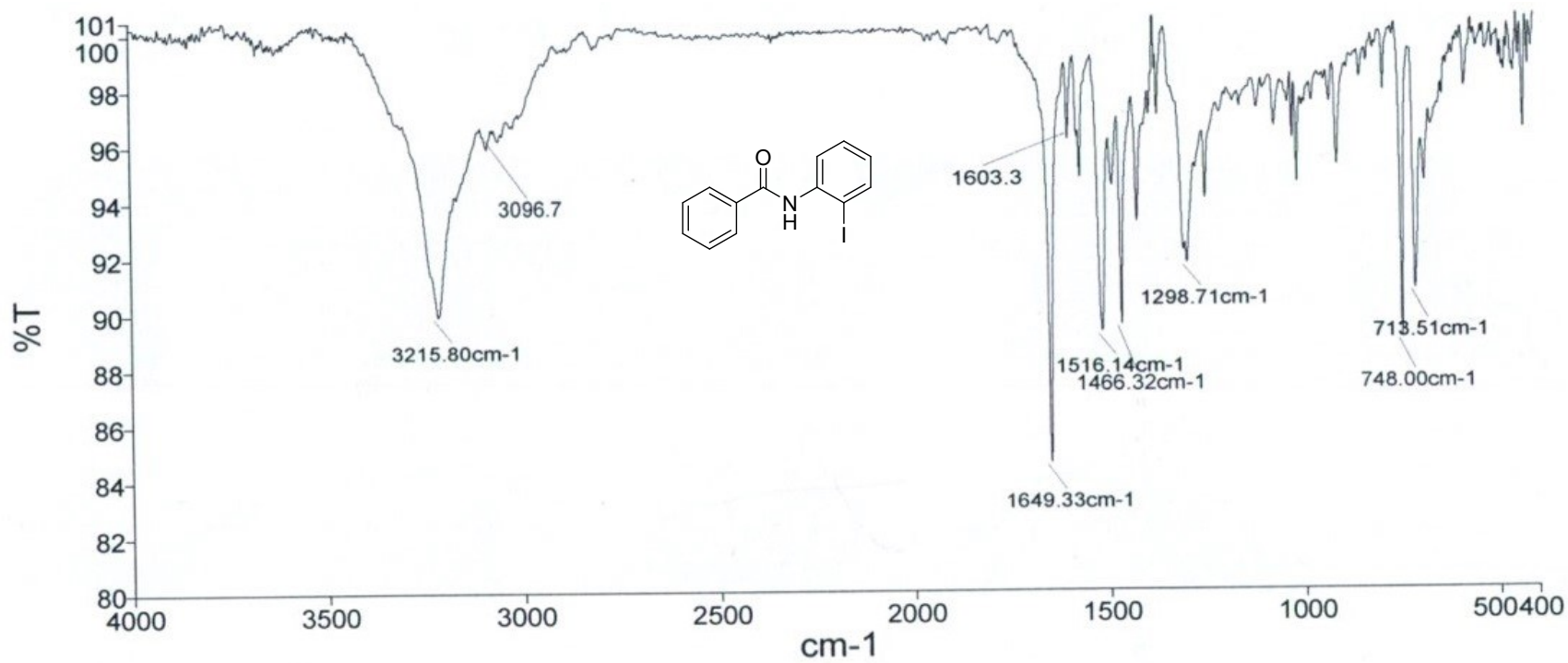
m/z-->

Abundance

Scan 1520 (10.751 min): IKA8 2.D\data.ms

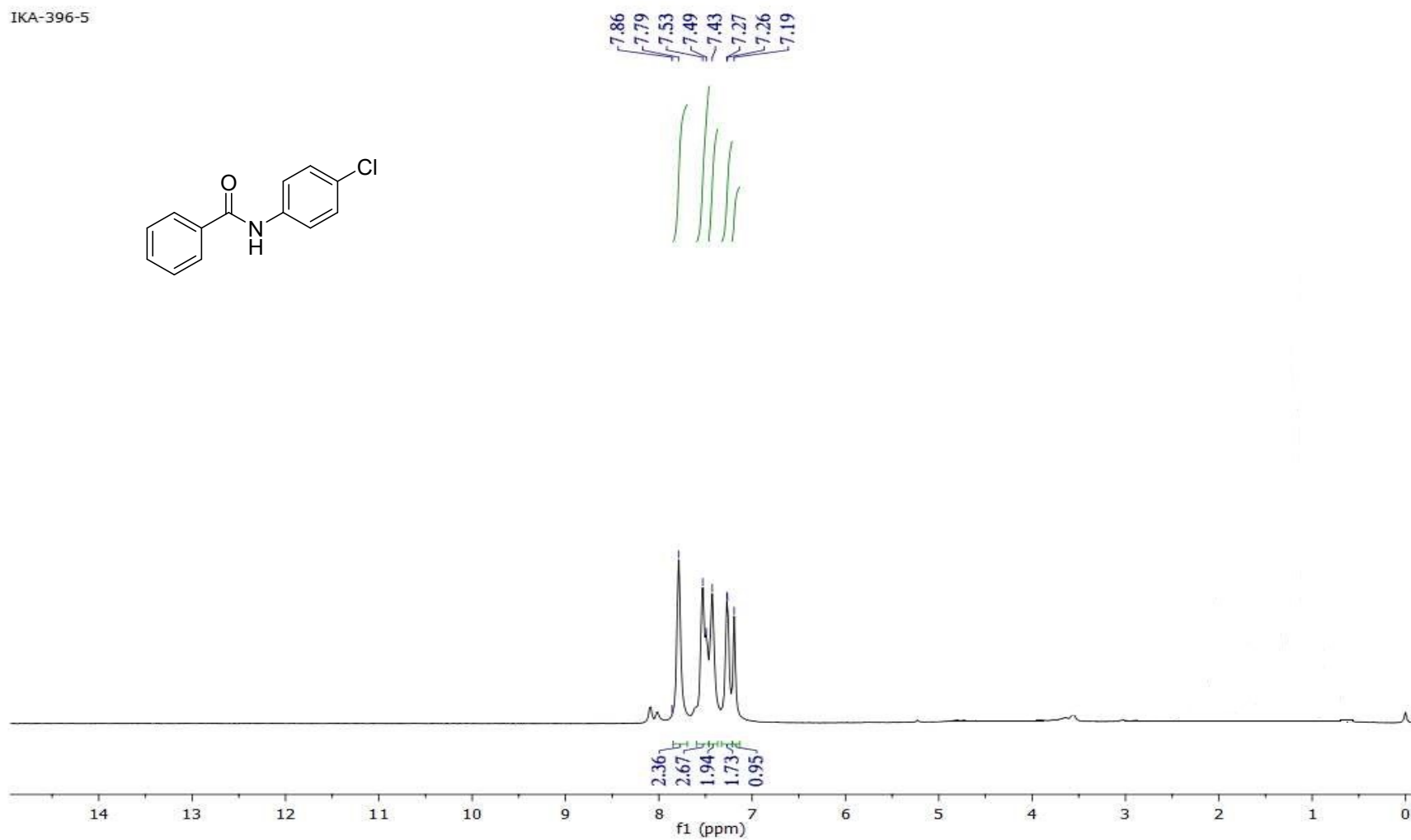
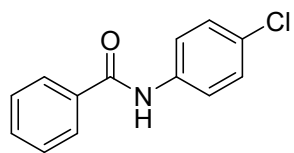


m/z-->



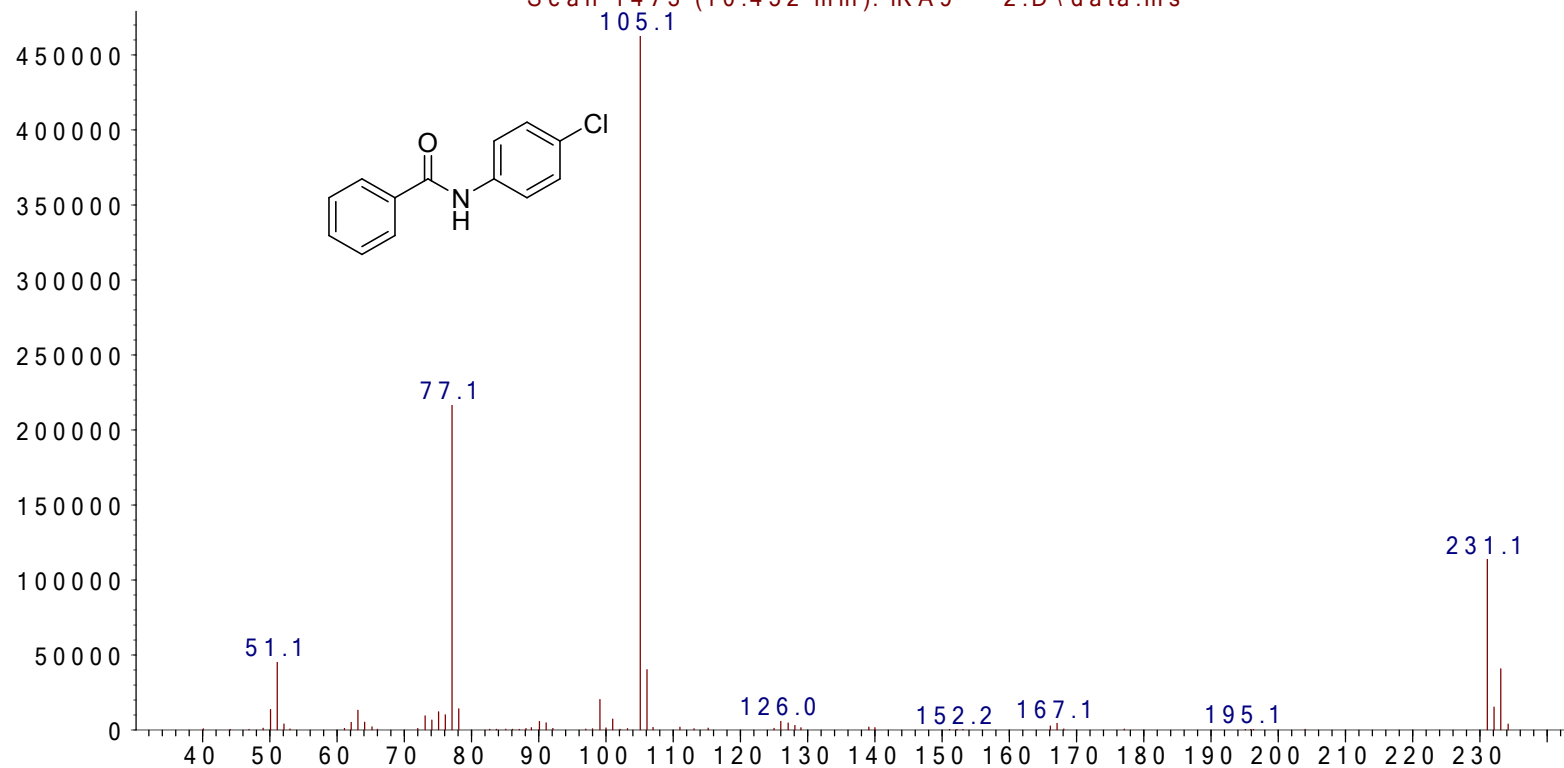
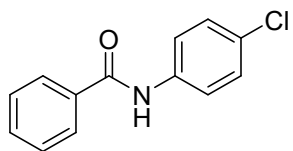
^1H NMR, mass and IR spectrum of *N*-(4-chlorophenyl)benzamide (3j)

IKA-396-5



Abundance

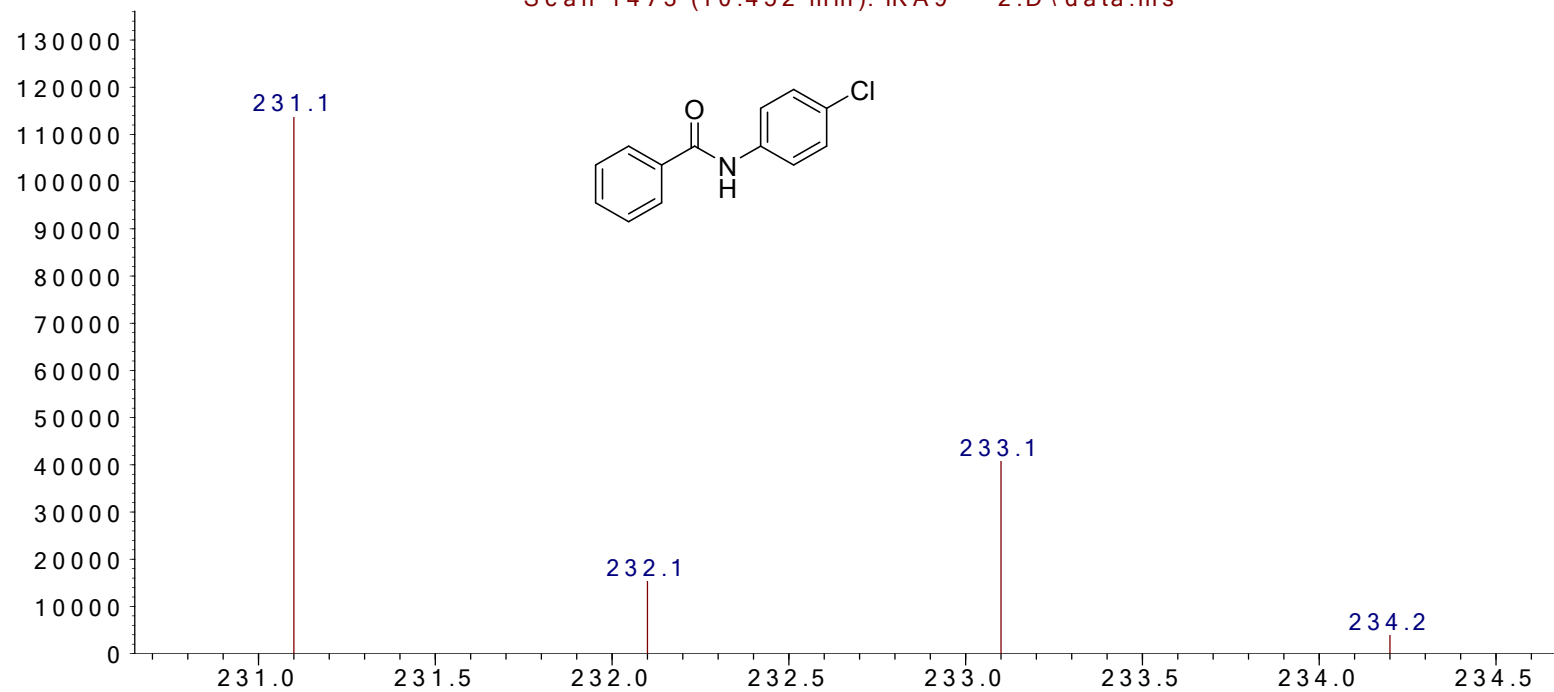
Scan 1473 (10.452 min): IKA9 2.D\data.ms



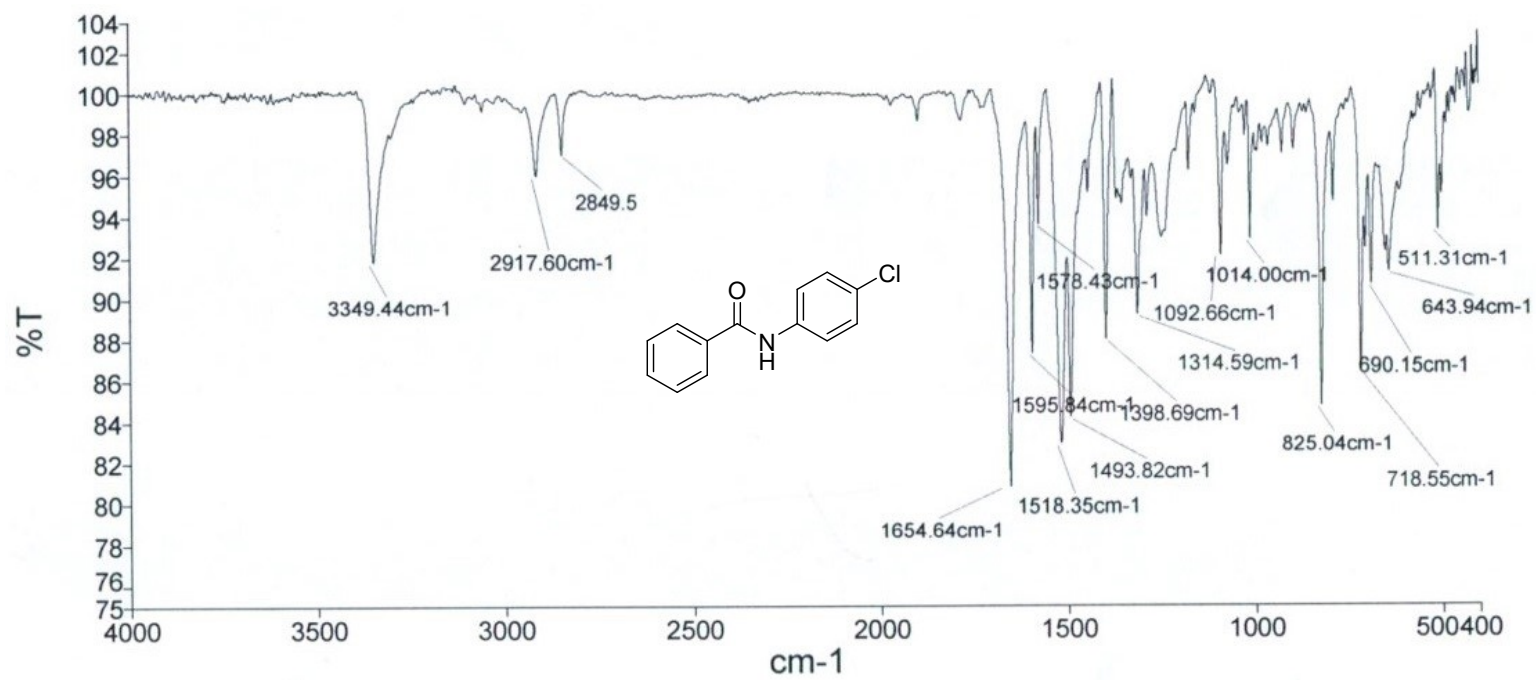
m/z-->

Abundance

Scan 1473 (10.452 min): IKA9 2.D\data.ms

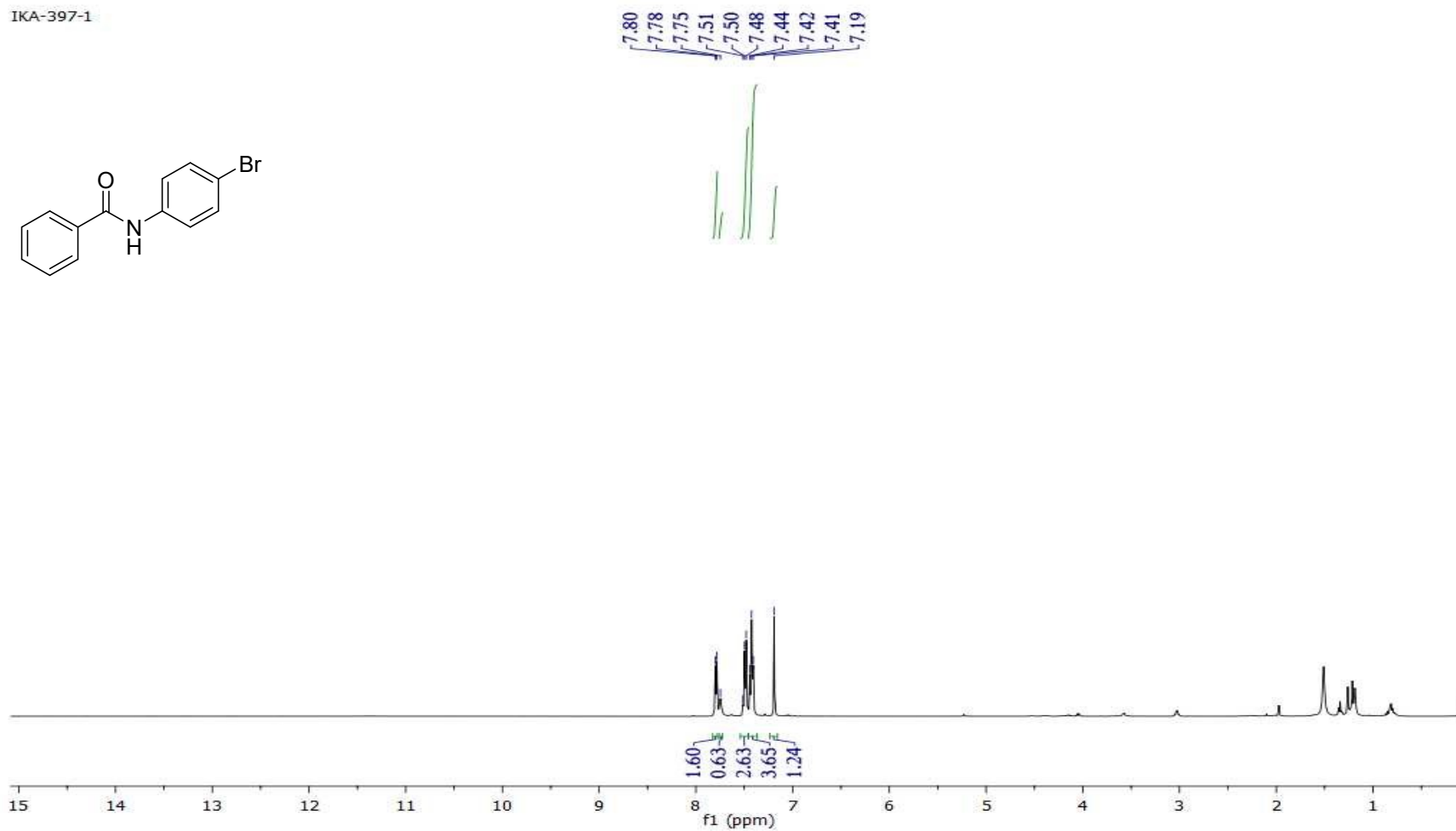


m/z-->

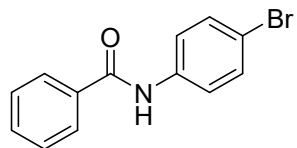


^1H NMR, ^{13}C NMR, mass and IR spectrum of *N*-(4-bromophenyl)benzamide (3i)

IKA-397-1

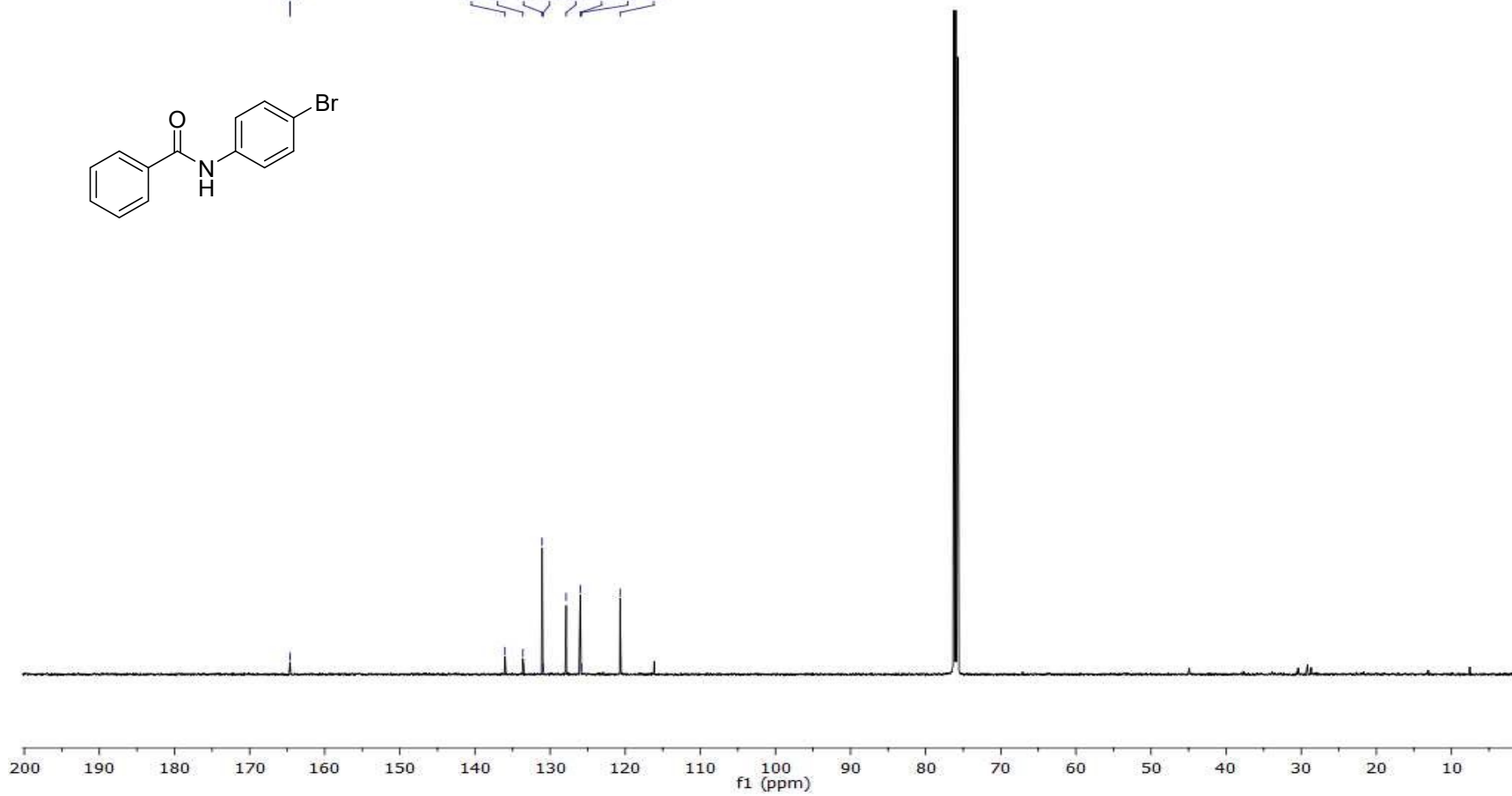


IKA-391-1



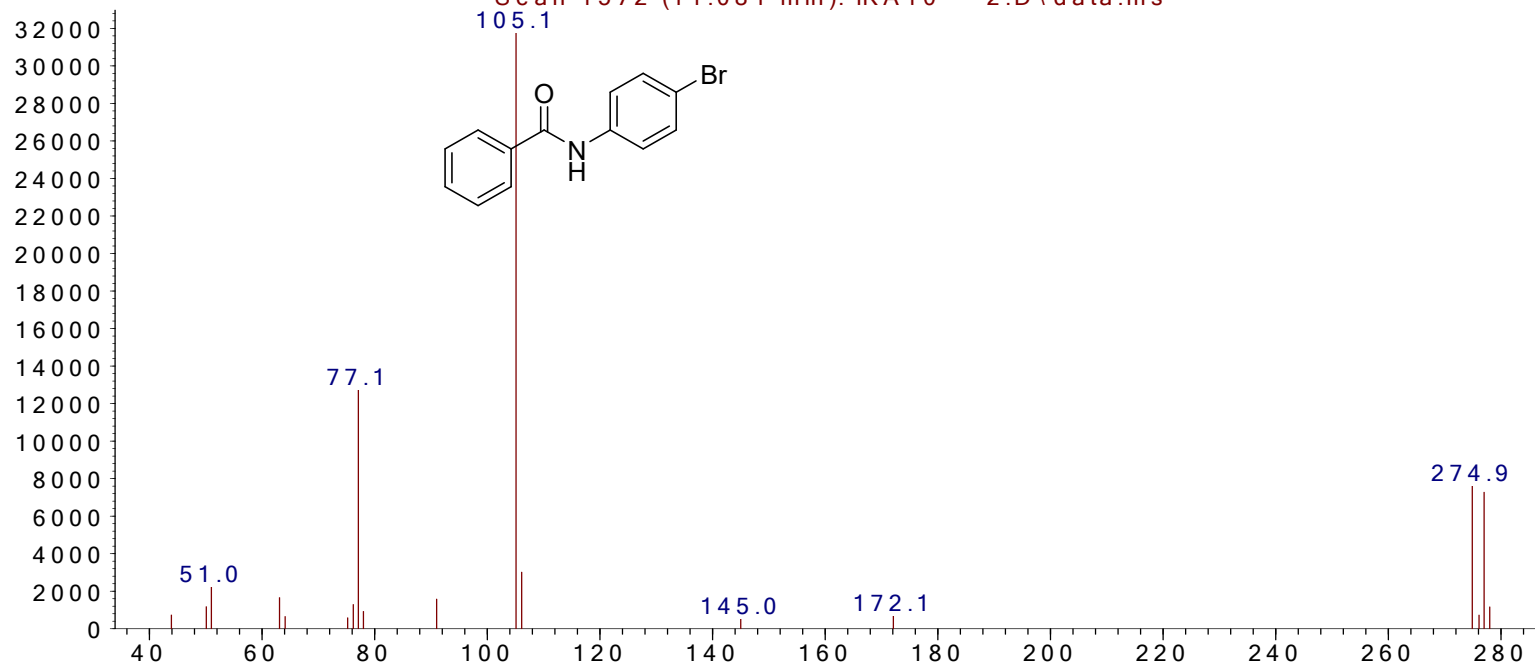
164.62

135.99
133.61
131.06
130.88
127.86
125.98
125.83
120.68



Abundance

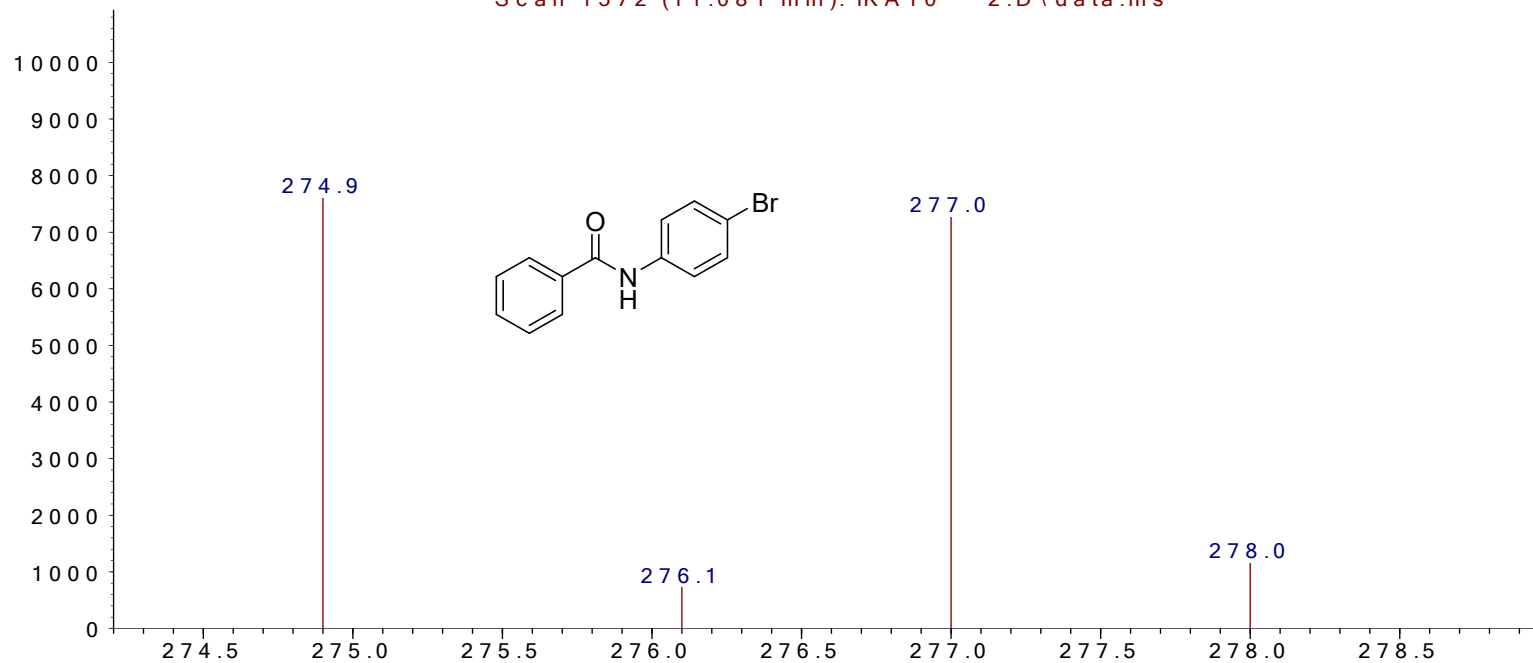
Scan 1572 (11.081 min): IKA10 2.D\data.ms



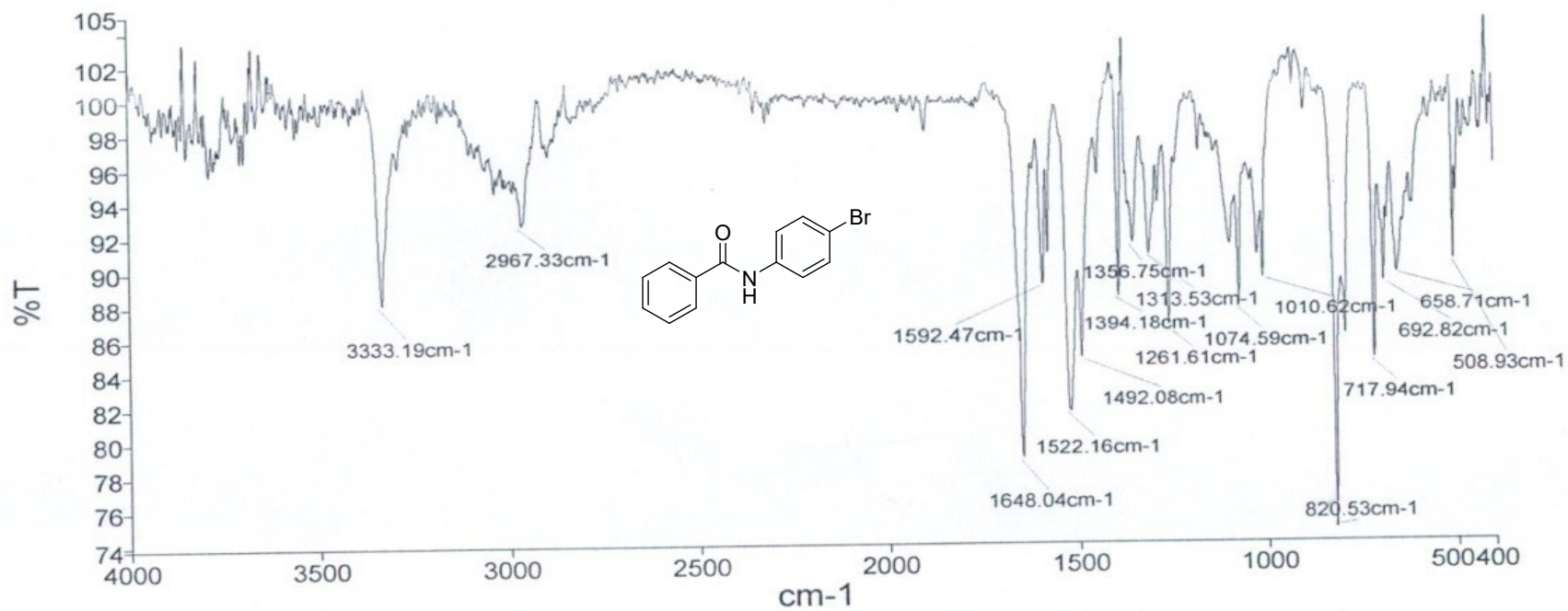
m/z-->

Abundance

Scan 1572 (11.081 min): IKA10 2.D\data.ms

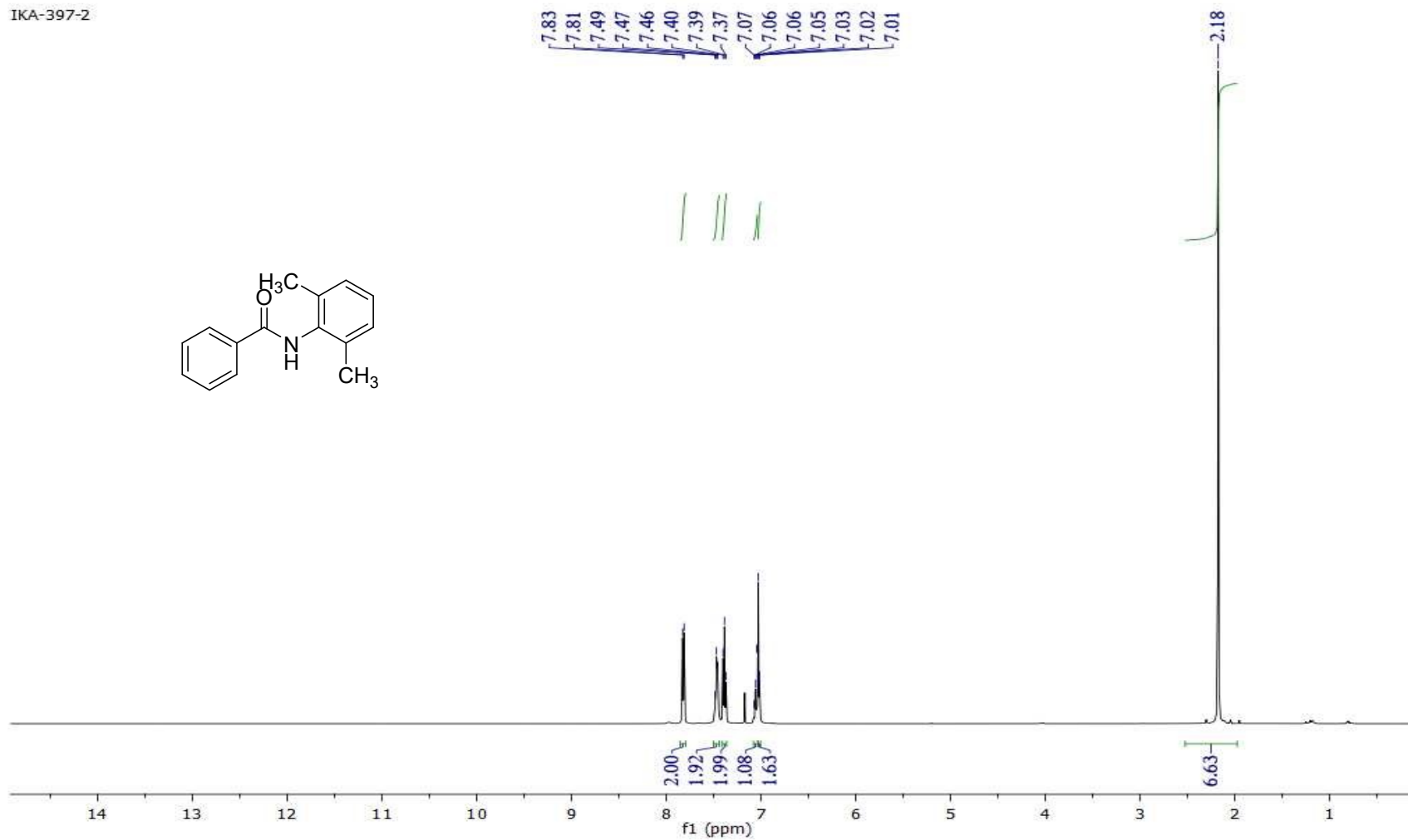


m/z-->

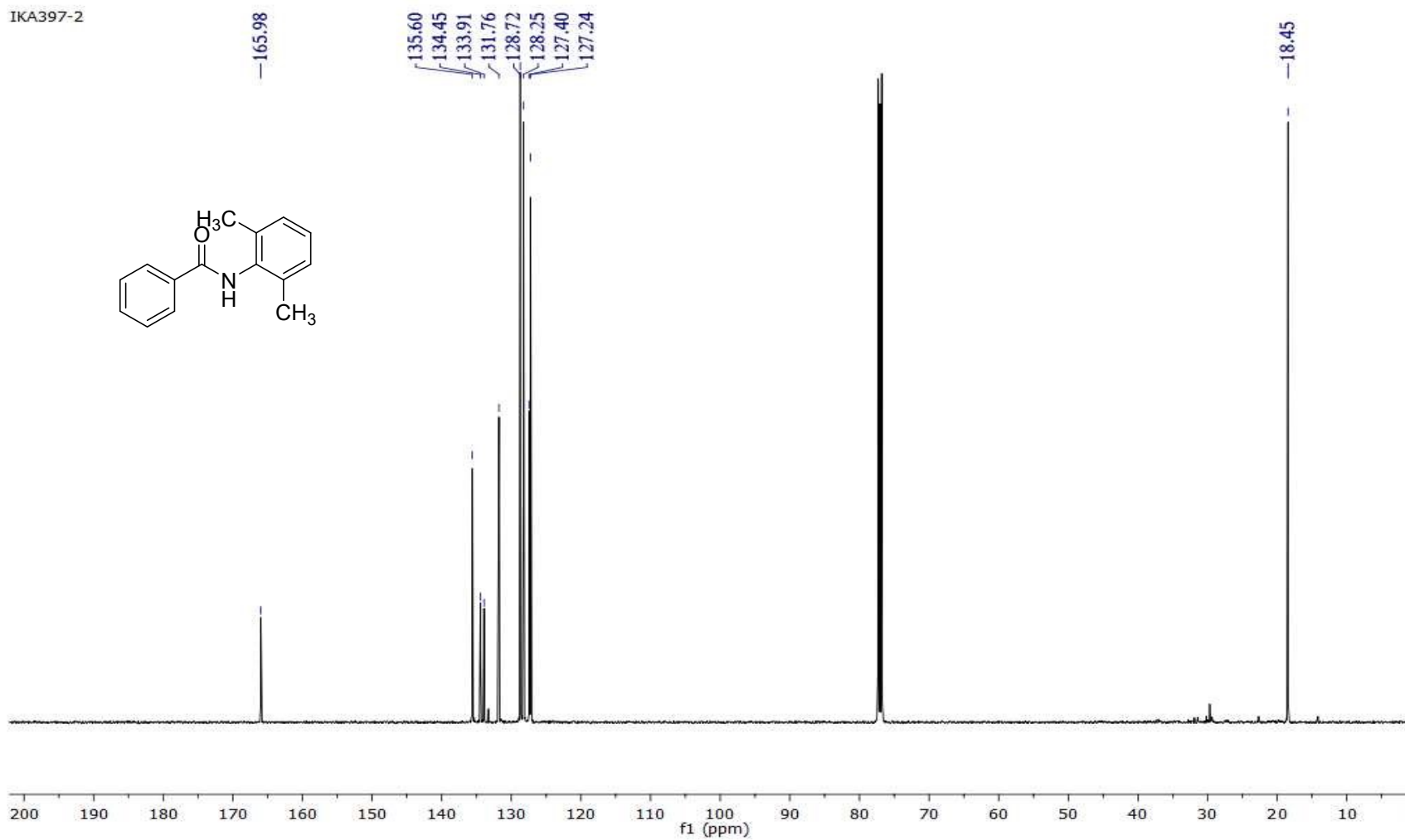
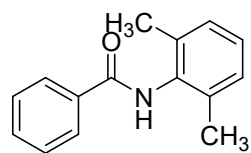


^1H NMR, ^{13}C NMR, mass and IR spectrum of *N*-(2,6-dimethylphenyl)benzamide (3n)

IKA-397-2

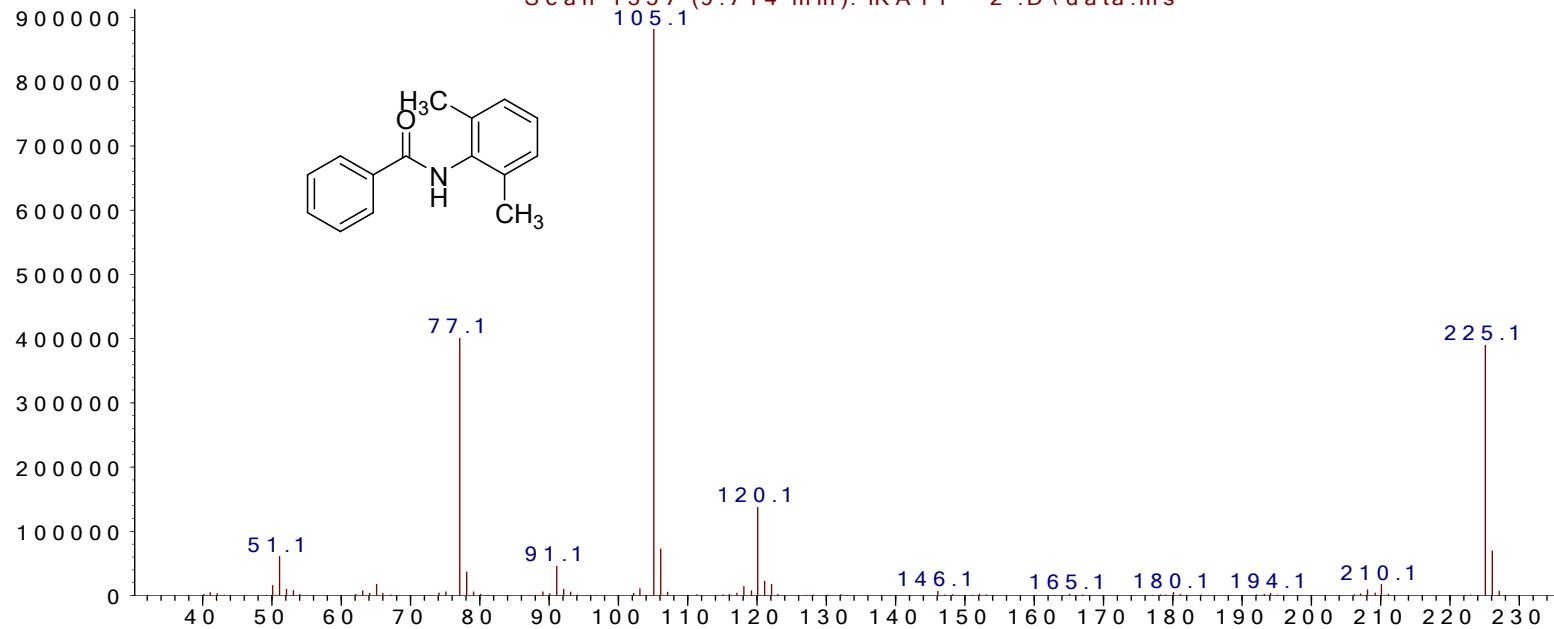
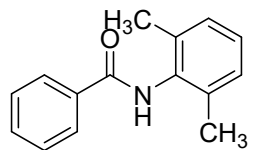


IKA397-2



Abundance

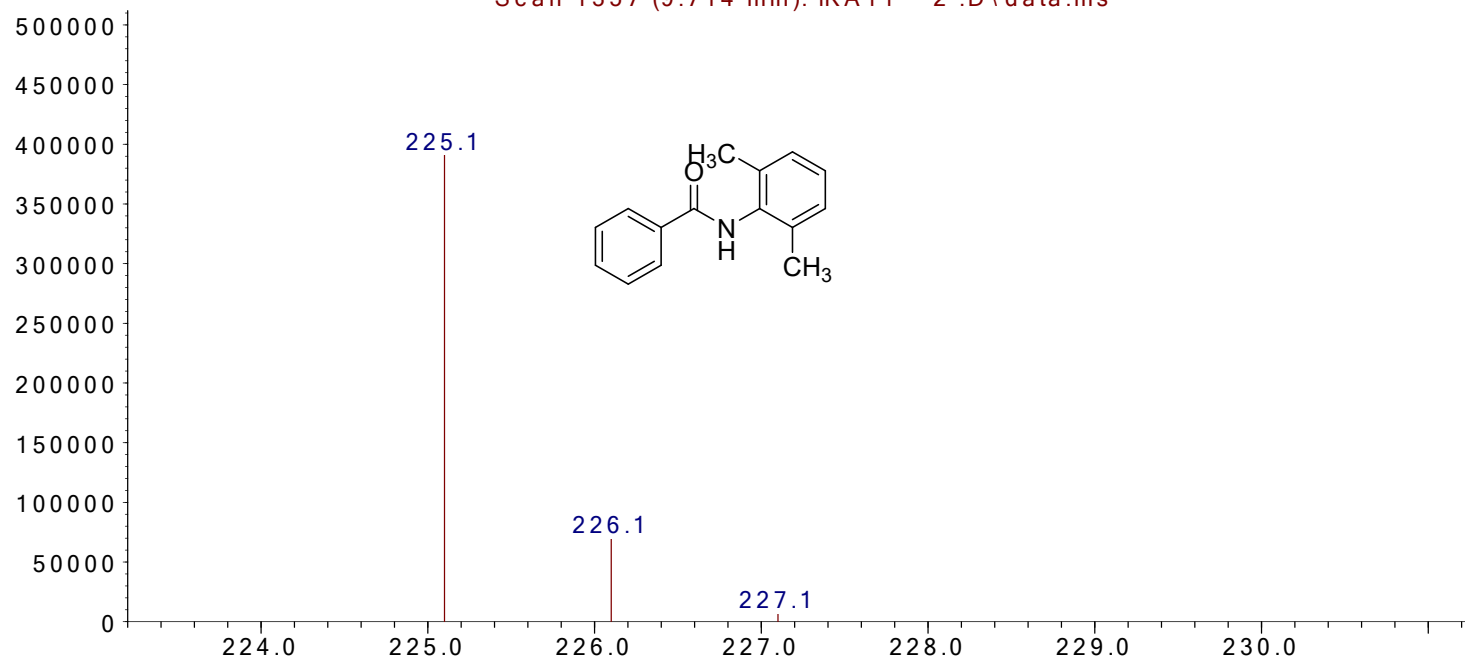
Scan 1357 (9.714 min): IKA11 2.D\data.ms



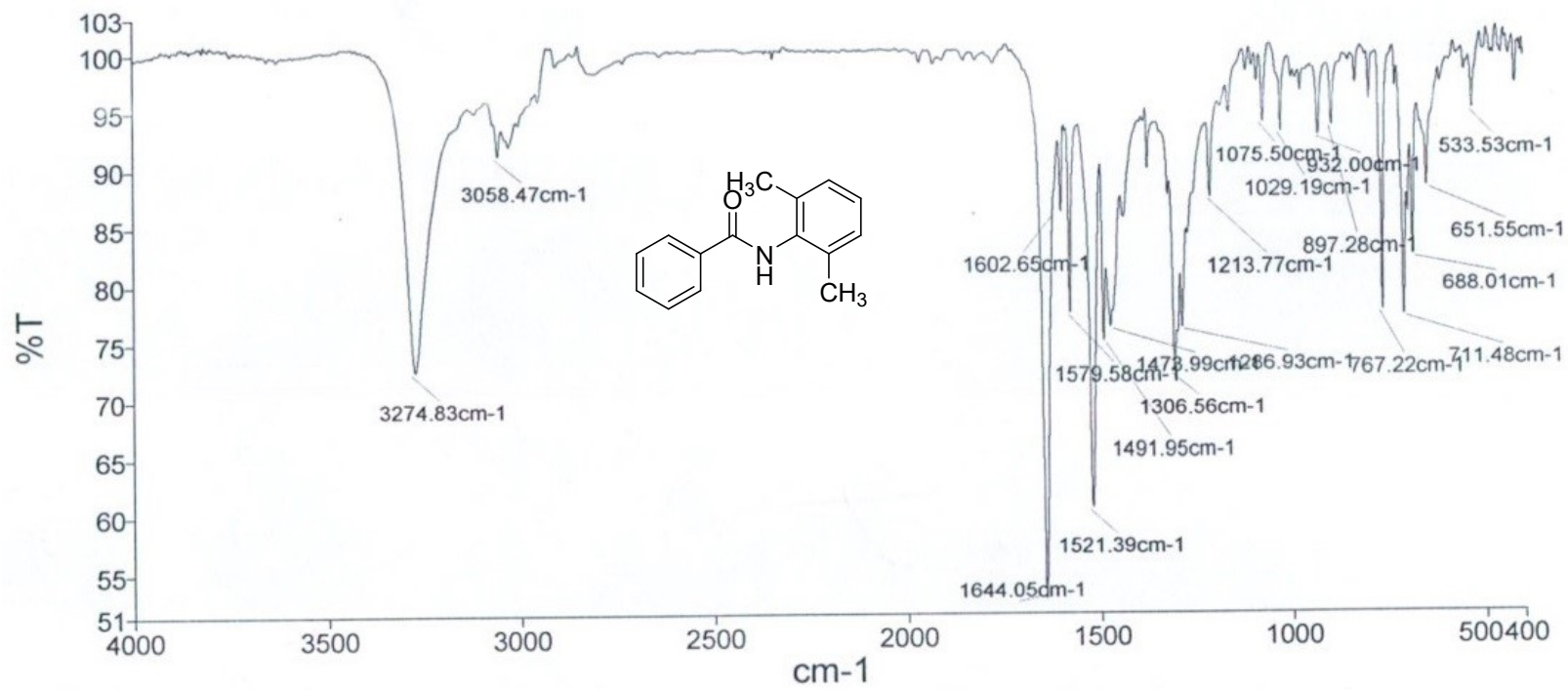
m/z-->

Abundance

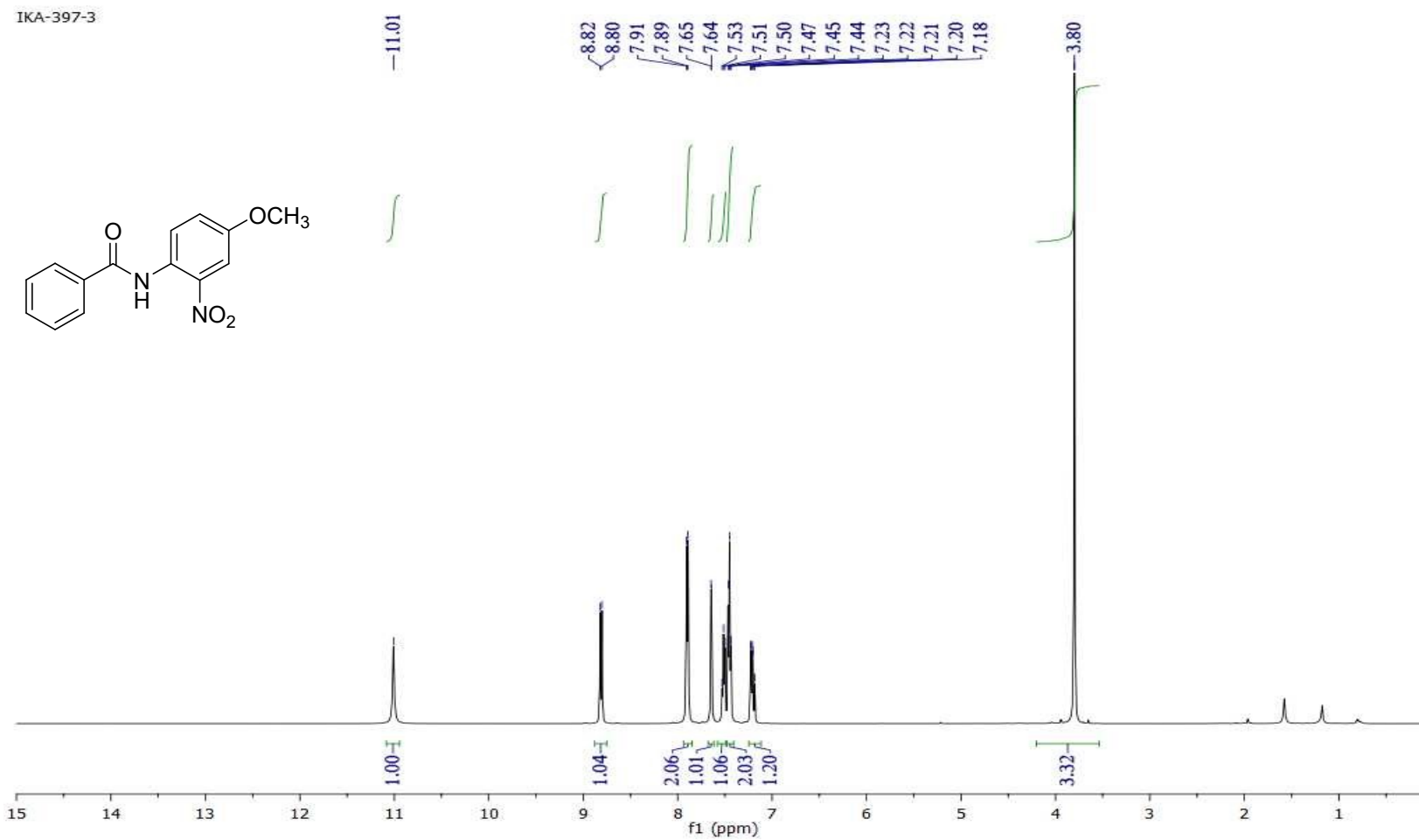
Scan 1357 (9.714 min): IKA11 2.D\data.ms



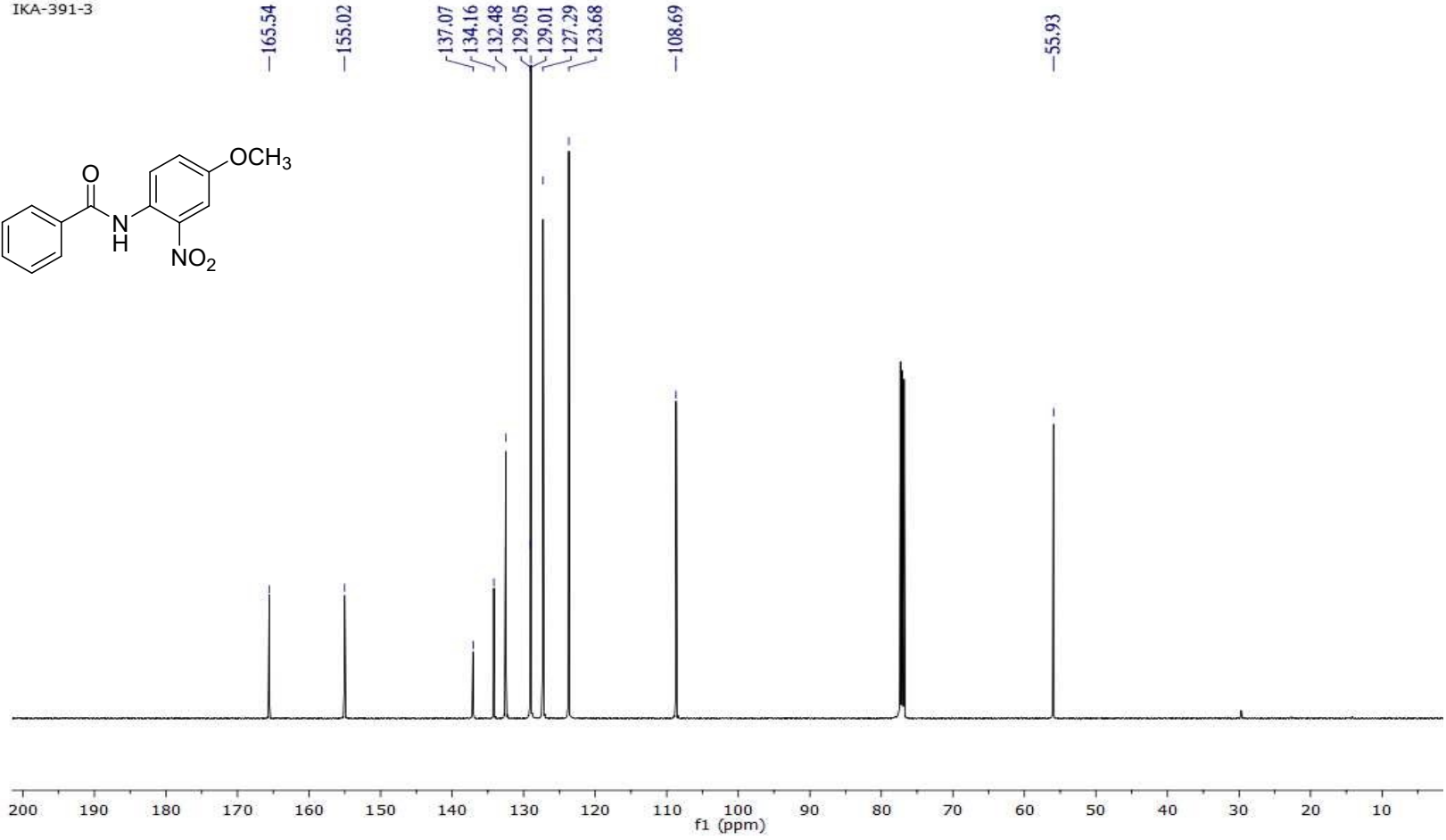
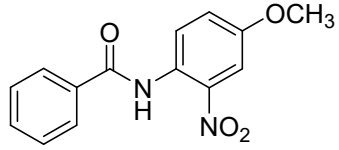
m/z-->



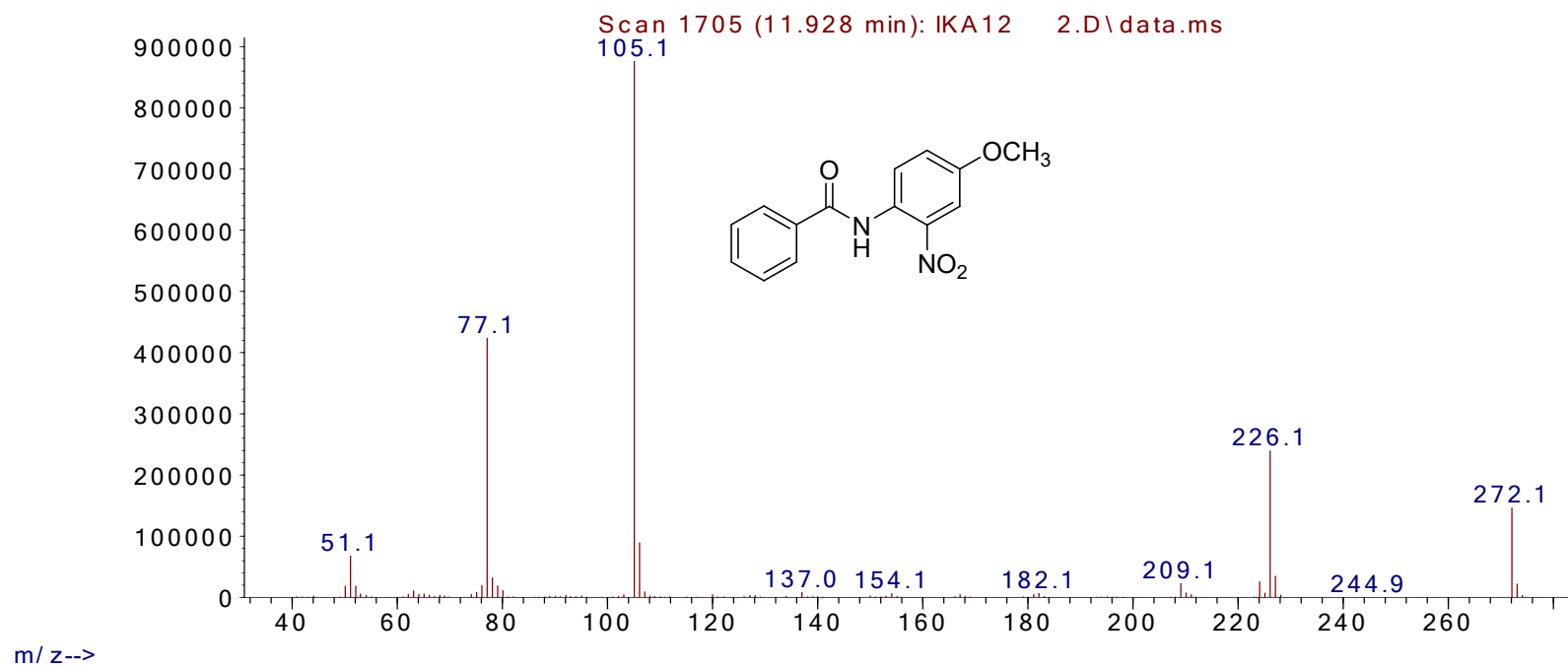
^1H NMR, ^{13}C NMR, mass and IR spectrum of *N*-(4-chloro-3-nitrophenyl)benzamide (3m)



IKA-391-3

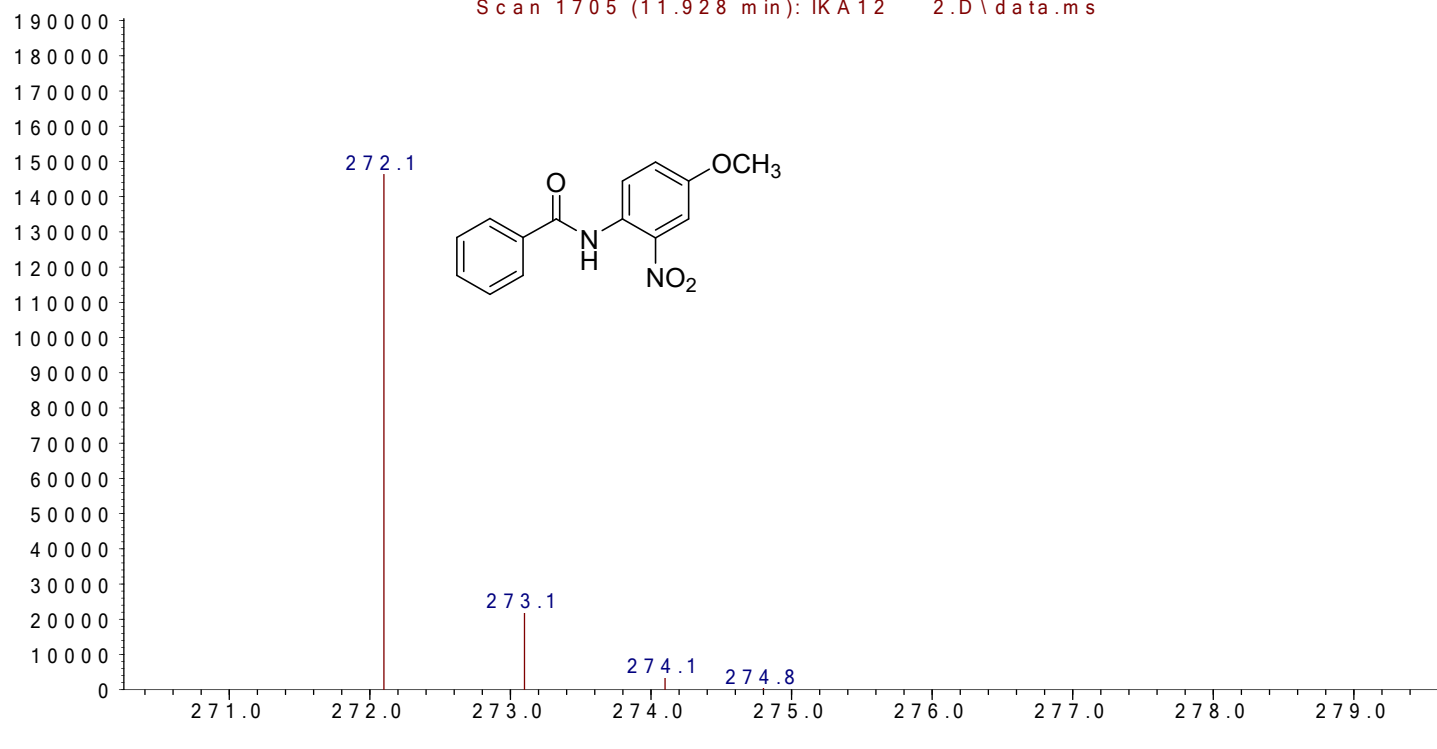


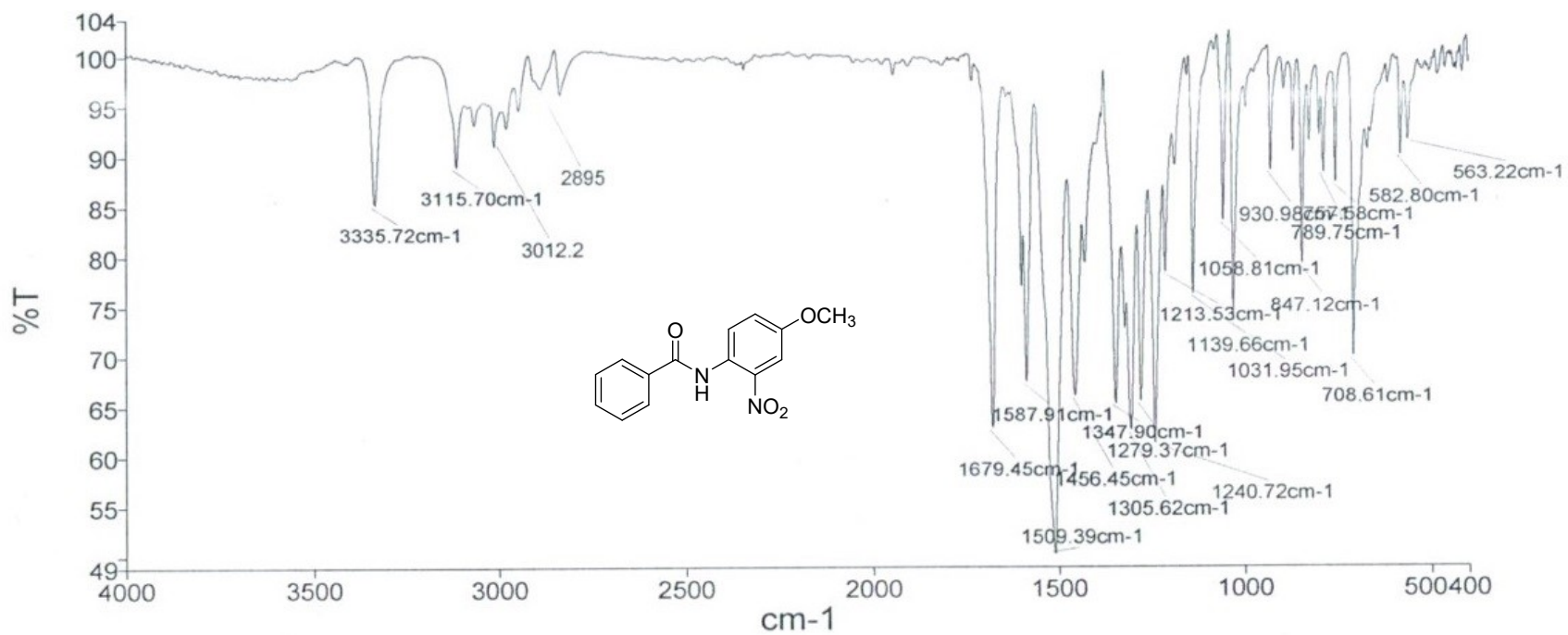
Abundance



Abundance

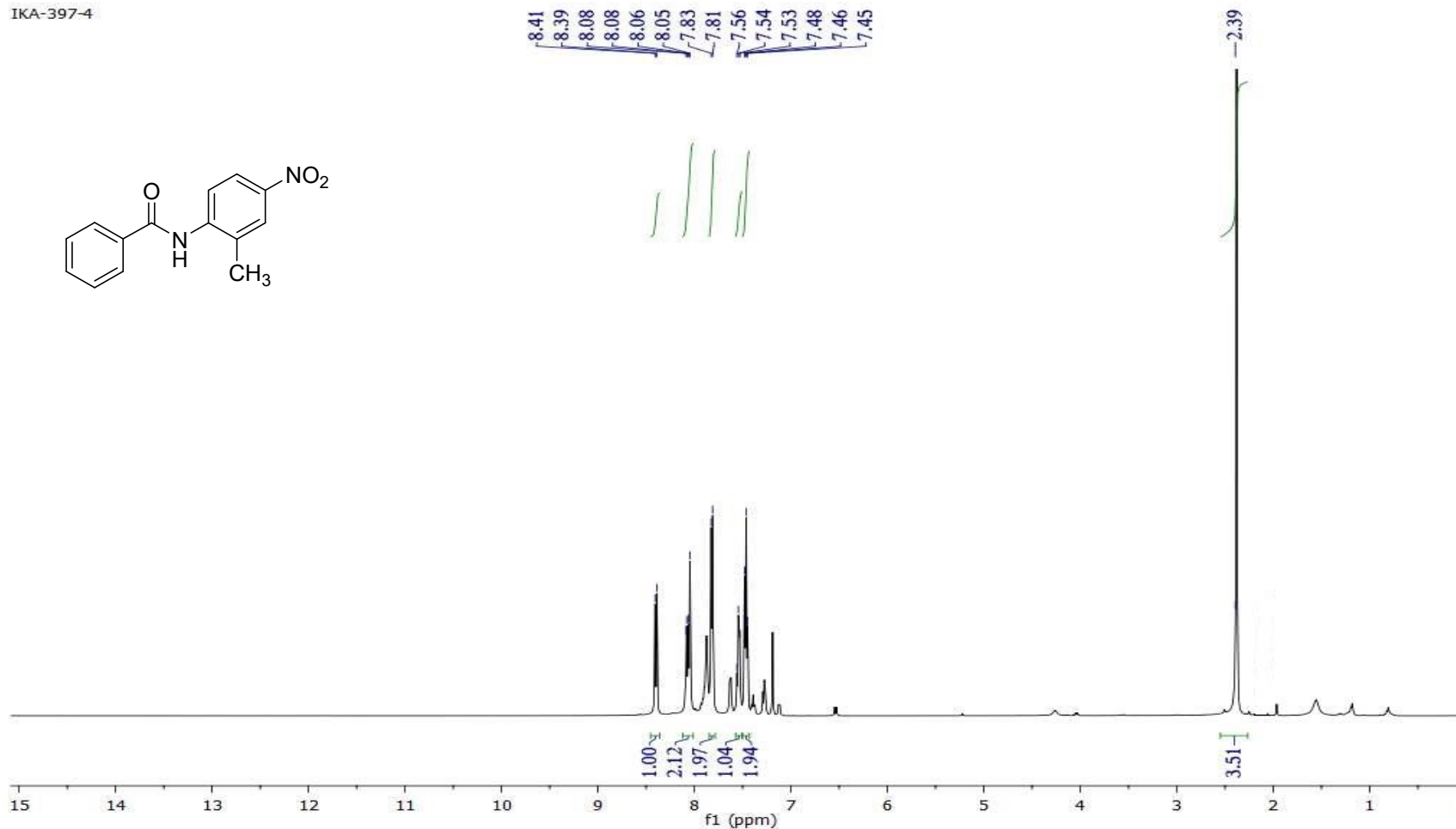
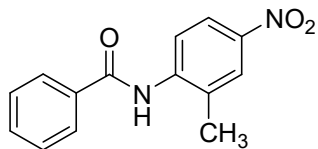
Scan 1705 (11.928 min): IKA12 2.D\data.ms



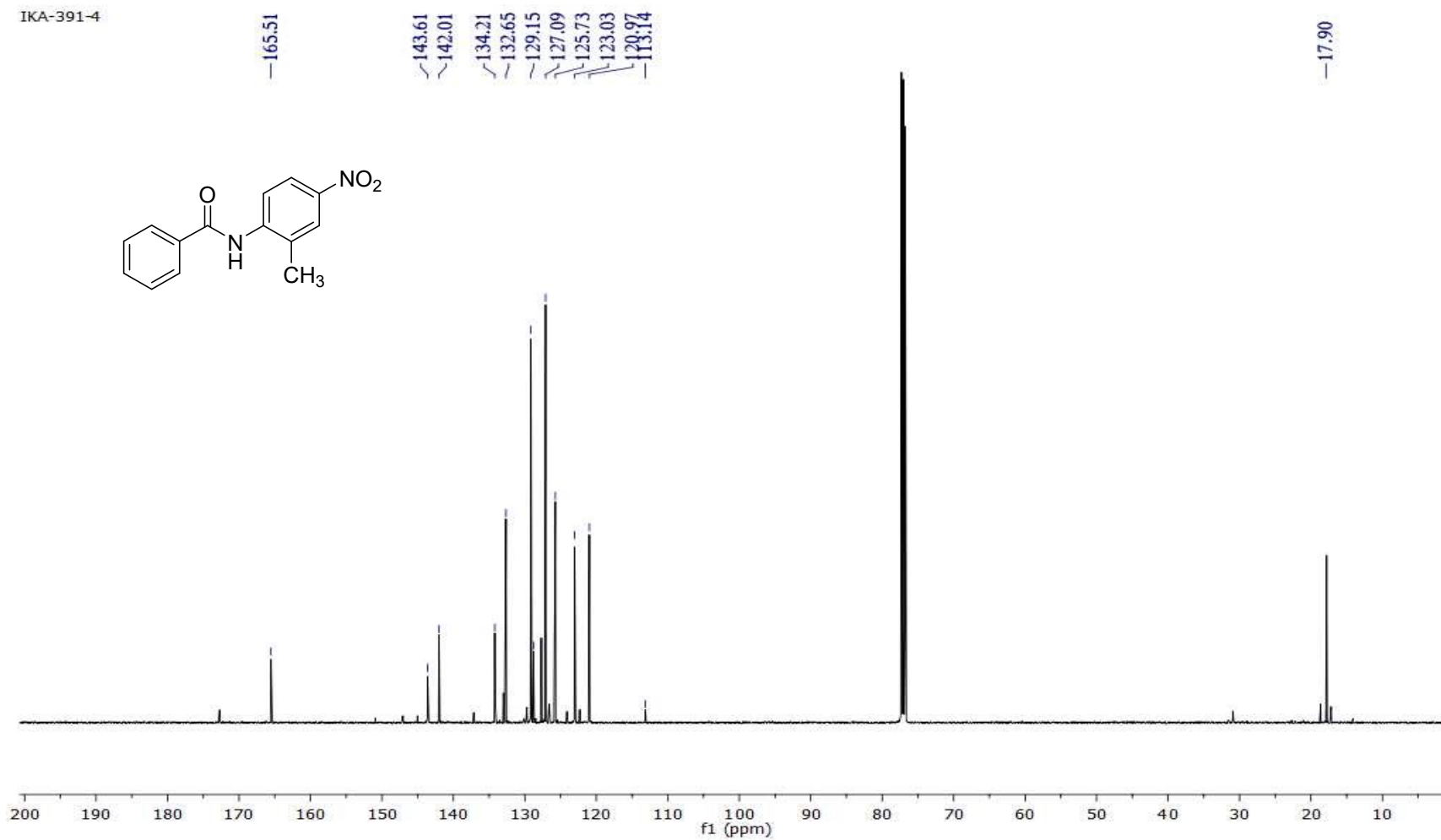
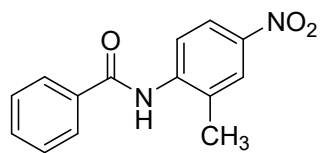


¹H NMR, ¹³C NMR, mass and IR spectrum of *N*-(2-methyl-4-nitrophenyl)benzamide (31)

IKA-397-4

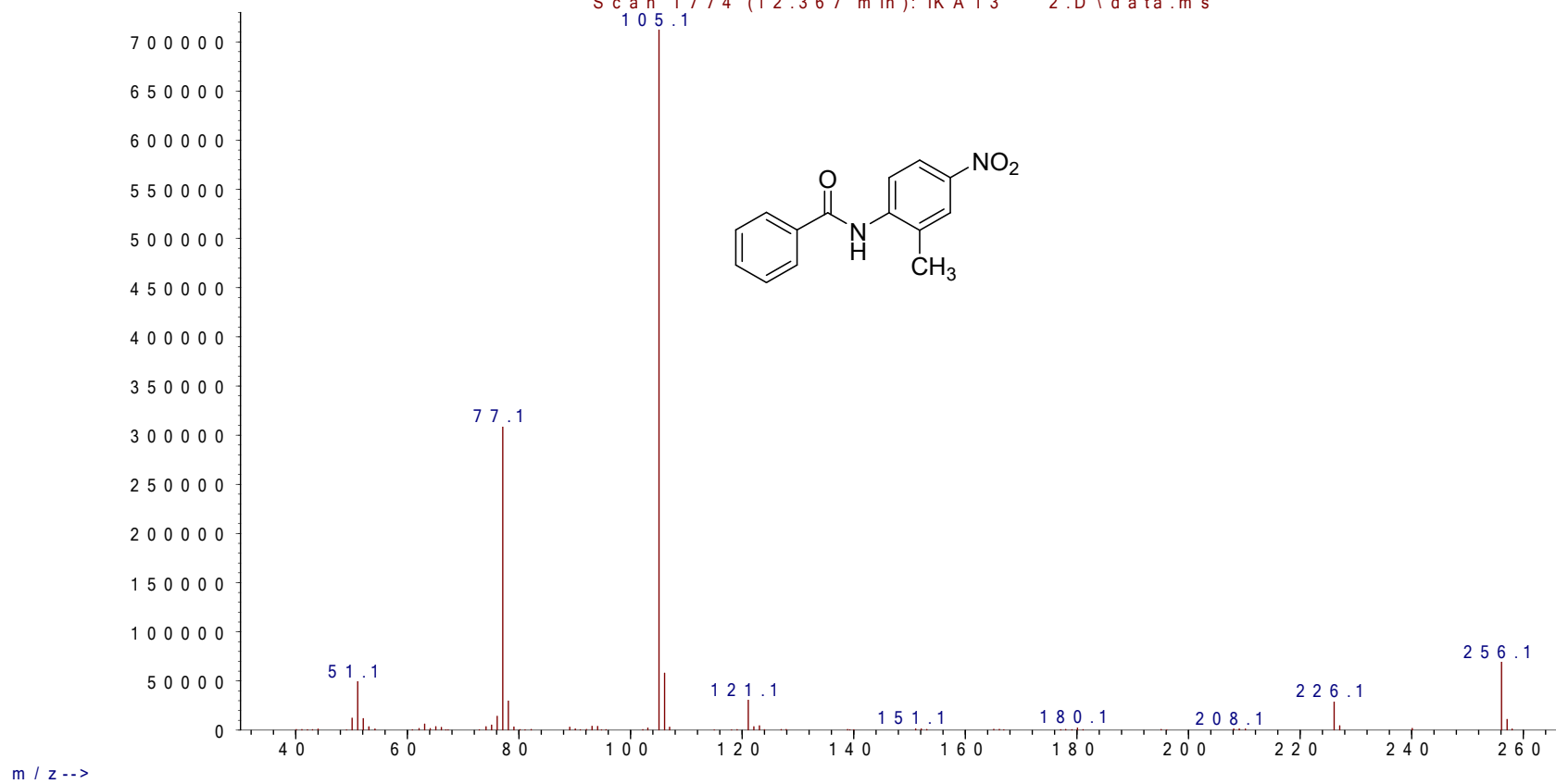
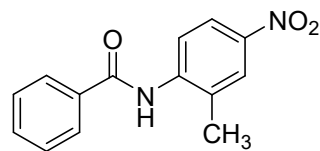


IKA-391-4



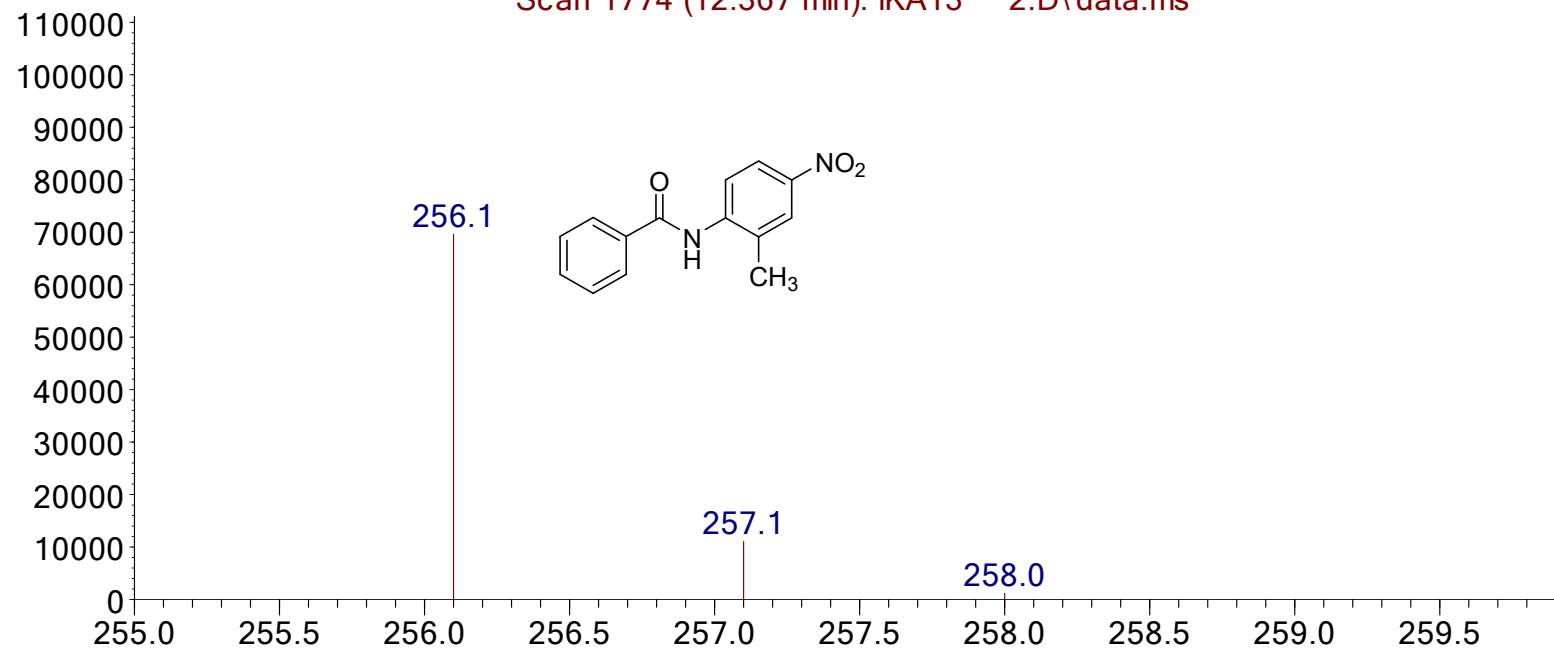
Abundance

Scan 1774 (12.367 min): IKA 13 2.D\data.ms
105.1

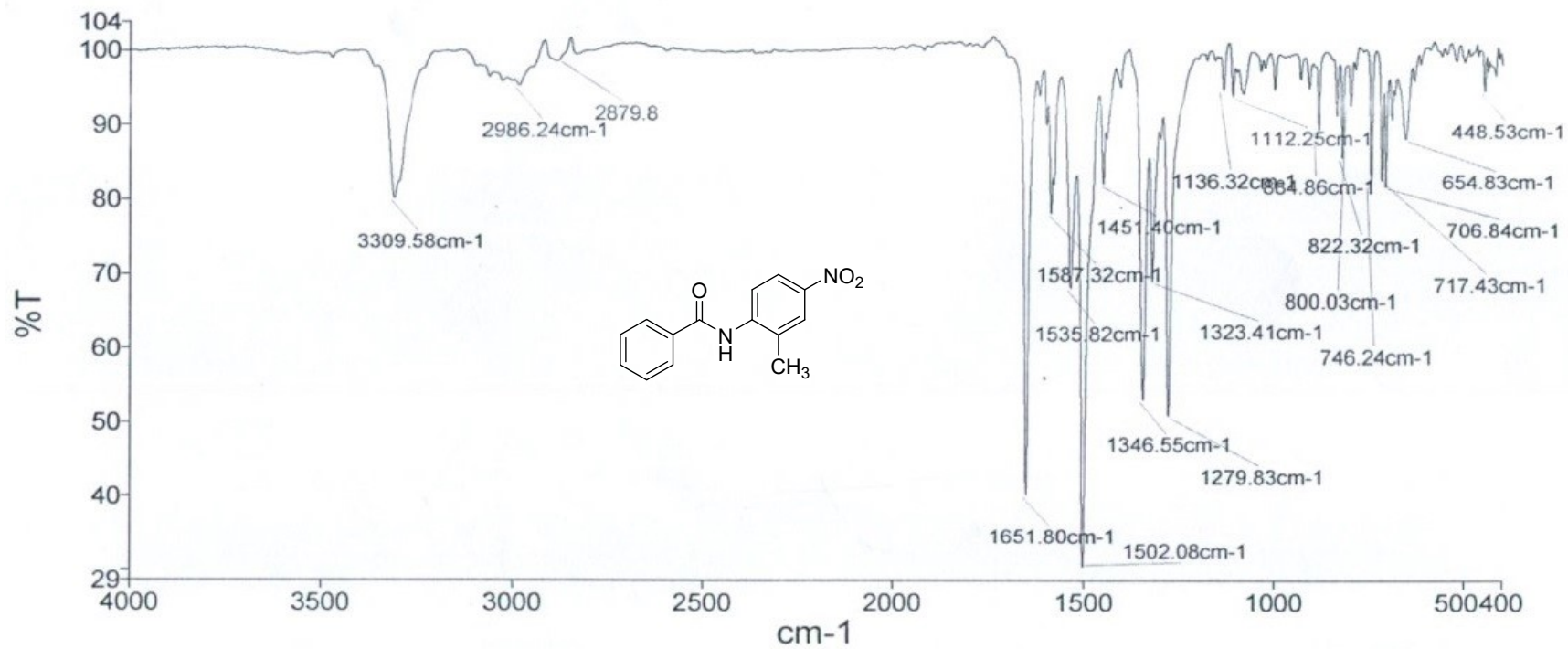


Abundance

Scan 1774 (12.367 min): IKA13 2.D\data.ms

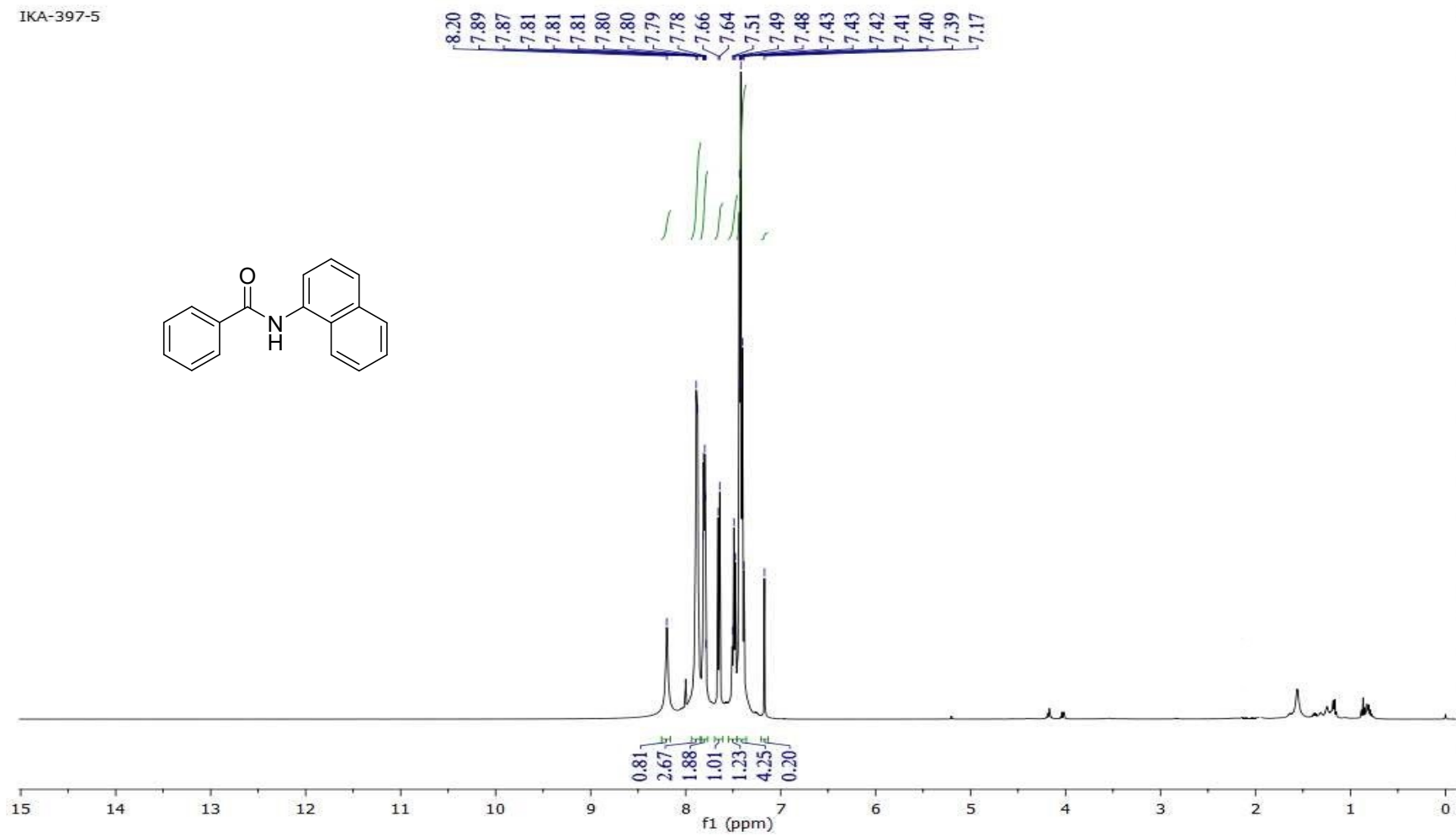


m/z-->

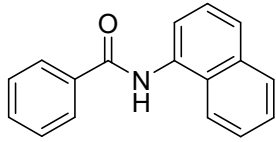


¹H NMR, ¹³C NMR, mass and IR spectrum of *N*-(naphthalen-1-yl)benzamide (3o)

IKA-397-5

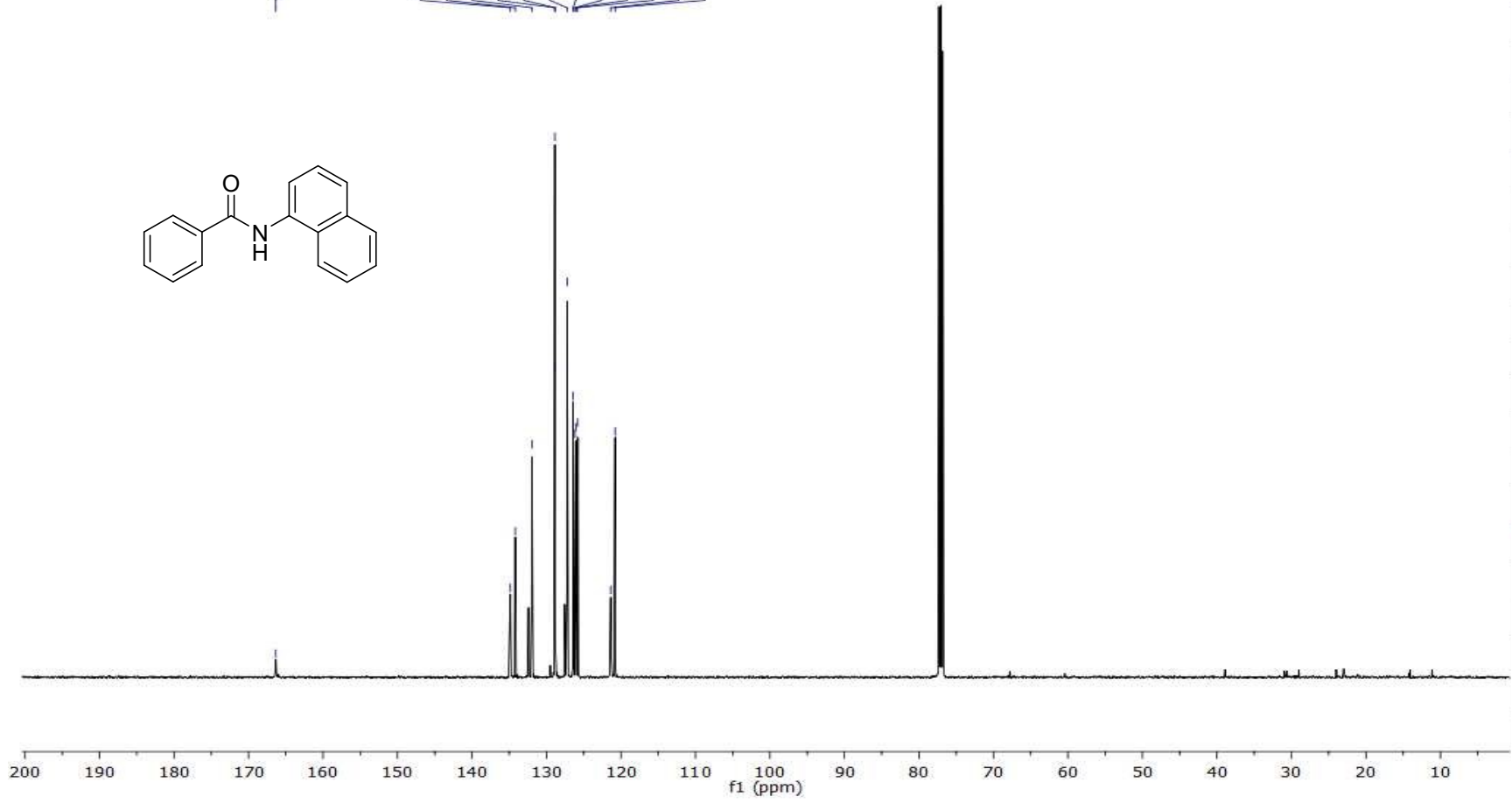


IKA-391-5



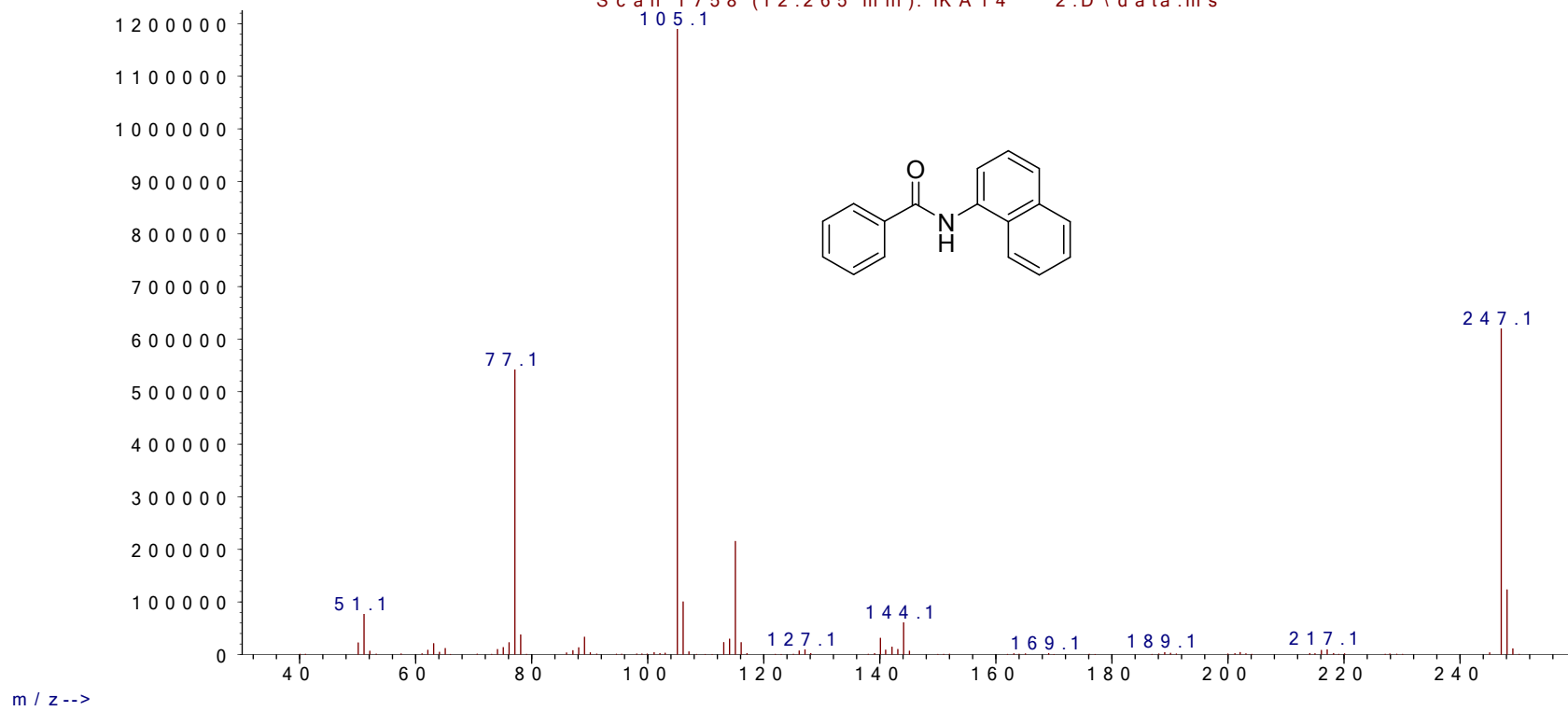
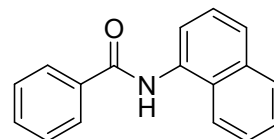
166.34

134.85
134.19
131.97
128.88
128.85
127.24
126.43
126.17
126.08
125.79
121.38
120.79



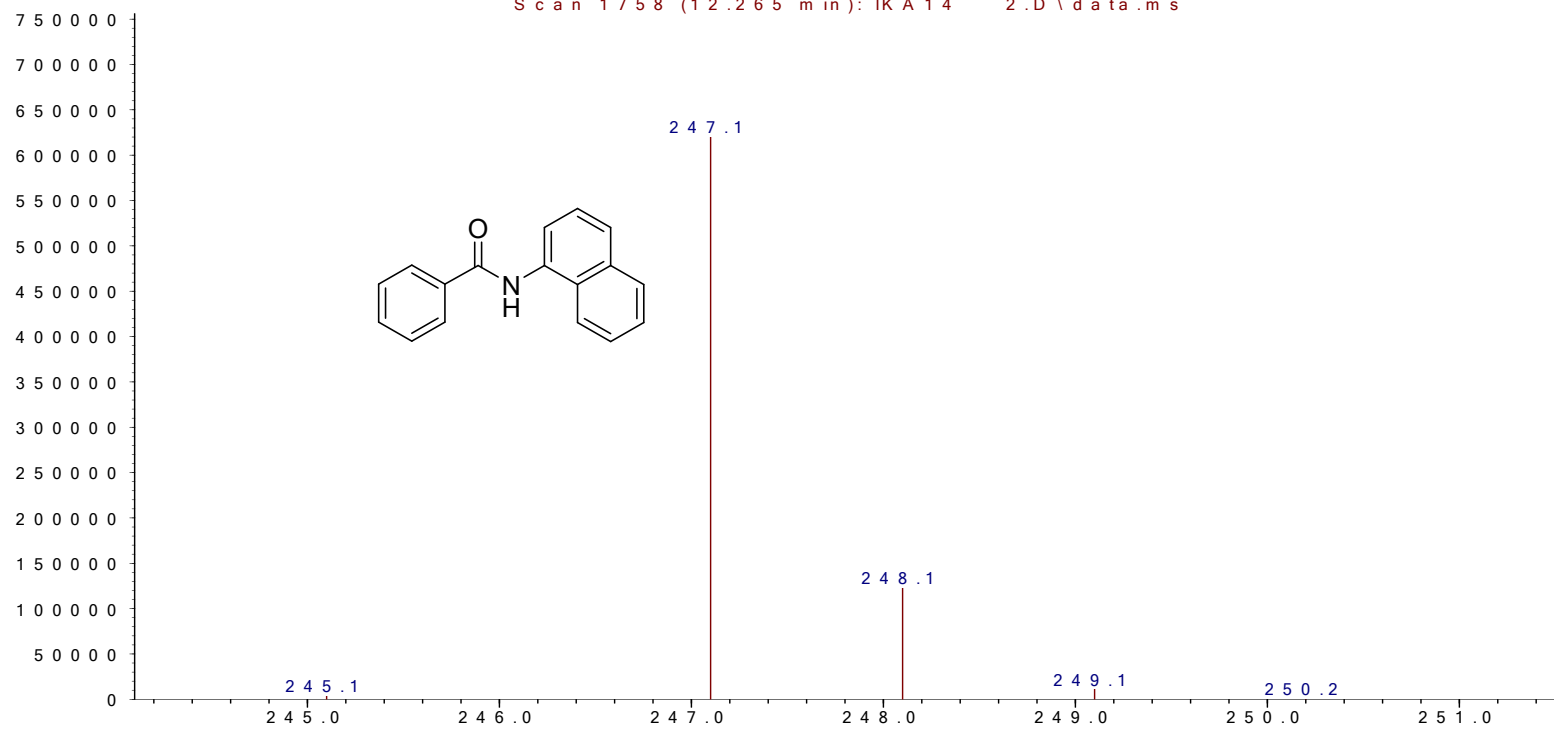
Abundance

Scan 1758 (12.265 min): IKA14 2.D\data.ms



Abundance

Scan 1758 (12.265 min): IKA14 2.D\data.ms



m / z-->

