Supporting information

An efficient magnetic copper ferrite nanoparticle catalysed ligand and solvent free synthesis of N-aryl amide from aldoximes and iodobenzene

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Experimental section

General: All chemicals were purchased from S.D Fine Chemical, Avra, Spectrochem Ltd and used without further purifications.

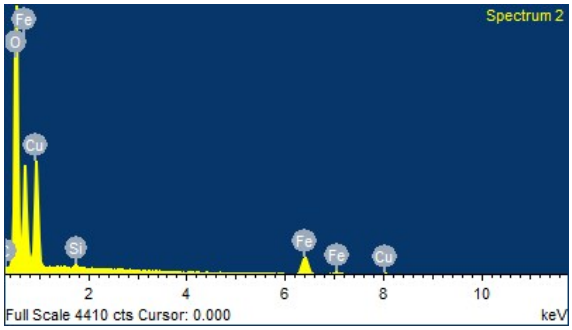
The as synthesized catalyst was characterized by various techniques such as X-ray diffraction (XRD), Field emission gun Scanning electron microscopy(FEG-SEM),Transmission electron 20 microscopy (TEM), FTIR. The XRD analysis was performed on Shimadzu XRD 2400 instrument using Cu Kα radiation (λ= 1.5406 A°) with scanning rate 2 degree per minute. TEM analysis was performed over PHILIPS 2200 instrument. FEG-SEM analysis was performed on 25 TESCAN MIRA Instrument. The energy dispersive X-ray spectral analysis (EDS) image was recorded with an Oxford instrument at 10 kV. Beam intensity was kept high to get good response by the detector. GC analysis is performed over PerkinElmer Clarus 480 instrument. GC-MS Spectra recorded over Shimadzu QP-2010 Instrument. 1H spectra's were recorded on Agilent 400MHz and 100MHz instrument respectively.

Preparation of Copper ferrite Nanoparticles.

Catalyst was prepared by using known simple co-precipitation followed by thermal decomposition method.[15b] Copper (II) nitrate (4.1 mmol) and iron (III) nitrate (8.2 mmol) were taken in stoichiometric proportions and 100 mL of deionised water was added to produce clear solution. 1M of NaOH solution was added drop wise under stirring to the above solution till the pH becomes 10.Reddish-black precipitate of copper ferrite was formed. The reaction mixture was warmed to 90°C and stirred for 2h which on cooling gave magnetic particles. The catalyst was then washed with water and ethanol and separated by using a magnet. It was kept in air oven at 80°C for 12 h. Then the catalyst was grinded in a mortar-pestle and kept in a furnace at 700°C for 5 h. It was then cooled to room temperature slowly to get the magnetic copper ferrite NPs.

EDAX Analysis of Copper ferrite NPs.

Element	Weight%	Atomic%
C K	2.02	5.58
O K	25.75	53.27
Si K	0.28	0.33
Fe L	46.49	27.55
Cu L	25.46	13.26
Totals	100.00	

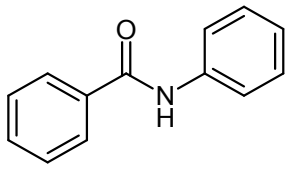


General procedure for N- Arylation of amide

An oven-dried Schlenk tube equipped with a magnetic stirring bar was charged with benzaldoxime (2 mmol), iodobenzene (1 mmol), Copper ferrite NPs (10mol %), and K_2CO_3 (2 mmol). Reaction mixture was heated in an oil bath at 150°C and was stirred for 12 h. The reaction was monitored by GC and TLC. After completion of the reaction, the reaction mixture was cooled to room temperature and the reaction mass was diluted with ethyl acetate and filtered through plug of celite. The resulting filtrate was washed with water and 20% brine solutions. The organic layer was separated and dried over anhydrous sodium sulphate. The solvent was removed under vacuum to get the crude product, which was purified by column chromatography on silica gel eluting with the mixture of pet ether / EtOAc (80: 20) mixture to afford the pure product. The purity and identity of known products are conformed by ^{1}H NMR and GC-MS Spectroscopic techniques.

Spectroscopic data:

Table 1 entry 1

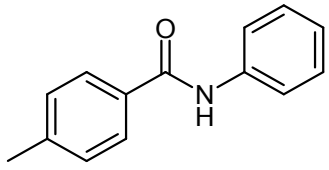


N- Phenyl benzamide: White solid mp 163-166°C

^{1}H NMR: (300 MHZ, $CDCl_3$, TMS): δ 7.9-7.1(m, 10H, Ar-H)

GC-MS m/z (% relative intensity): 197(M+, 30), 105 (100), 77(60), 51(17)

Table 1 entry 2

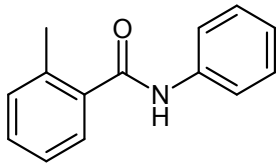


4- Methyl- N- Phenyl benzamide: Light brown solid; yield: 0.185 g (88%); mp 148-150°C

^{1}H NMR : (300 MHZ, $CDCl_3$, TMS): δ7.8-7.1(m,9H,Ar-H), 2.4(s, 3H,CH_3)

GC-MS m/z (% relative intensity): 211(M+,27), 119(100), 91(44), 65(26)

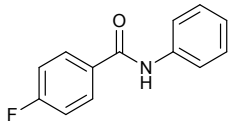
Table 1 entry 3



2- Methyl –N- Phenyl benzamide: mp 123-126°C

GC-MS m/z (% relative intensity): 211(M+, 16), 194(10), 119(100), 91(54), 65(26)

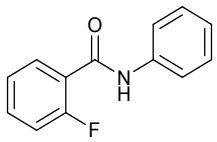
Table 1 entry 4



4-Flouro-N-Phenyl benzamide: mp 170-181°C

GC-MS m/z (% relative intensity): 215 (M+) 123 (100) 95 (44)

Table 1 entry 5



2-Flouro-N-Phenyl benzamide: mp 95-96 °C

GC-MS m/z (% relative intensity):

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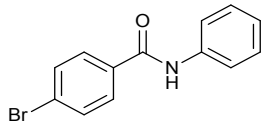
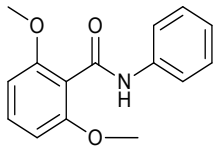


4-Bromo-N-phenyl benzamide: 202°C

GC-MS m/z (% relative intensity):276 (M+),155(35),183(100)

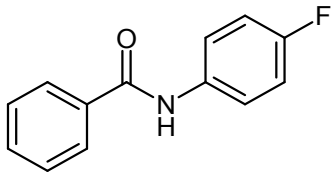
Table 1 entry 7



2,6-dimethoxy-N-Phenyl benzamide: 168°C

GC-MS m/z (% relative intensity): 257(M+),165(100),137 (37%)

Table 3 Entry 1



N-(4- Fluoro phenyl) benzamide: mp 182-185°C

^{1}H NMR: (300 MHZ, $CDCl_3$, TMS): δ 7.9-7.1(m, 9H, Ar-H)

GC-MS m/z (% relative intensity):215(M+,26), 105(100), 77(61), 51(18), 44(13), 40(15)

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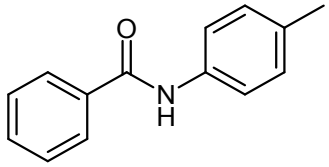
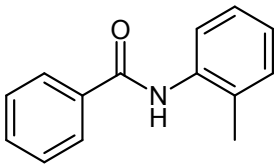


N-(4- Methyl phenyl)benzamide: mp 154-156°C

^{1}H NMR: (300 MHZ, $CDCl_3$, TMS): δ 7.9-7.1(m, 9H, Ar-H), 2.3(s, 3H, CH_3)

GC-MS m/z (% relative intensity):- 211(M+, 33), 105(100), 77(57), 51(13)

Table 3 Entry 3

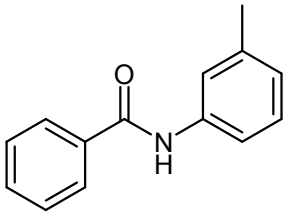


N-(2-Methyl phenyl) benzamide : mp 142-144°C

^{1}H NMR : (300 MHZ, $CDCl_3$, TMS): δ 7.9-7.1(m,9H,Ar-H), 2.3(s, 3H, CH_3)

GC-MS m/z (% relative intensity): 211(M+, 30), 105(100), 77(58), 51(14)

Table 2 Entry 4

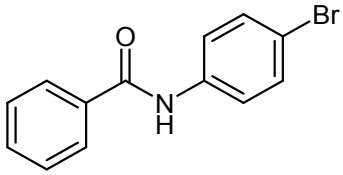


N- (3-Methyl phenyl) benzamide : mp 127-129°C

^{1}H NMR: (300 MHZ, $CDCl_3$, TMS): δ 7.9-6.94(m, 9H, Ar-H), 2.3 (s, 3H, CH_3)

GC-MS m/z (% relative intensity): 211(M+, 42), 105(100), 77(55), 51(12)

Table 3 Entry 5

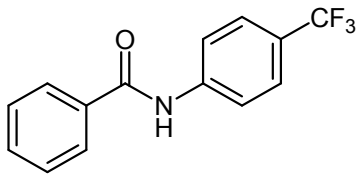


N-(4- Bromo phenyl) benzamide : mp 203-205°C

^{1}H NMR: (300 MHZ, $CDCl_3$, TMS): δ 7.9-7.1(m, 10H, Ar-H)

GC-MS m/z (% relative intensity): 276(M+,11), 275(M+, 11), 105(100), 77(49), 51(13.2)

Table 3Entry 6

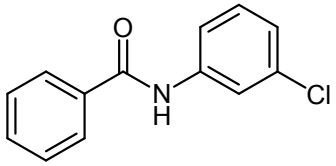


N-(4-(Trifluoro methyl) phenyl) benzamide) :

mp 204- 206°C

GC-MS m/z (% relative intensity): 265(M+, 21), 105(100), 77(53)

Table 3 Entry 7



N-(3 Chloro phenyl) benzamide: mp 120-122°C

^{1}H NMR: (300 MHZ, $CDCl_3$, TMS): δ 7.9-7.1(m, 9H, Ar-H)

GC-MS m/z (% relative intensity): 231(M+,22), 105(100), 77(52), 51(13)

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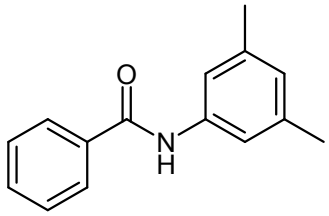

N-(3, 5-Dimethyl phenyl) benzamide : mp 141-143°C

^{1}H NMR: (300 MHZ, $CDCl_3$, TMS): δ 7.9-7.1(m, H, Ar-H)

GC-MS m/z (% relative intensity): 225(M+34), 105(100), 77(45)

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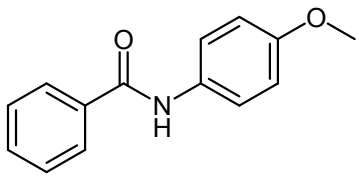
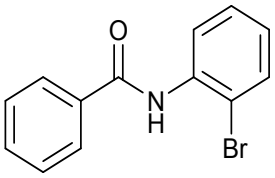


N- (4-Methoxy phenyl) benzamide : mp 156-158°C

^{1}H NMR: (300 MHZ, $CDCl_3$, TMS): δ 7.9-7.1(m, 9H, Ar-H), 3.9 (s, 3H, CH_3)

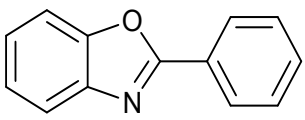
GC-MS m/z (% relative intensity): 227(M+, 40), 105(100), 77(47), 51(10)

Table 3 Entry 10



N-(2-bromo phenyl) benzamide: mp 111-112°C

GC-MS m/z (% relative intensity): 275 (M+ 3.27),183(100)

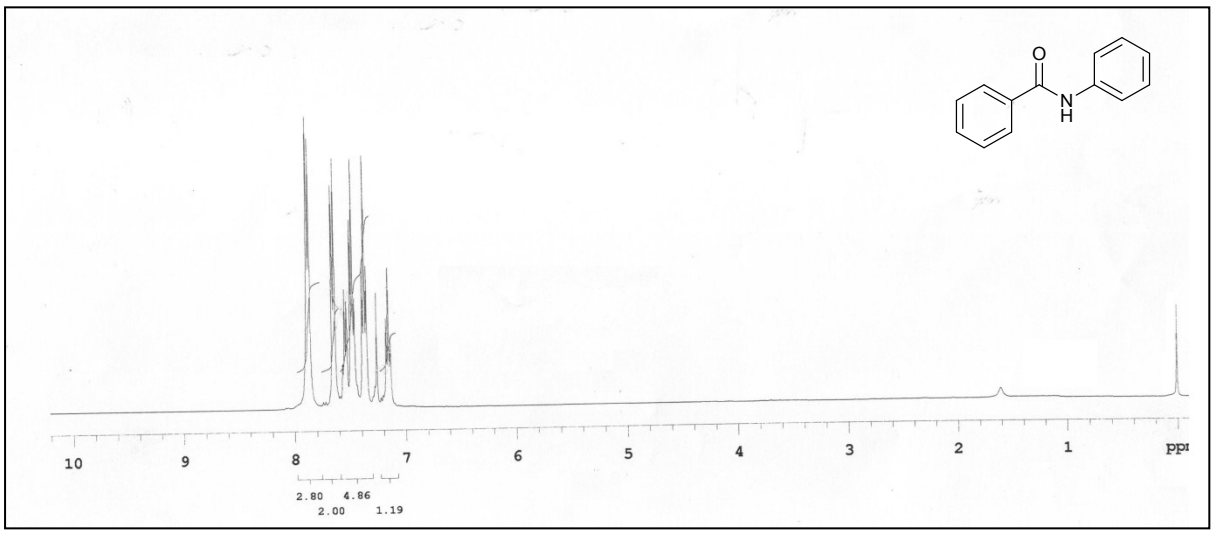


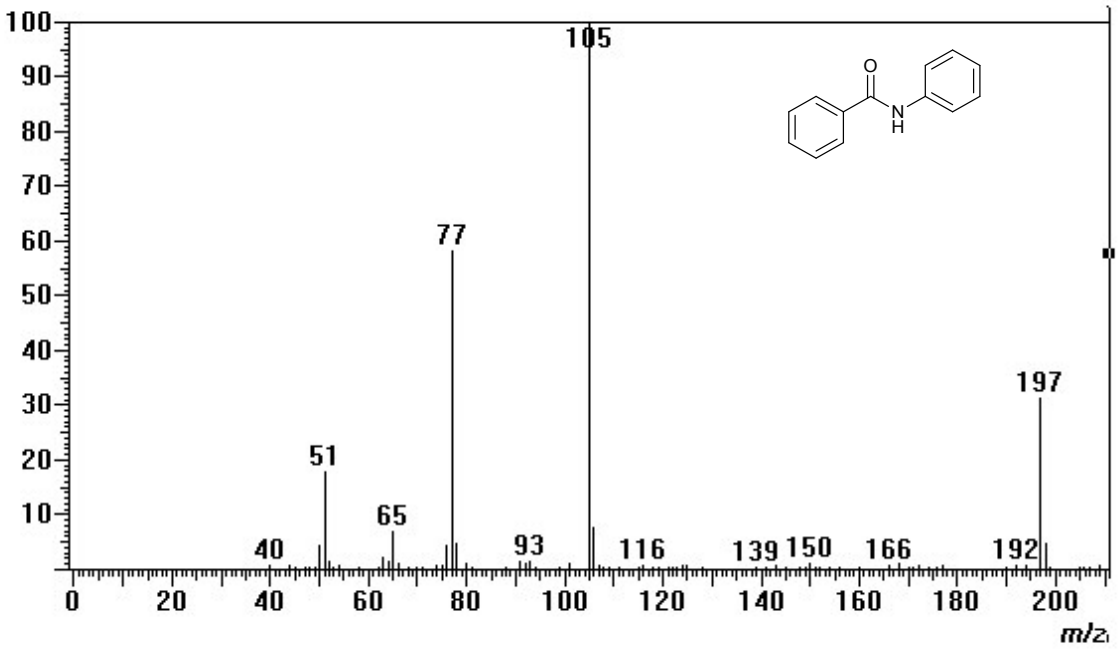
2-phenyl benzaoxazole: mp 102-103°C

GC-MS m/z (% relative intensity): 195(M^+):63(25.0), 77(14),167(21.2), 195(100)

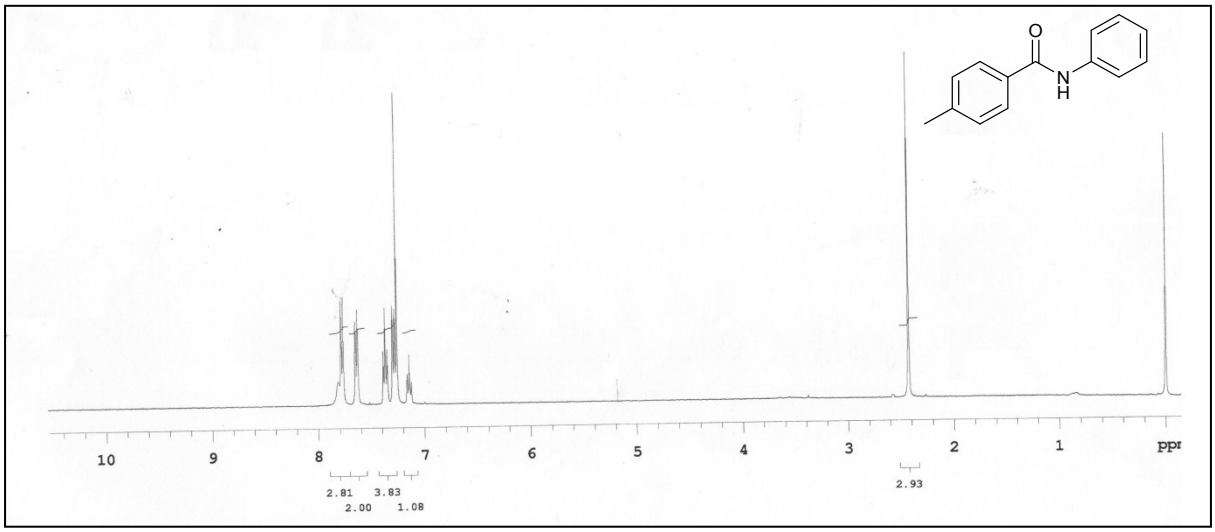
^{1}H NMR and GC-MS data of the product:

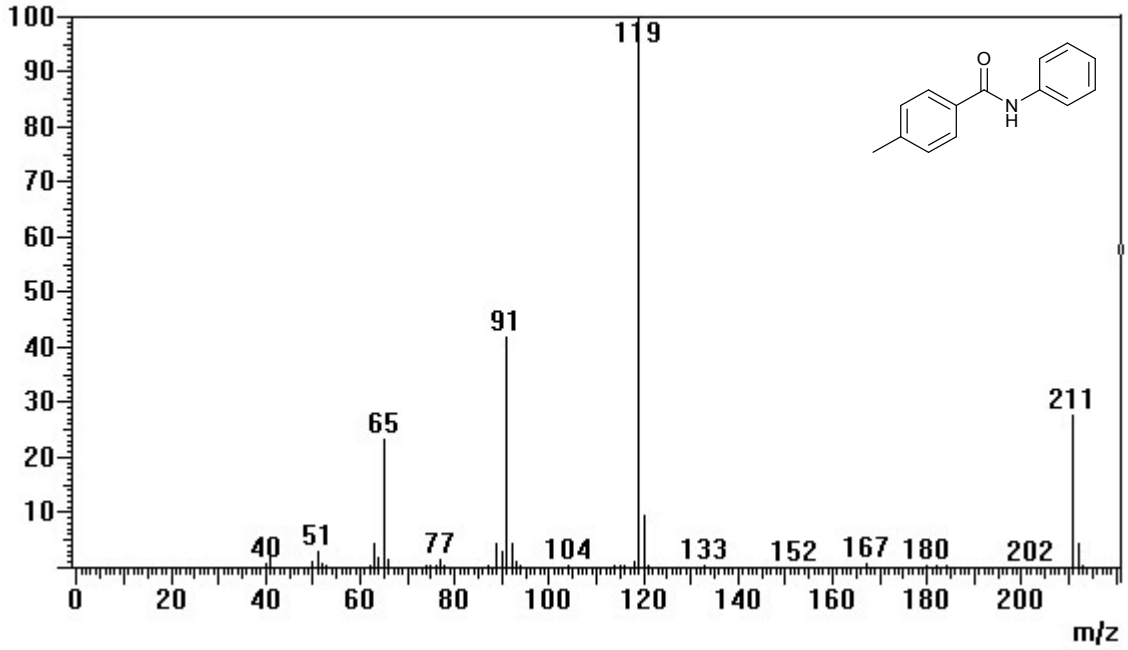
cN- Phenyl benzamide



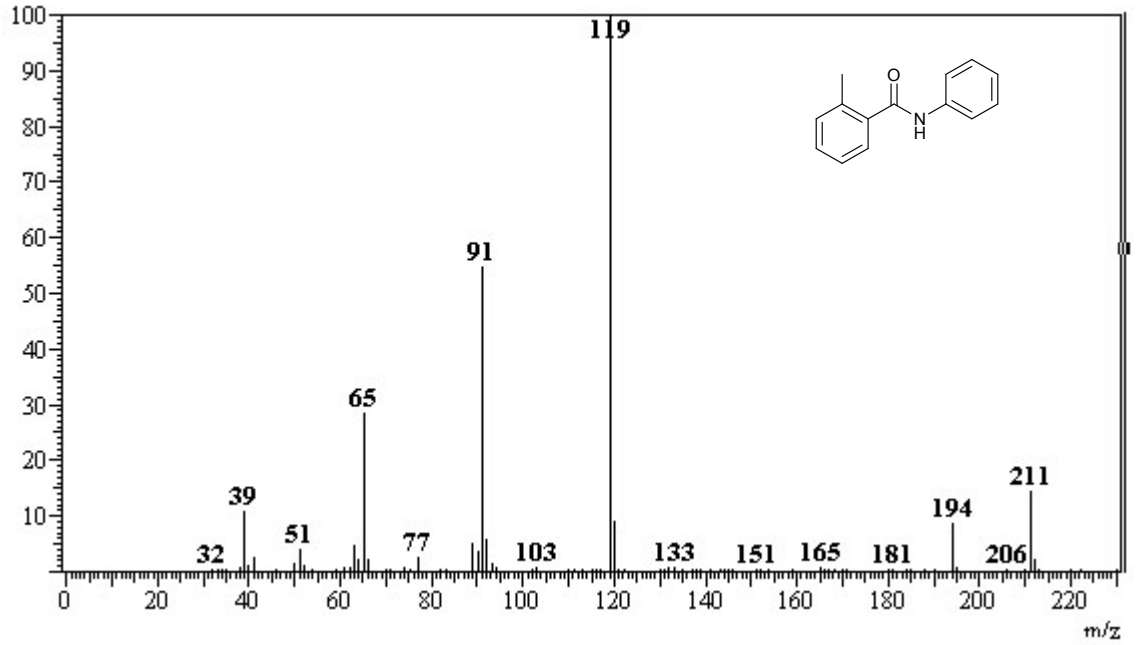


4- Methyl- N- Phenyl benzamide

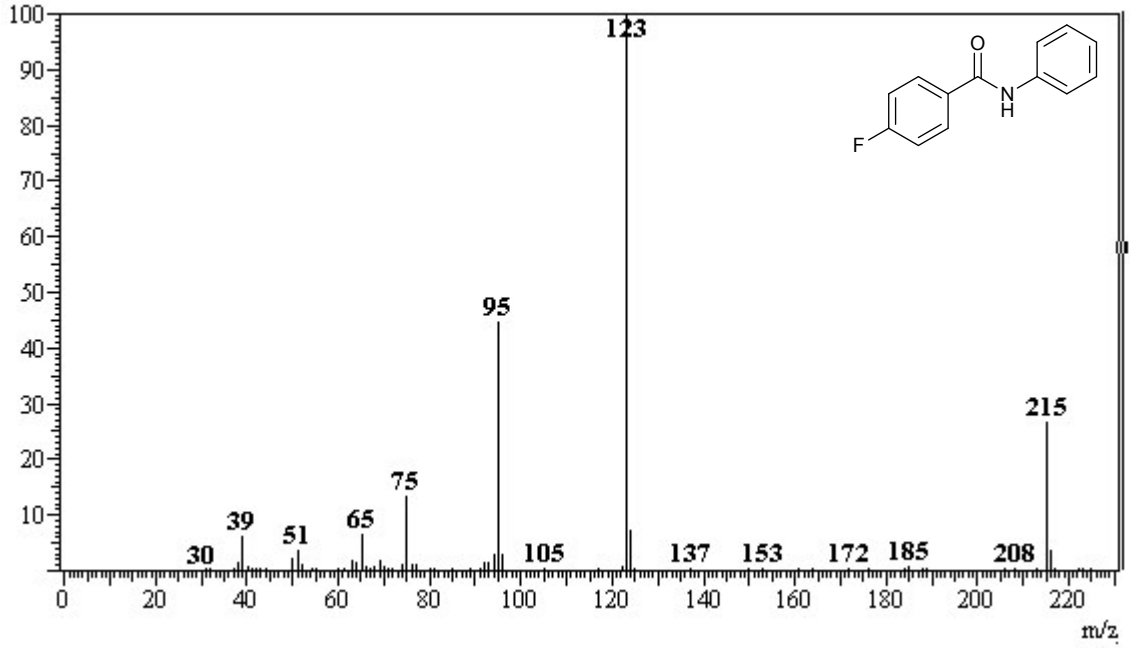




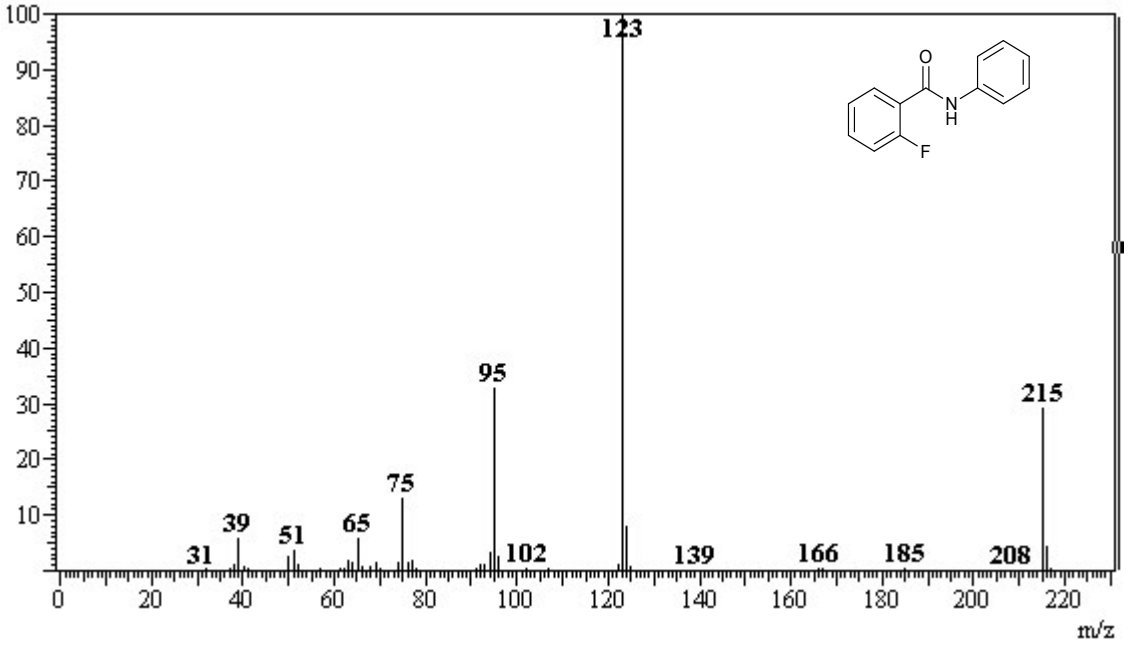
2- Methyl- N- Phenyl benzamide



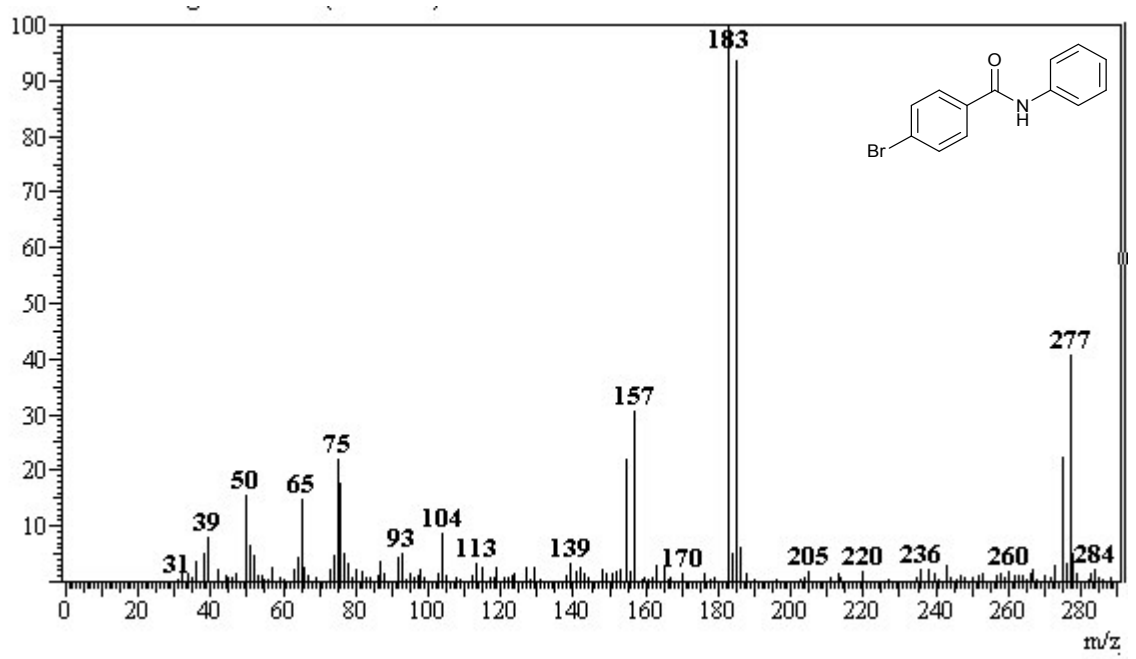
4- floro- N- Phenyl benzamide



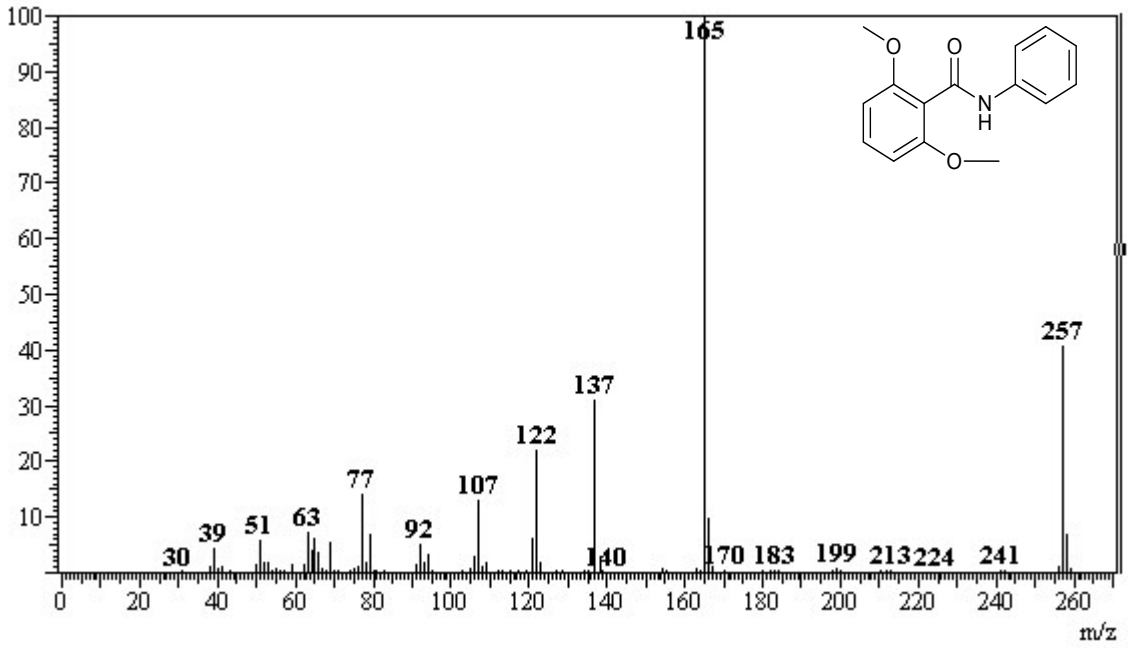
2-floro - N- Phenyl benzamide



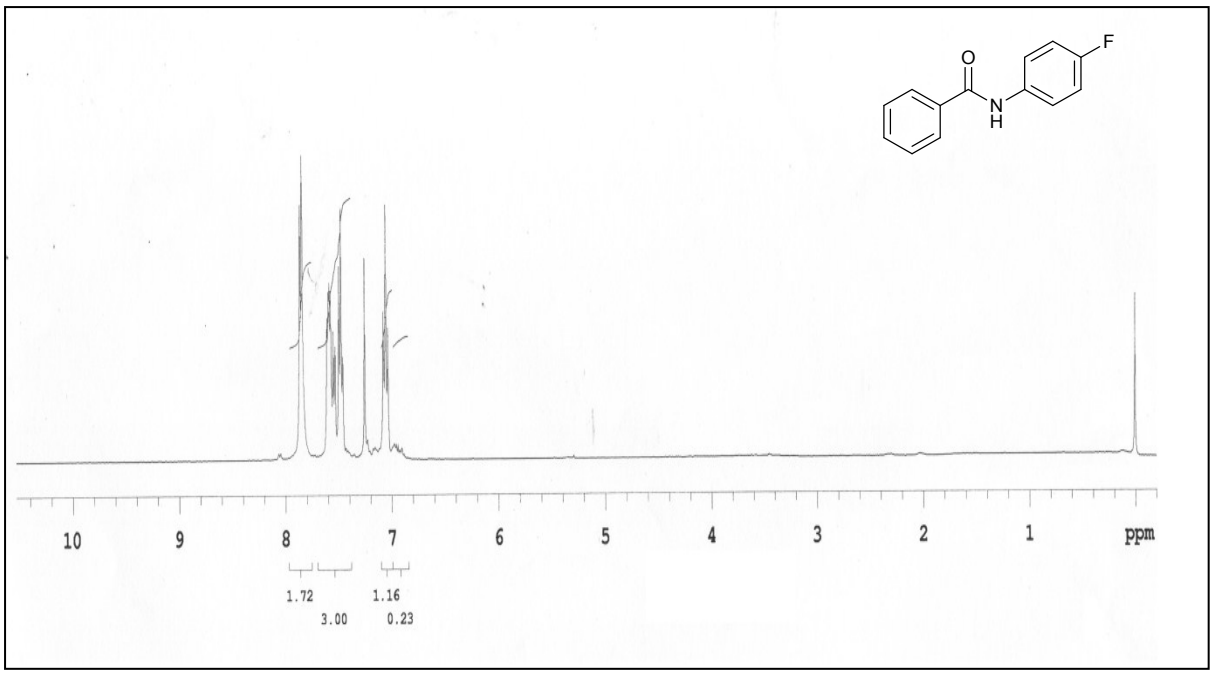
4-bromo- N- Phenyl benzamide



2,6-dimethoxy-N-phenyl benzamide



N-(4- Fluoro phenyl) benzamide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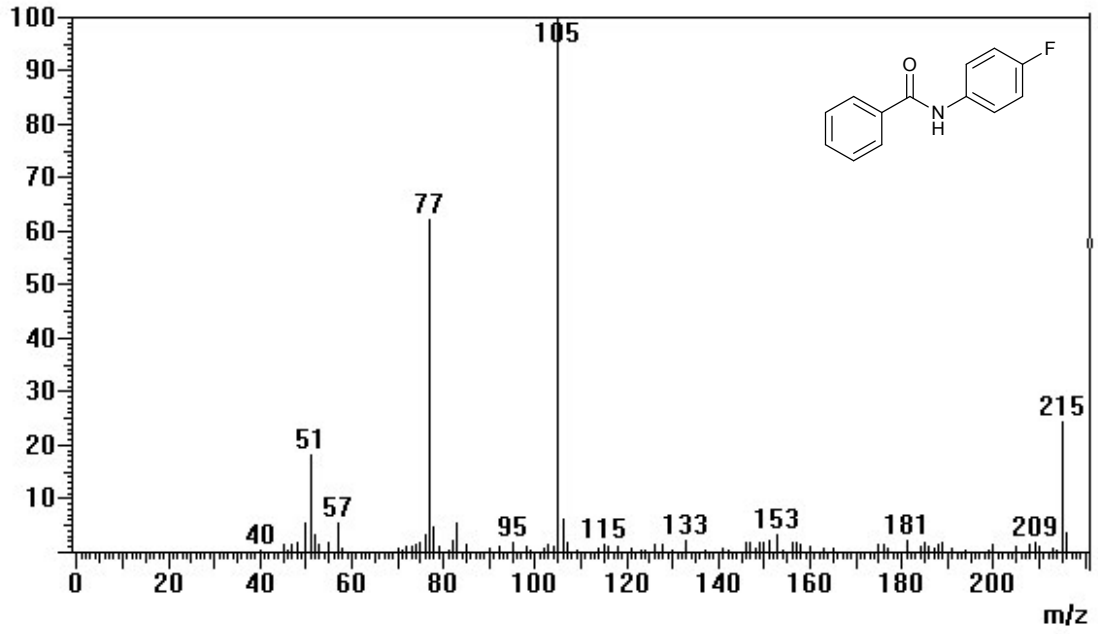
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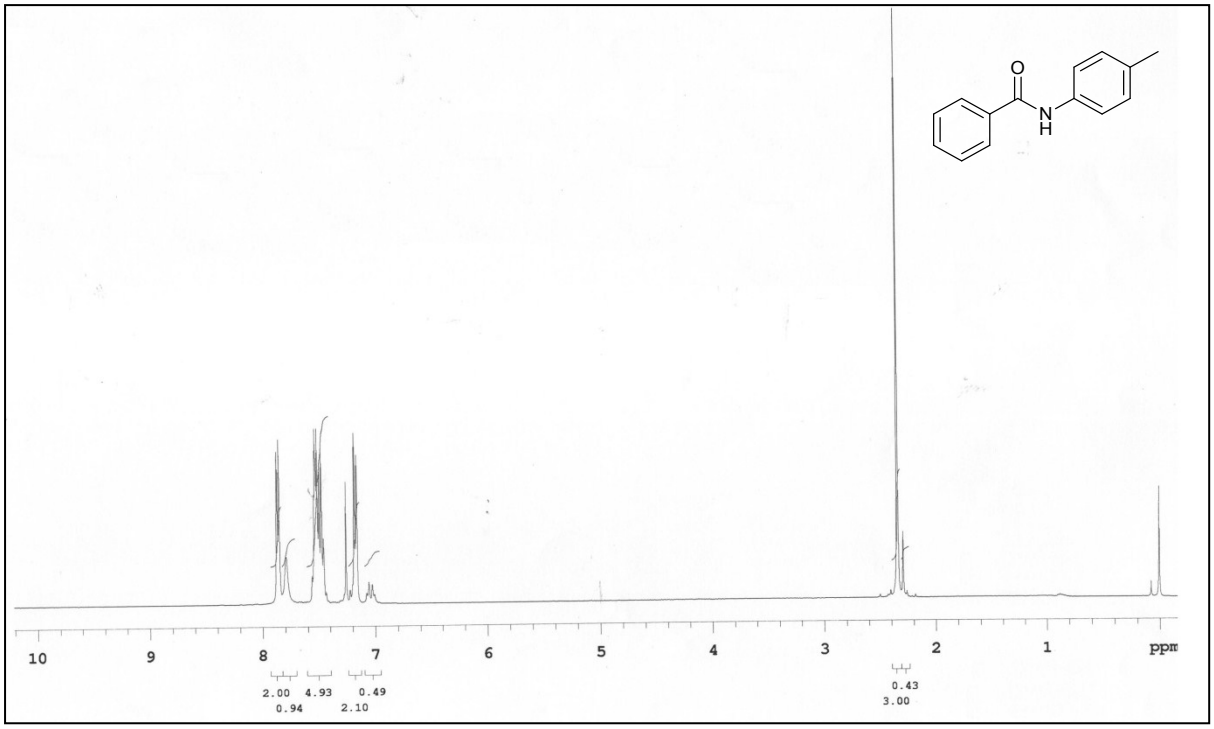
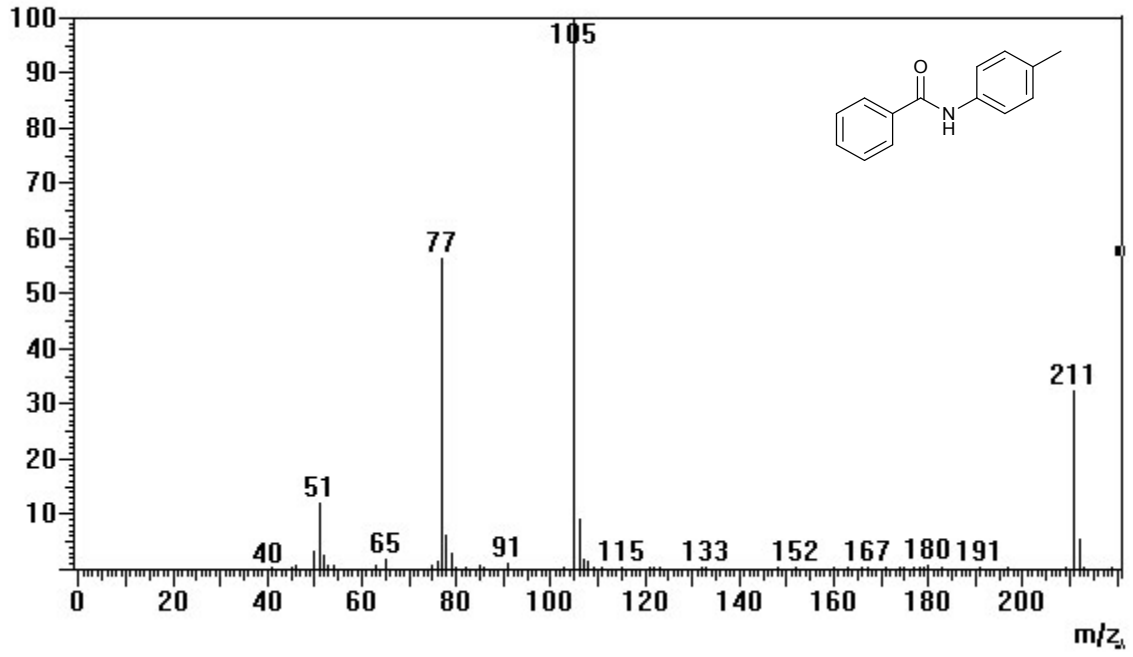
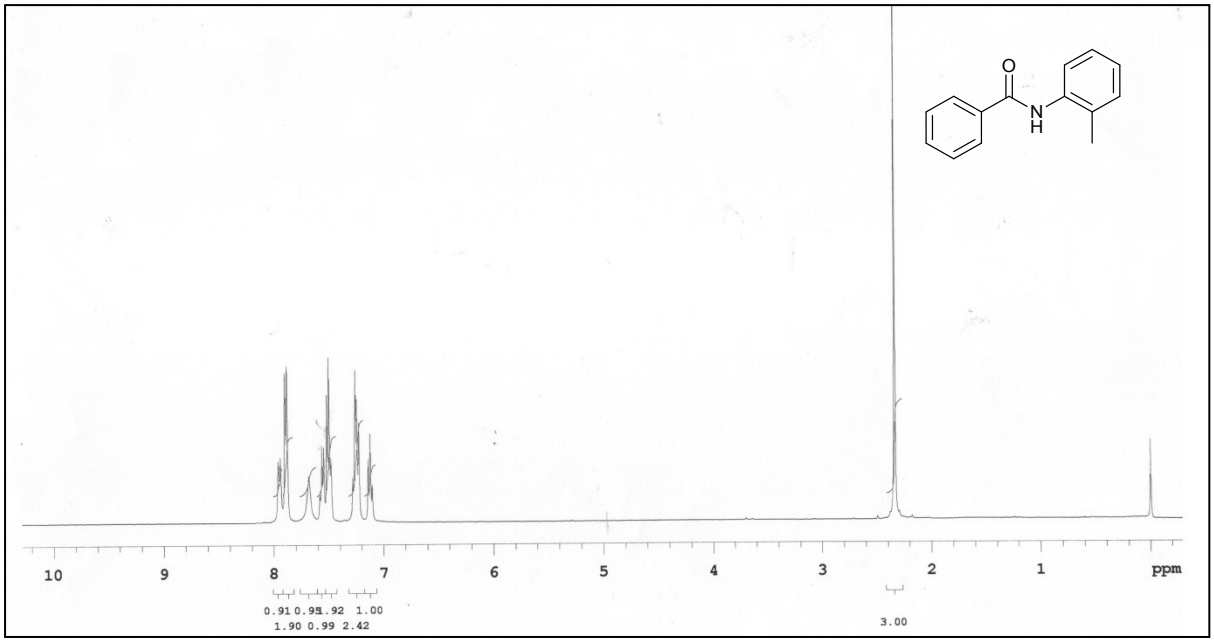
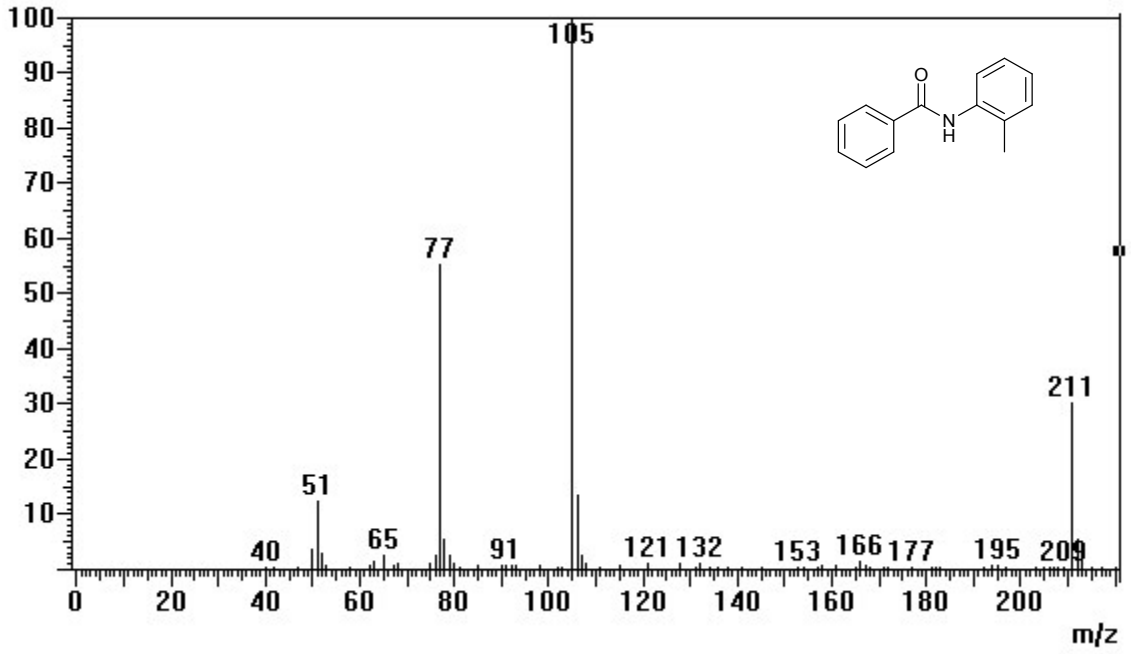
N-(4- Methyl phenyl)benzamide



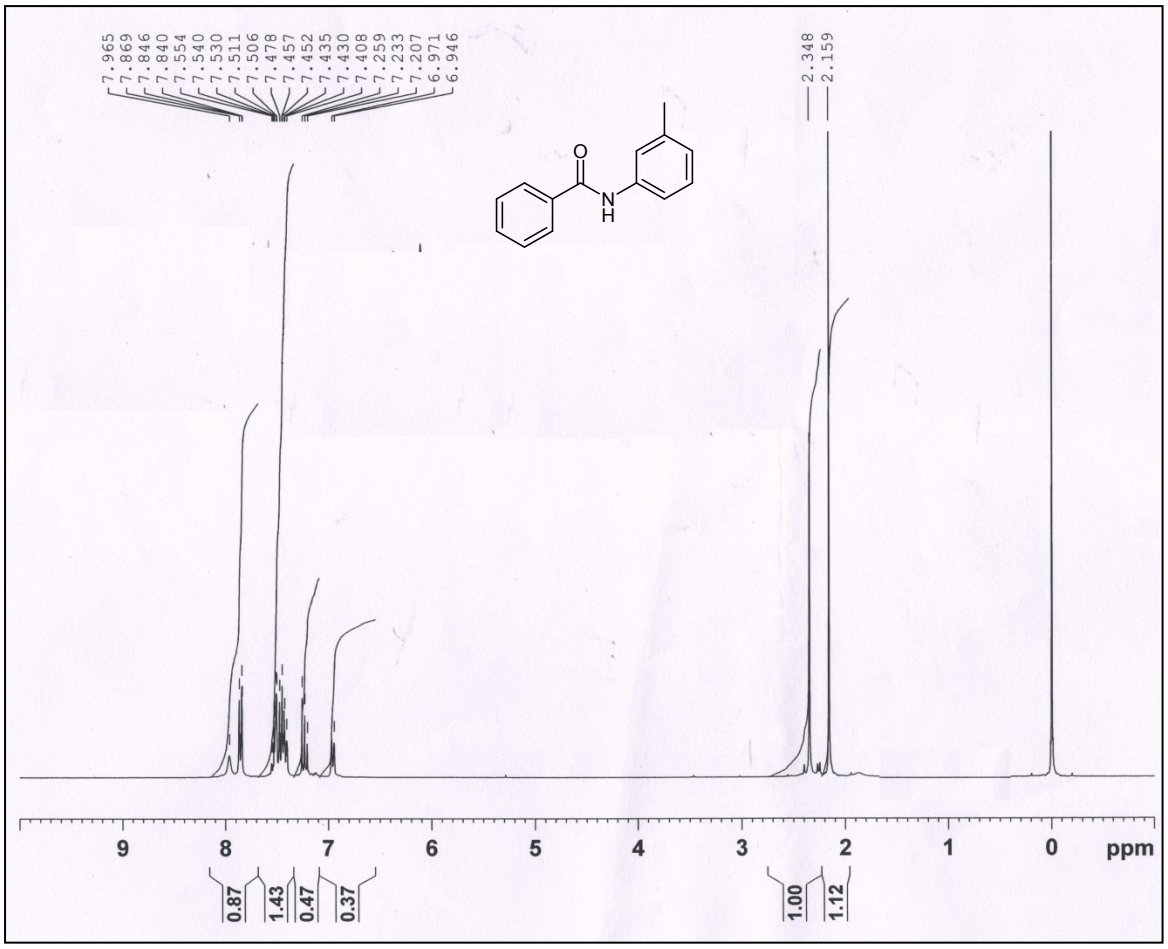


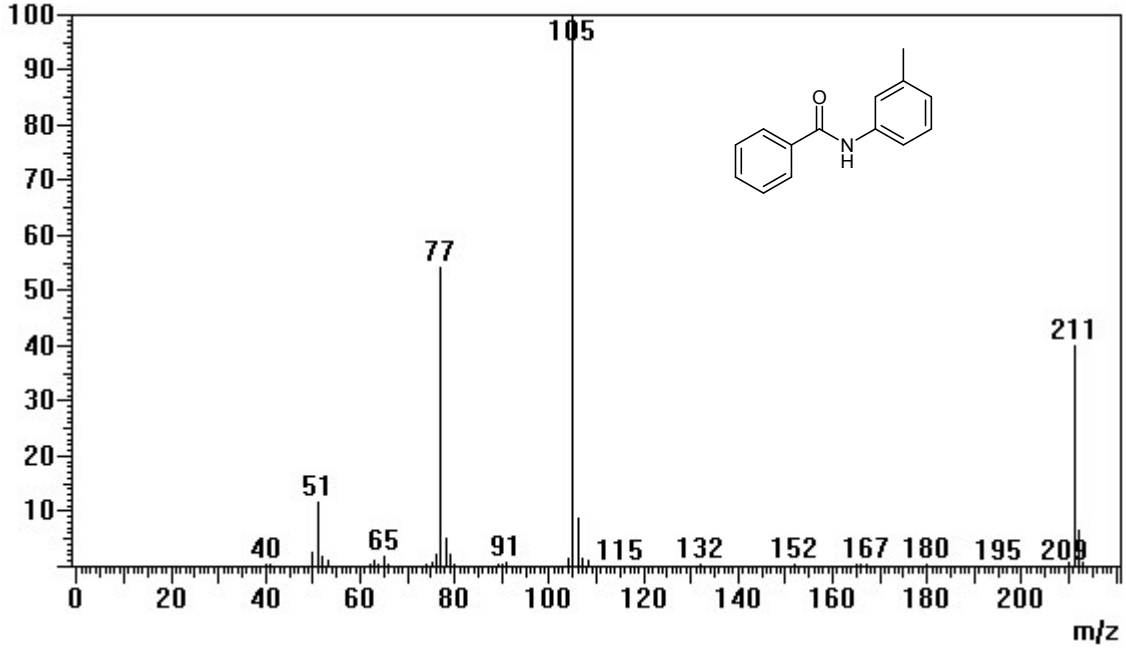
3 N-(2-Methyl phenyl) benzamide



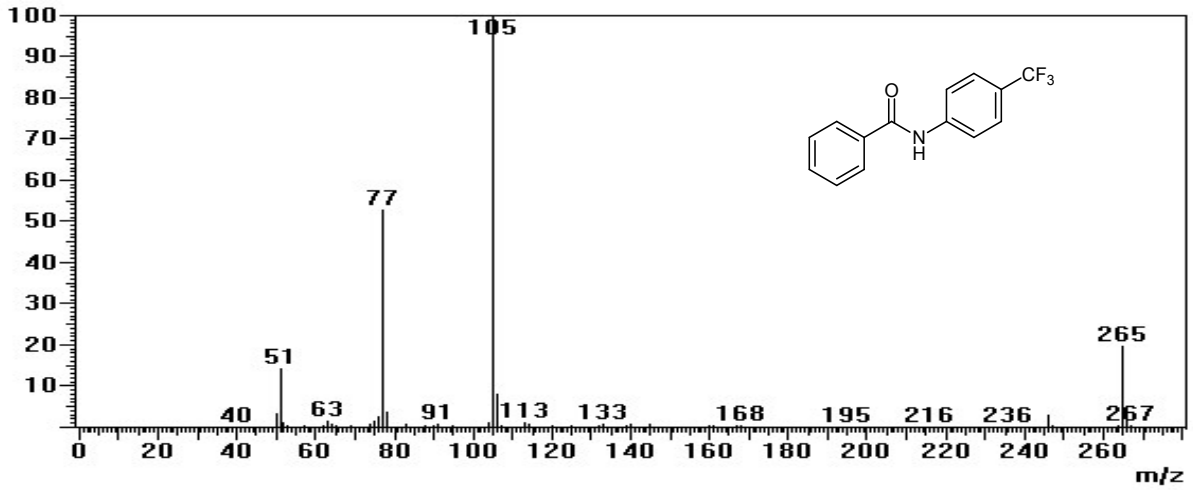


N- (3-Methyl phenyl) benzamide (3f)

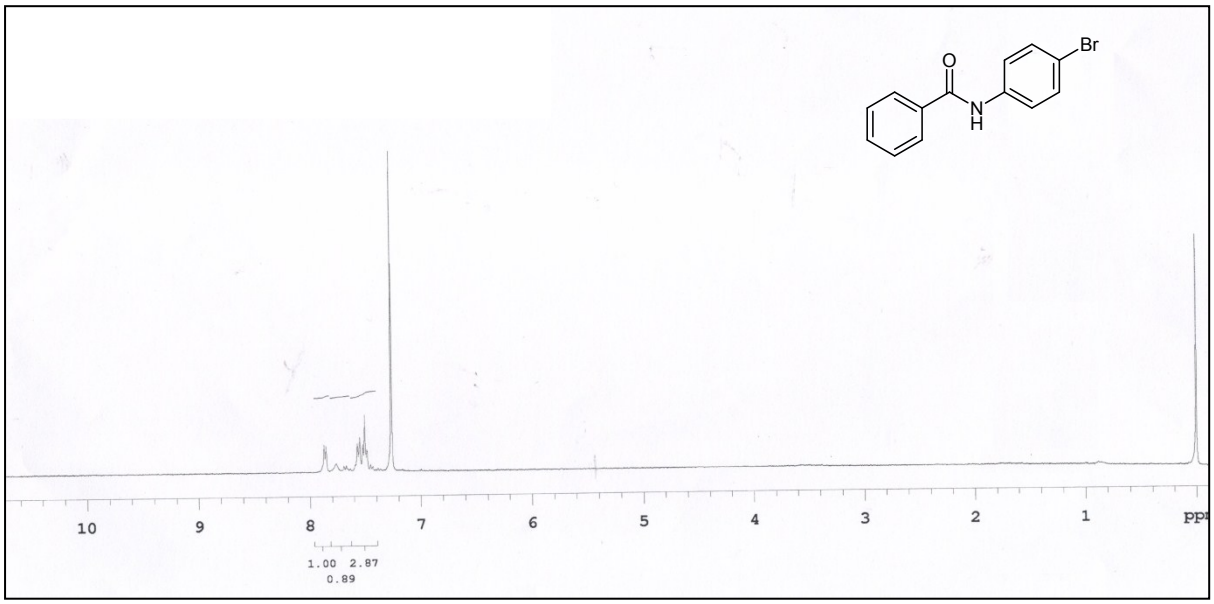


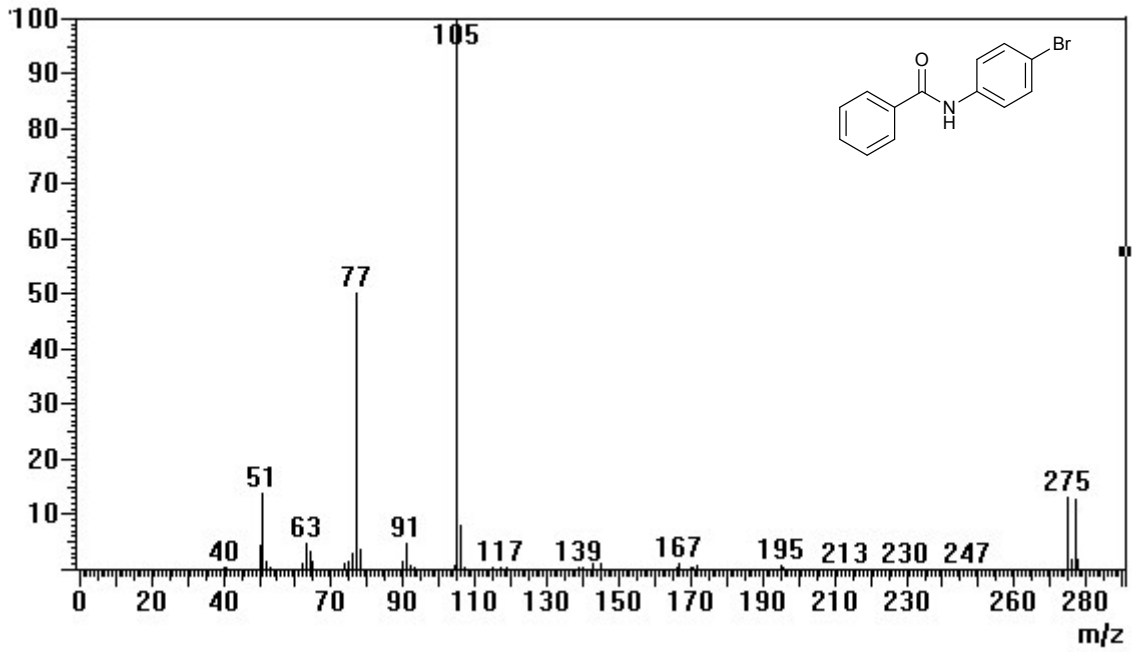


N-(4-(Trifluoro methyl) phenyl) benzamide) (3j)

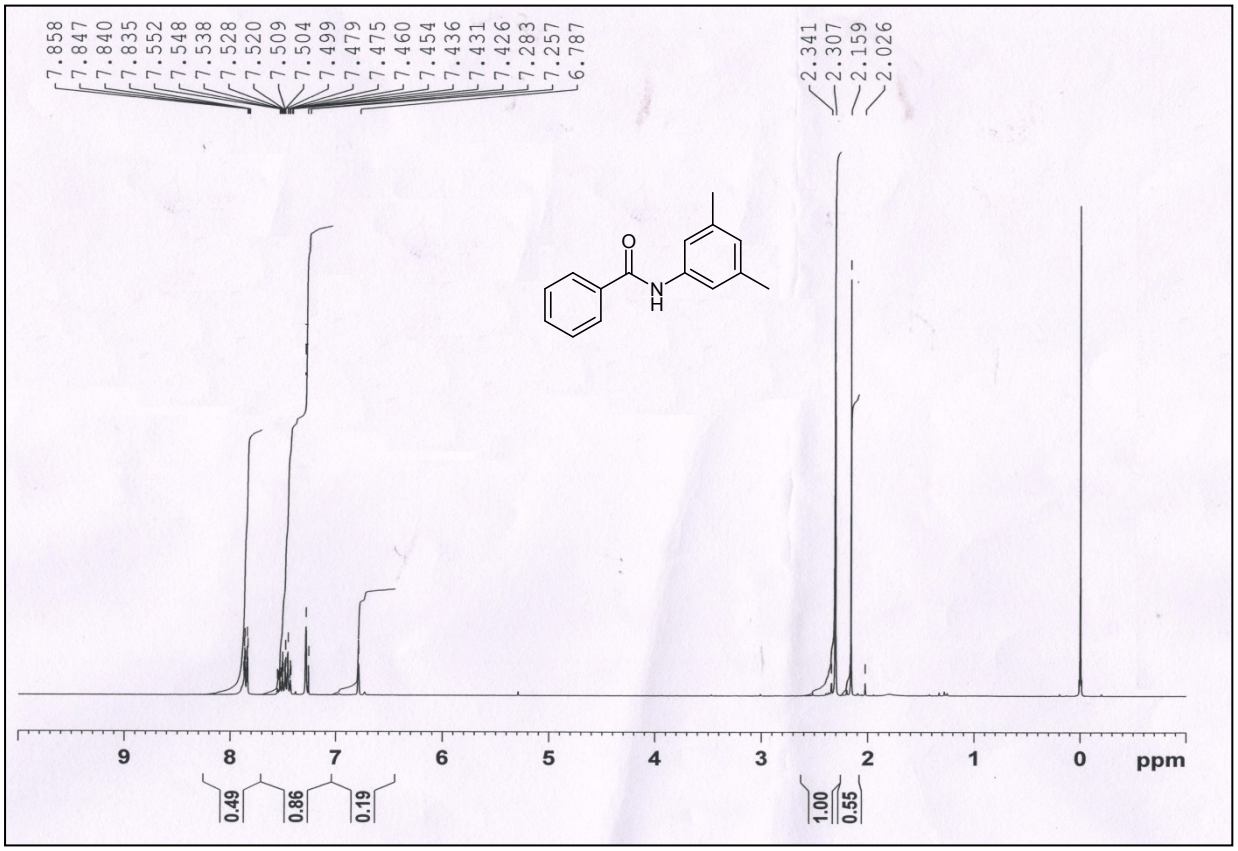


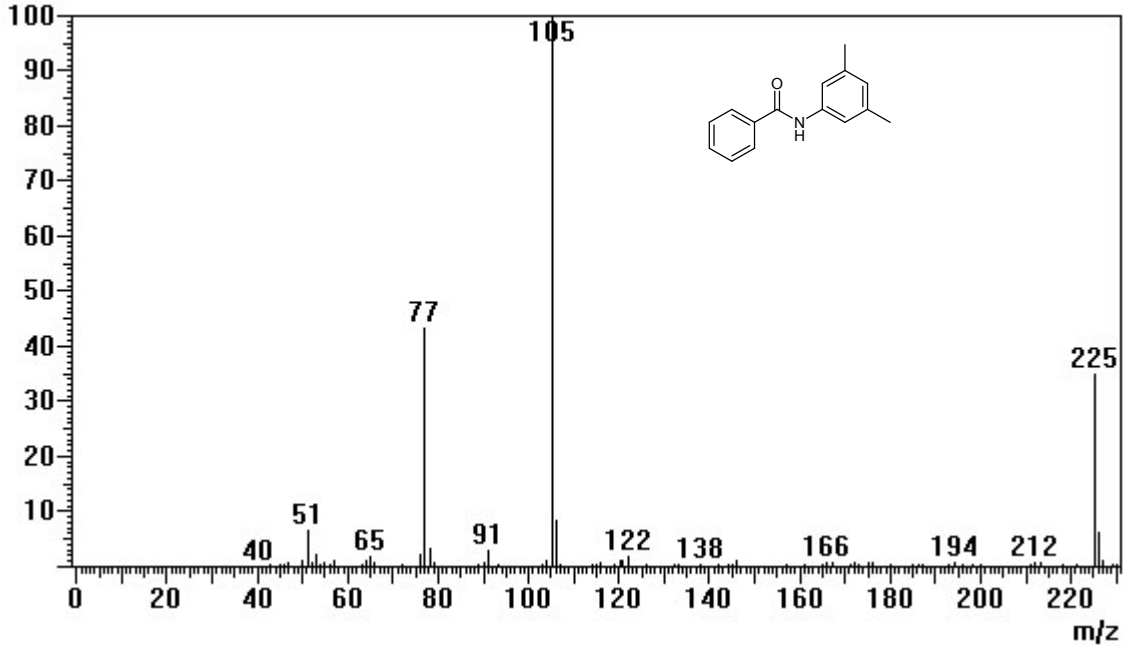
N-(4- Bromo phenyl) benzamide (3k)



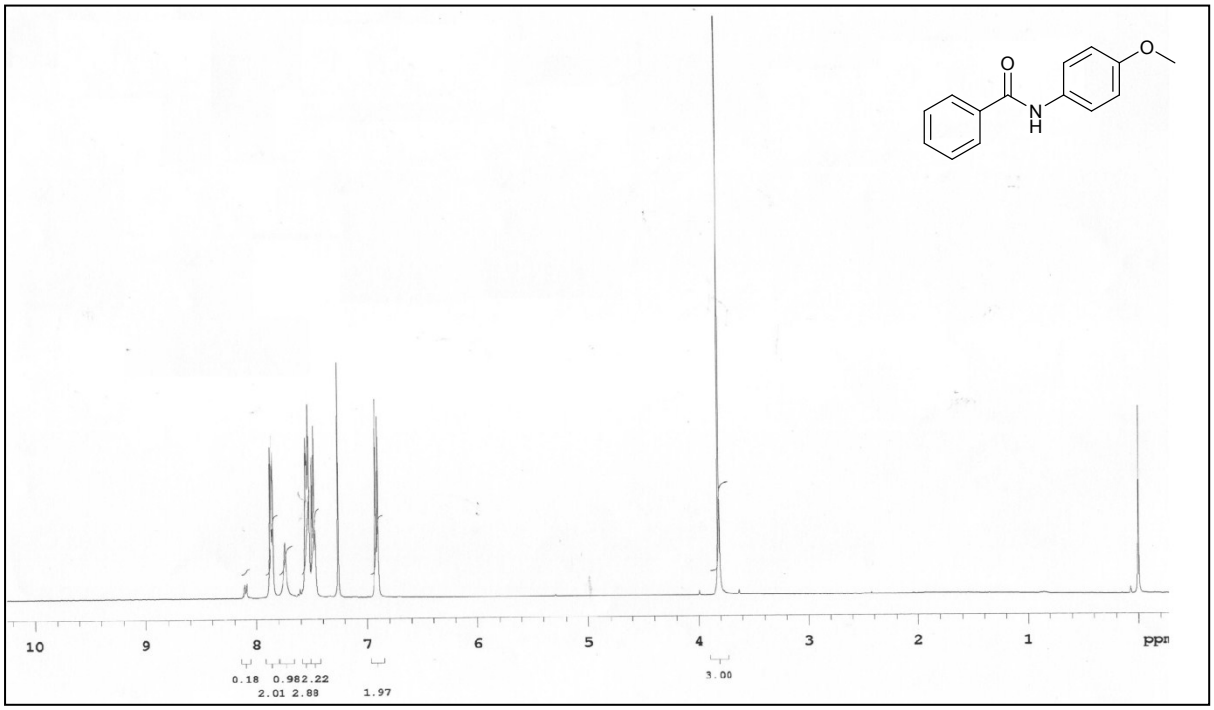


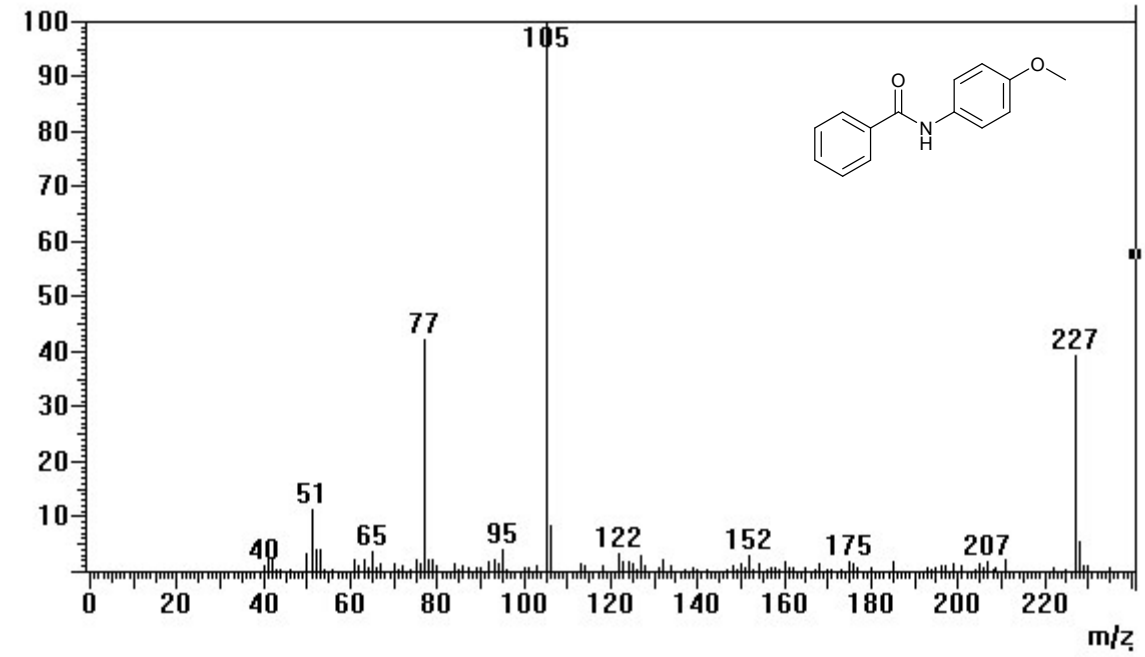
N-(3, 5-Dimethyl phenyl) benzamide





N- (4-Methoxy phenyl) benzamide

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m/z
O
N
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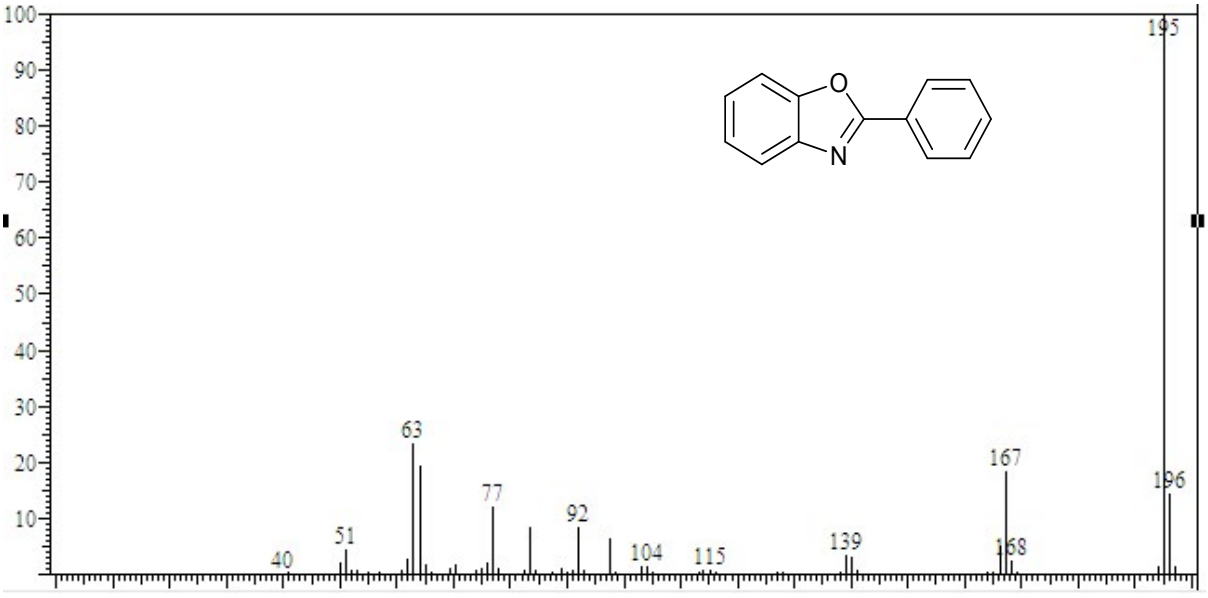
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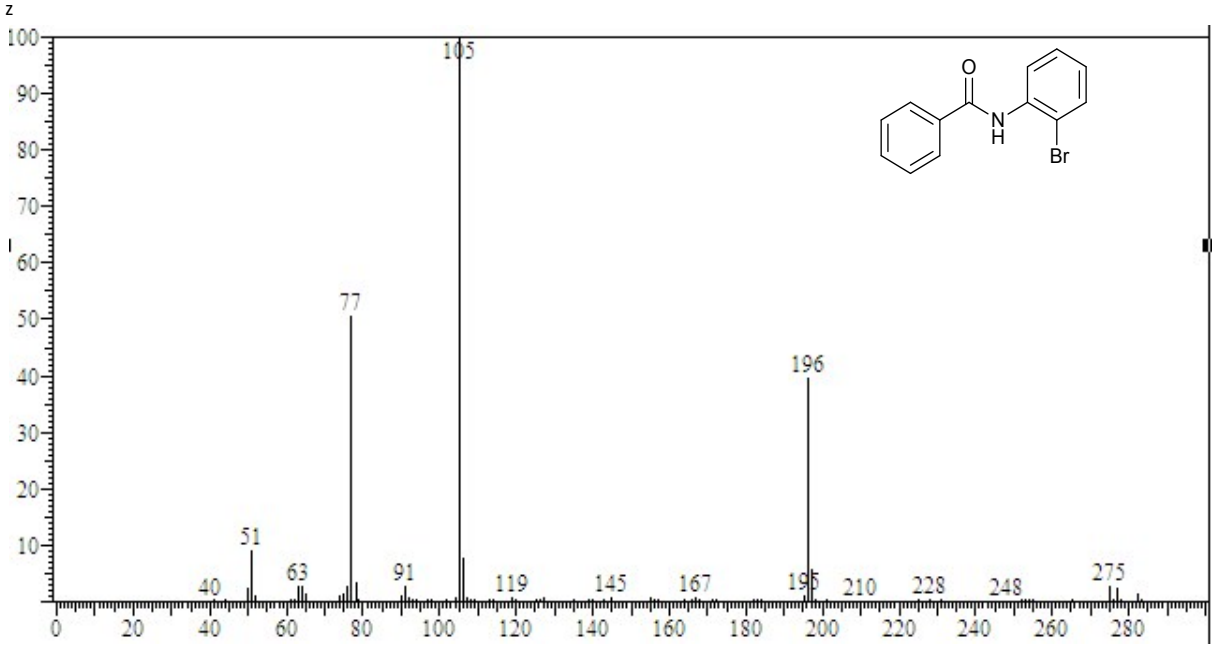
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H
Br