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SUPPORTING INFORMATION

Highly efficient synthesis of amides from ketoximes using trifluoromethanesulfonic anhydride

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EXPERIMENTAL (GENERAL)

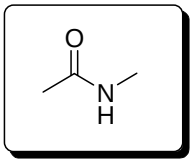
The reagents employed were of high purity commercial samples which were used as received (Fischer, Merck and Sigma Aldrich). Reactions were carried out in oven-dried RB flask. Column chromatography was performed on silica gel (200-400 mesh). TLC was performed on alumina silica gel $60F_{254}$ (Fischer) detected by UV light (254 nm) and iodine vapors. The melting points were determined by open capillaries on a Buchi apparatus and are uncorrected. The IR spectra were recorded on a Nicolet-Impact-410 FT-IR spectrometer, using KBr pellets. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker AC-300F, 300 MHz, spectrometer in DMSO-d_6 using TMS as an internal standard with ^{1}H resonance frequency of 300 MHz, ^{13}C resonance frequency of 75 MHz. GC analyses were performed on Nucon 5700 series Gas chromatograph. GC-MS analyses were performed on Shimadzu 2010 series mass selective detector instrument. Elemental analysis was carried out by using Heraus CHN rapid analyzer. All the compounds gave C, H and N analysis within ± 0.4% of the theoretical values. Dry DCM was obtained from commercial source by the standard procedure.

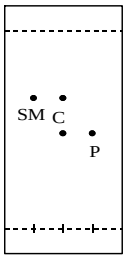
Typical procedure for the synthesis of amides using triflic anhydride

The desired ketoxime (2.0 mmol) in 5 mL dry DCM was taken in an oven-dried RB flask. To the reaction mixture was added drop wise triflic anhydride (2.0 mmol) in DCM (5mL) under nitrogen for 10 to 15 minutes. The reaction mixture was stirred at RT and the progress of the reaction was monitored by TLC and GC-MS (Table 1). After completion of reaction, the contents were poured to crushed ice (100 mL) and neutralized with 10% $NaHCO_3$ solution (20 mL) and extracted with DCM (15 mL x 3). The pure products were obtained by column chromatography with hexane-ethyl acetate and DCM-methanol mixture. All the amides were characterized by GC-MS, ^{1}H, ^{13}C NMR and by elemental analysis and the results are compared with authentic samples.

Characterization data of various amides

N-Methylacetamide (1) [1, 2, 3, 4, 5, 6]

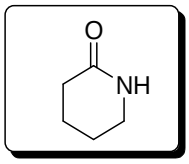


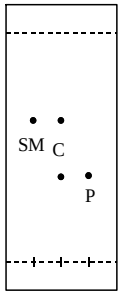


Yield: 170 mg (72%); colorless liquid, MP = 204-206 ^{O}C; Rf: 0.48 (80:20 Hexane:EtOAc, Iodine vapors); IR (KBr): 3345, 1660, 1246, 1028, 822 cm^{-1}. ^{1}H NMR (300MHz, DMSO-d_6): δ = 2.18 (s, 3H, CH_3), 2.83 (s, 3H, CH_3), 10.31 (s, 1H, NH). ^{13}C NMR (75MHz, DMSO-d_6): δ = 168.2, 28.7, 23.4 GC-MS: m/z 73 $[M]^+$. Anal. Calc. For C_3H_7NO: C, 49.30; H, 9.65; N, 19.16% found: C, 49.28; H, 9.62; N, 19.15 %.

Note: TLC Plate, SM= Starting material, C = Co spot, P = Product

Piperidin-2-one (2) [1, 2, 3, 5]

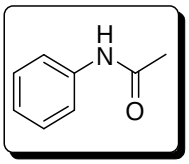


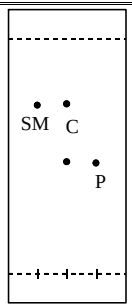


Yield: 190 mg (73%); colorless liquid, Rf: 0.37 (80:20 Hexane:EtOAc, Iodine vapors); IR (KBr): 3342, 1672, 1541, 1308, 1132, 796 cm^{-1}. ^{1}H NMR (300MHz, DMSO-d_6): δ = 1.62 (m, 4H), 2.34 (m, 2H), 3.36 (m, 2H), 10.44 (s, 1H, NH). ^{13}C NMR (75MHz, DMSO-d_6): δ = 169.1, 38.8, 36.2, 26.5, 24.2. GC-MS: m/z 99 $[M]^+$. Anal. Calc. For C_5H_9NO: C, 60.58; H, 9.15; N, 14.13% found: C, 60.57; H, 9.14; N, 14.13 %.

Note: TLC Plate, SM= Starting material, C = Co spot, P= Product

_N_-Phenylacetamide (4) [1, 2, 3, 4, 5, 6]

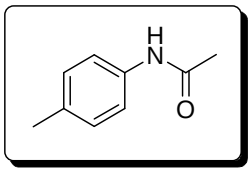


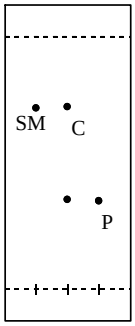


Yield: 288 mg (96%); colorless solid, MP = 114-116 ^{O}C; Rf: 0.47 (95:5, DCM:MeOH, UV); IR (KBr): 3351, 3031, 1669, 1582, 1530, 1474, 1421, 1285, 1230, 698 cm^{-1}. ^{1}H NMR (300MHz, DMSO-d_6): δ = 2.48 (s, 3H, CH_3), 7.75 (m, 5H), 10.15 (s, 1H, NH). ^{13}C NMR (75MHz, DMSO-d_6): δ = 167.1, 138.8, 128.3, 127.6, 124.1, 121.9, 121.6, 22.9. GC-MS: m/z 135 [M]$^{+}$. Anal. Calc. For C_8H_9NO: C, 71.09; H, 6.71; N, 10.36% found: C, 71.07; H, 6.70; N, 10.34 %.

Note: TLC Plate, SM= Starting material, C= Co spot, P= Product

_N-p_-tolylacetamide (5) [1, 2, 3, 4]

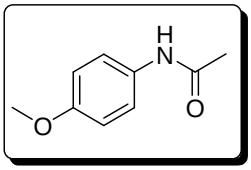


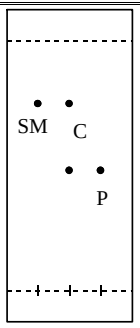


Yield: 245 mg (92%); colorless solid, MP = 145-148 ^{O}C; Rf: 0.35 (98:2, DCM:MeOH, UV); IR (KBr): 3331, 1665, 1573, 1394, 1310, 921, 719 cm^{-1}. ^{1}H NMR (300MHz, DMSO-d_6): δ = 2.32 (s, 3H, CH_3), 2.38 (s, 3H, CH_3), 6.87 (d, 2H, j = 8.5 Hz), 7.42 (d, 2H, j = 8.5 Hz), 10.34 (s, 1H, NH). ^{13}C NMR (75MHz, DMSO-d_6): δ = 168.4, 142.5, 135.2, 129.6, 122.1, 24.6, 22.8. GC-MS: m/z 149 [M]$^{+}$. Anal. Calc. For $C_9H_{11}NO$: C, 72.46; H, 7.43; N, 9.39% found: C, 72.45; H, 7.41; N, 9.40 %.

Note: TLC Plate, SM= Starting material, C = Co spot, P= Product

_N_-(4-Methoxyphenyl)acetamide (6) [1, 3, 4]

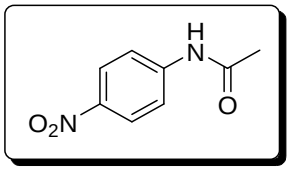


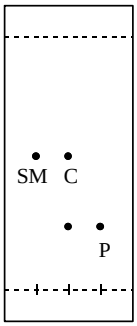


Yield: 286 mg (96%); colorless solid, MP = 130-132 ^{O}C; Rf: 0.48 (95:5 DCM:MeOH, UV); IR (KBr): 3341, 1669, 1576, 1343, 1320, 887, 689 cm^{-1}. ^{1}H NMR (400MHz, DMSO-d_6): δ = 2.37 (s, 3H, CH_3), 3.86 (s, 3H, OCH_3), 6.89 (d, 2H, j = 8.5 Hz), 7.46 (d, 2H, j = 8.5 Hz), 9.97 (s, 1H, NH). ^{13}C NMR (100MHz, DMSO-d_6): δ = 169.6, 154.5, 134.2, 130.2, 122.6, 56.4, 22.6. GC-MS: m/z 165 $[M]^+$. Anal. Calc. For $C_9H_{11}NO_2$: C, 65.44; H, 6.71; N, 8.48% found: C, 65.42; H, 6.70; N, 8.47 %.

Note: TLC Plate, SM= Starting material, C = Co spot, P= Product

_N_-(4-Nitrophenyl)acetamide (7) [1, 2, 3, 4]

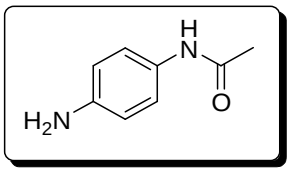


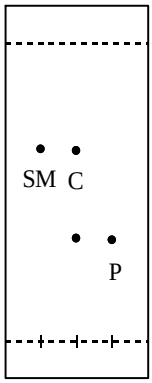


Yield: 266 mg (94%); pale yellow solid, MP = 215-218 ^{O}C; Rf: 0.25 (95:5, DCM:MeOH, UV); IR (KBr): 3320, 1667, 1565, 1380, 1302, 829, 834 cm^{-1}. ^{1}H NMR (400MHz, $CDCl_3$ + DMSO-d_6): δ = 2.30 (s, 3H, CH_3), 7.84 (d, 2H, j = 9.0 Hz), 8.40 (d, 2H, j = 9.0 Hz), 10.12 (s, 1H, NH). ^{13}C NMR (100MHz, $CDCl_3$ + DMSO-d_6): δ = 169.1, 145.7, 142.8, 124.6, 122.1, 22.2. GC-MS: m/z 180 $[M]^+$. Anal. Calc. For $C_8H_8N_2O_3$: C, 53.33; H, 4.48; N, 15.55% found: C, 53.32; H, 4.46; N, 15.54 %.

Note: TLC Plate, SM= Starting material, C= Co spot, P= Product

_N_-(4-Aminophenyl)acetamide (8) [1, 2, 4, 6]

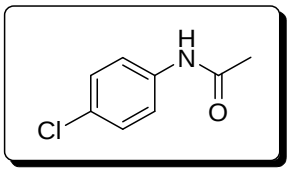


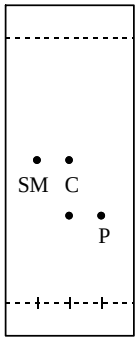


Yield: 285 mg (94%); colorless solid, MP = 165-167 ^{O}C; Rf: 0.34 (95:5, DCM:MeOH, UV); IR (KBr): 3340, 1672, 1580, 1323, 1314, 857, 670 cm^{-1}. ^{1}H NMR (300MHz, DMSO-d_6): δ = 2.27 (s, 3H, CH_3), 6.68 (d, 2H, j = 8.5 Hz), 7.42 (d, 2H, j = 8.5 Hz), 10.18 (s, 1H, NH). ^{13}C NMR (75MHz, DMSO-d_6): δ = 170.2, 142.5, 130.6, 122.8, 118.3, 22.8. GC-MS: m/z 150 $[M]^+$. Anal. Calc. For $C_8H_{10}N_2O$: C, 63.98; H, 6.71; N, 18.65% found: C, 63.97; H, 6.70; N, 18.66 %.

Note: TLC Plate, SM= Starting material, C= Co spot, P= Product

_N_-(4-Chlorophenyl)acetamide (9) [1, 2, 3]

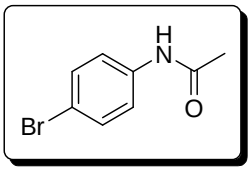


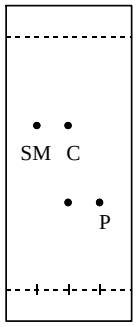


Yield: 288 mg (95%); colorless solid, MP = 176-178 ^{O}C; Rf: 0.33 (95:5, DCM:MeOH, UV); IR (KBr): 3342, 1670, 1590, 1353, 1322, 890, 667 cm^{-1}. ^{1}H NMR (400MHz, DMSO-d_6): δ = 2.38 (s, 3H, CH_3), 7.42 (d, 2H, j = 9.0 Hz), 7.60 (d, 2H, j = 9.0 Hz), 10.28 (s, 1H, NH). ^{13}C NMR (100MHz, DMSO-d_6): δ = 168.4, 138.8, 130.9, 129.4, 124.3, 23.4. GC-MS: m/z 169 $[M]^+$. Anal. Calc. For C_8H_8ClNO: C, 56.65; H, 4.75; N, 8.26% found: C, 56.66; H, 4.74; N, 8.25 %.

Note: TLC Plate, SM= Starting material, C = Co spot, P= Product

_N_-(4-Bromophenyl)acetamide (10)[1, 2]

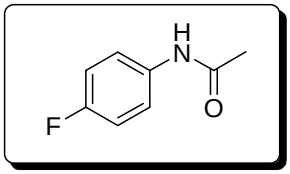


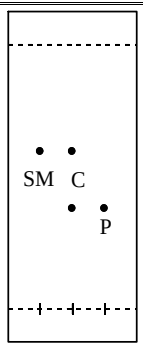


Yield: 268 mg (90%); brown solid, MP = 163-165 ^{O}C; Rf: 0.34 (95:5, DCM:MeOH, UV); IR (KBr): 3336, 1665, 1570, 1323, 1310, 812, 654 cm^{-1}. ^{1}H NMR (400MHz, DMSO-d_6): δ = 2.18 (s, 3H, CH_3), 7.40 (d, 2H, j = 9.0 Hz), 7.68 (d, 2H, j = 9.0 Hz), 9.98 (s, 1H, NH). ^{13}C NMR (100MHz, DMSO-d_6): δ = 169.2, 138.8, 131.2, 125.4, 121.3, 23.8. GC-MS: m/z 213 $[M-1]^+$, 215 $[M+1]^+$ Anal. Calc. For C_8H_8BrNO: C, 44.89; H, 3.77; N, 6.54% found: C, 44.88; H, 3.75; N, 6.55 %.

Note: TLC Plate, SM= Starting material, C = Co spot, P= Product

_N_-(4-Fluorophenyl)acetamide (11)[1, 2]

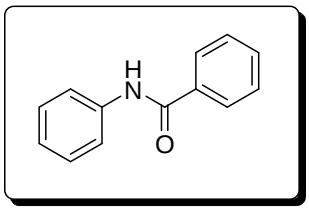


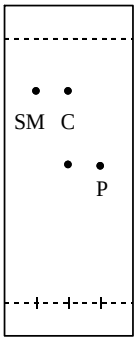


Yield: 280 mg (92%); colorless solid, MP = 153-155 ^{O}C; Rf: 0.38 (95:5, DCM:MeOH, UV); IR (KBr): 3330, 1668, 1530, 1345, 1312, 845, 656 cm^{-1}. ^{1}H NMR (400MHz, DMSO-d_6): δ = 2.10 (s, 3H, CH_3), 7.12 (d, 2H, j = 9.0 Hz), 7.80 (d, 2H, j = 9.0 Hz), 10.38 (s, 1H, NH). ^{13}C NMR (100MHz, DMSO-d_6): δ = 169.2, 159.8 (C-F, J = 245.20Hz), 134.3, 124.3, 116.4, 22.8. GC-MS: m/z 153 $[M]^+$. Anal. Calc. For C_8H_8FNO: C, 62.74; H, 5.26; N, 9.15% found: C, 62.75; H, 5.24; N, 9.15 %.

Note: TLC Plate, SM= Starting material, C= Co spot, P= Product

_N_-phenylbenzamide (12) [1, 2, 3, 4, 5, 6]

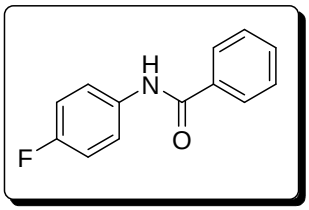


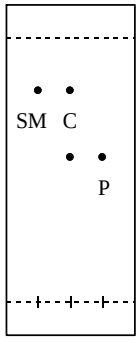


Yield: 285 mg (96%); colorless solid, MP = 161-163 ^{O}C; Rf: 0.52 (80:20, Hexane:EtOAc, UV); IR (KBr): 3359, 1660, 1580, 1530, 1478, 1420, 1320, 1285, 688 cm^{-1}. ^{1}H NMR (300MHz, DMSO-d_6): δ = 7.35 (m, 2H), 7.62 (m, 3H), 7.86 (m, 2H), 8.10 (m, 3H), 10.32 (s, 1H, NH). ^{13}C NMR (75MHz, DMSO-d_6): δ = 166.3, 136.8, 134.8, 132.3, 131.6, 129.1, 128.9, 128.4, 127.6, 122.9, 121.4, 120.6. GC-MS: m/z 197 [M]$^{+}$. Anal. Calc. For $C_{13}H_{11}NO$: C, 79.16; H, 5.62; N, 7.10% found: C, 79.17; H, 5.60; N, 7.10 %.

Note: TLC Plate, SM= Starting material, C= Co spot, P= Product

_N_-(4-Fluorophenyl)benzamide (13) [1, 2, 4]

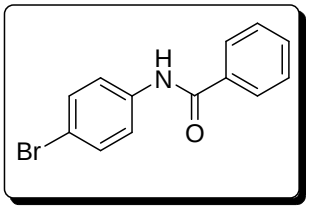


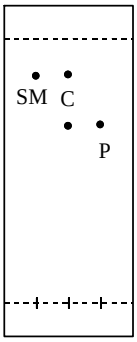


Yield: 260 mg (88%); colorless solid, MP = 169-172 ^{O}C; Rf: 0.55 (80:20, Hexane:EtOAc, UV); IR (KBr): 3341, 1652, 1535, 1325, 1312, 845, 650 cm^{-1}. ^{1}H NMR (300MHz, DMSO-d_6): δ = 7.05 (d, 2H, j = 9.0 Hz), 7.54 (m, 2H), 7.68 (d, 2H, j = 9.0 Hz), 7. 95 (m, 3H), 10.18 (s, 1H, NH). ^{13}C NMR (75MHz, DMSO-d_6): δ = 166.6, 160.2, (C-F, J = 246.12Hz), 135.2, 131.6, 130.7, 129.8, 129.3, 128.5, 128.0, 127.4, 124.6, 123.4, 116.8. GC-MS: m/z 215 [M]$^{+}$. Anal. Calc. For $C_{13}H_{10}FNO$: C, 72.55; H, 4.68; N, 6.51% found: C, 72.55; H, 4.66; N, 6.50 %.

Note: TLC Plate, SM= Starting material, C= Co spot, P= Product

_N_-(4-Bromophenyl)benzamide (14)[1, 2, 3]

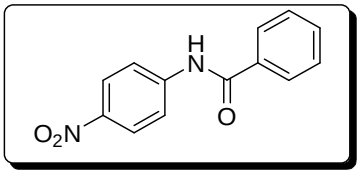


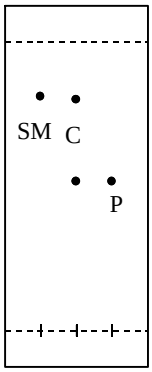


Yield: 266 mg (90%); pale yellow solid, MP = 162-165 ^{O}C; Rf: 0.67 (80:20, Hexane:EtOAc, UV); IR (KBr): 3334, 1656, 1540, 1320, 1332, 840, 760 cm^{-1}. ^{1}H NMR (300MHz, DMSO-d_6): δ = 7.36 (d, 2H, j = 9.0 Hz), 7.58 (m, 2H), 7.72 (d, 2H, j = 9.0 Hz), 8. 16 (m, 3H), 10.28 (s, 1H, NH). ^{13}C NMR (75MHz, DMSO-d_6): δ = 164.5, 159.4, 136.2, 132.6, 130.7, 129.8, 129.0, 128.5, 128.1, 127.2, 123.8, 123.2, 118.4. GC-MS: m/z 275 [M-1]$^{+}$, 277 [M+1]$^{+2}$ Anal. Calc. For $C_{13}H_{10}BrNO$: C, 56.55; H, 3.65; N, 5.07% found: C, 56.53; H, 3.64; N, 5.05 %.

Note: TLC Plate, SM= Starting material, C= Co spot, P= Product

_N_-(4-Nitrorophenyl)benzamide (15)[1, 2]

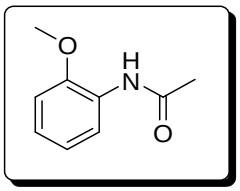


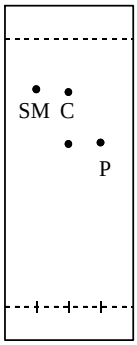


Yield: 256 mg (87%); pale yellow solid, MP = 202-204 ^{O}C; Rf: 0.611 (98:2, DCM:MeOH, UV); IR (KBr): 3333 1650, 1563, 1325, 1312, 1280, 945, 780, 694 cm^{-1}. ^{1}H NMR (300MHz, DMSO-d_6): δ = 7.56 (m, 5H), 7.80 (d, 2H, j = 8.5 Hz), 8.34 (d, 2H, j = 8.5 Hz), 9.80 (s, 1H, NH). ^{13}C NMR (75MHz, DMSO-d_6): δ = 164.6, 148.2, 142.2, 132.6, 131.1, 129.8, 129.3, 128.7, 128.1, 127.4, 124.6, 123.4, 121.2. GC-MS: m/z 242 [M]$^{+}$. Anal. Calc. For $C_{13}H_{10}N_2O_3$: C, 64.46; H, 4.16; N, 11.56% found: C, 64.45; H, 4.16; N, 11.54 %.

Note: TLC Plate, SM= Starting material, C= Co spot, P= Product

_N_-(2-Methoxyphenyl)acetamide (16) [1, 3, 4]

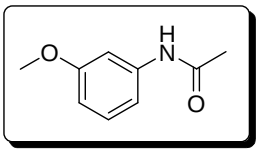


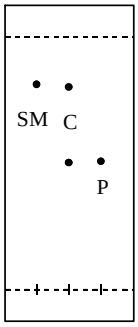


Yield: 248 mg (86%); colorless solid, MP = 87-90 ^{O}C; Rf: 0.611 (98:2, DCM:MeOH, UV); IR (KBr): 3340, 3331, 1674, 1580, 1520, 1466, 1411, 1280, 1230, 792 cm^{-1}. ^{1}H NMR (300MHz, DMSO-d_6): δ = 2.42 (s, 3H, CH_3), 3.86 (s, 3H, OCH_3), 6.96 (m, 4H), 10.25 (s, 1H, NH). ^{13}C NMR (75MHz, DMSO-d_6): δ = 171.1, 153.2, 126.3, 124.1, 121.9, 121.6, 113.4, 56.8, 22.9. GC-MS: m/z 165 $[M]^+$. Anal. Calc. For C_8H_9NO: C, 71.09; H, 6.71; N, 10.36% found: C, 71.08; H, 6.71; N, 10.35 %.

Note: TLC Plate, SM= Starting material, C = Co spot, P= Product

_N_-(3-Methoxyphenyl)acetamide (17) [3, 4]

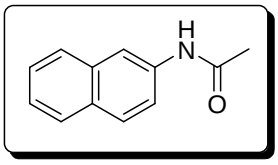


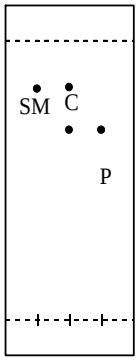


Yield: 265 mg (90%); colorless solid, MP = 103-105 ^{O}C; Rf: 0.514 (98:2, DCM:MeOH, UV); IR (KBr): 3338, 1670, 1540, 1530, 1468, 1421, 1230, 1218, 754 cm^{-1}. ^{1}H NMR (300MHz, DMSO-d_6): δ = 2.36 (s, 3H, CH_3), 3.92 (s, 3H, OCH_3), 6.72 (d, 2H, j = 8.5 Hz), 7.76 (m, 2H), 10.15 (s, 1H, NH). ^{13}C NMR (75MHz, DMSO-d_6): δ = 170.6, 161.5, 140.4, 131.6, 114.4, 110.3, 105.6, 55.4, 24.1. GC-MS: m/z 165 $[M]^+$. Anal. Calc. For C_8H_9NO: C, 71.09; H, 6.71; N, 10.36% found: C, 71.07; H, 6.71; N, 10.34 %.

Note: TLC Plate, SM= Starting material, C= Co spot, P= Product

***N*-(Naphthalene-2-yl)acetamide (18)** [5]

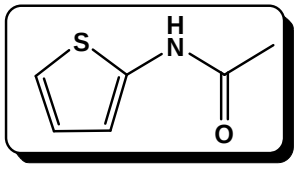


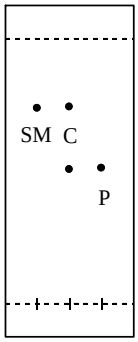


Yield: 230 mg (85%); colorless solid, MP = 176-178 ^{O}C; Rf: 0.36 (80:20, Hexane:EtOAc, UV); IR (KBr): 3331, 3020, 1668, 1584, 1530, 1424, 1410, 1235, 1250, 658 cm^{-1}. ^{1}H NMR (300MHz, DMSO-d_6): δ = 2.18 (s, 3H, CH_3), 6.75 (m, 2H), 7.10 (m, 2H), 7.89 (m, 3H), 10.10 (s, 1H, NH). ^{13}C NMR (75MHz, DMSO-d_6): δ = 170.2, 136.4, 132.4, 127.8, 126.4, 125.8, 124.6, 124.1, 121.9, 118.6, 110.3, 23.2. GC-MS: m/z 185 $[M]^+$. Anal. Calc. For $C_{12}H_{11}NO$: C, 77.81; H, 5.99; N, 7.56% found: C, 77.80; H, 5.98; N, 7.54 %.

Note: TLC Plate, SM= Starting material, C = Co spot, P= Product

***N*-(Thiophen-2-yl)acetamide (19)** [2, 3]

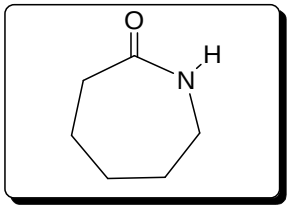


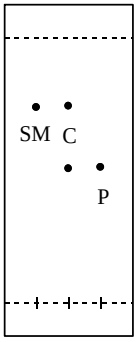


Yield: 230 mg (85%); colorless solid, MP = 159-161 ^{O}C; Rf: 0.36 (97:3, DCM:MeOH , UV); ^{1}H NMR (300MHz, DMSO-d_6): δ = 2.08 (s, 3H, CH_3), 6.75 (m, 1H), 6.90 (m, 1H), 7.38 (m, 1H), 10.24 (s, 1H, NH). ^{13}C NMR (75MHz, DMSO-d_6): δ = 171.3, 144.4, 126.6, 122.4, 112.8, 23.2. GC-MS: m/z 141 $[M]^+$. Anal. Calc. For C_6H_7NOS: C, 51.04; H, 5.00; N, 9.92 % found; C, 51.01; H, 4.98; N, 9.90 %

Note: TLC Plate, SM= Starting material, C = Co spot, P= Product

ε-Caprolactam (3) [1, 3, 4]





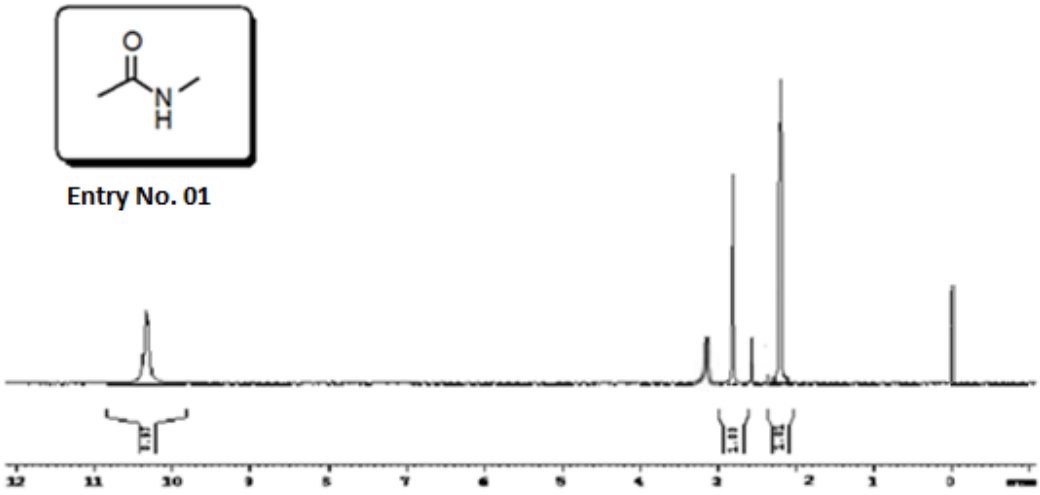
Yield: 230mg; colorless solid, MP = 68-71 ^{O}C; Rf: 0.51 (80:20, Hexane:EtOAc, Iodine vapors); ^{1}H NMR (300MHz, DMSO-d_6): δ= 1.2 (m, 2H, CH_2), 1.55 (m, 2H, CH_2), 2.15 (m, 2H, CH_2), 2.44 (m, 2H, CH_2), 10.08 (s, 1H, NH). ^{13}C NMR (75MHz, DMSO-d_6): δ = 179.2, 39.2, 35.8, 30.1, 29.6, 21.8. GC-MS: m/z 113 [M] $^{+}$. Anal. Calc. For $C_6H_{11}NO$: C, 63.68; H, 9.80; N, 12.38 % found; C, 63.70; H, 9.78; N, 12.35%

Note: TLC Plate, SM= Starting material, C = Co spot, P= Product

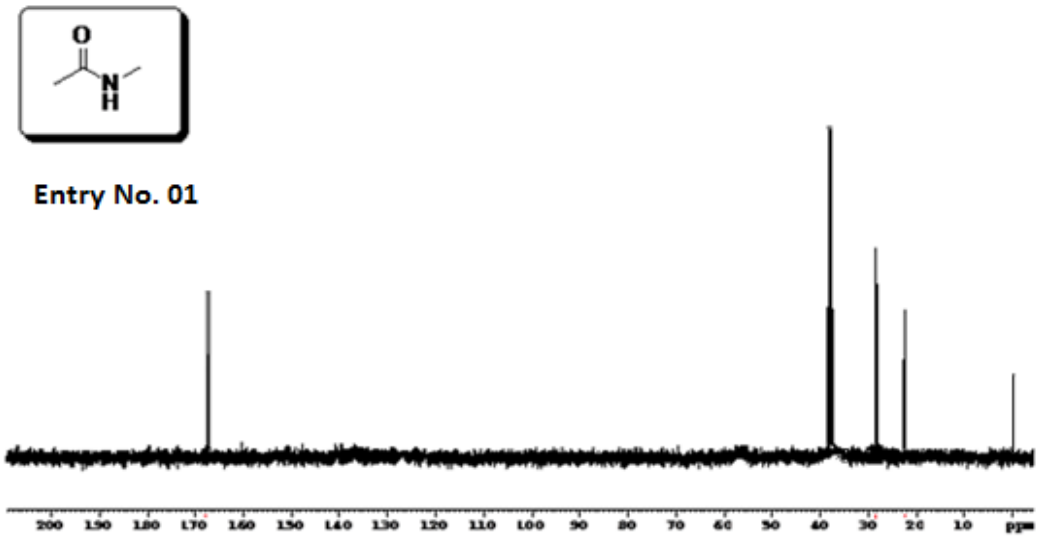
References and notes

1. Dongmei, L.; Feng, S.; Shu, G.; Youquan, D. *Tetrahedron Lett.* **2005**, *46*, 671.
2. Yadav, L. D. S.; Garima, V.; Srivastava, P. *Tetrahedron Lett.* **2010**, *51*, 739.
3. Minze, Z.; Chuantao, C.; Wei-Ping, D.; Xiao-Xin, S. *Tetrahedron Lett.* **2005**, *46*, 4861.
4. Yan, P.; Batamack, P.; Surya Prakash, G. K.; Olah, G. A. *Catal Lett.* **2005**, *103, 3-4*, 165.
5. Lin-fei, X.; Chun-gu, X.; Jing, C. *Tetrahedron Lett*. **2007**, *48*, 7218.
6. Li, Z.; Ding, R.; Lu, Z.; Xiao, S.; Ma, X. *J. Mol. Cat. A: Chem*. **2006**, *250*, 100.

^{1}H-NMR Spectra of *N*-Methylacetamide (Table No. 2, Entry No. 1)



^{13}C-NMR Spectra of *N*-Methylacetamide(Table No. 2, Entry No. 1)

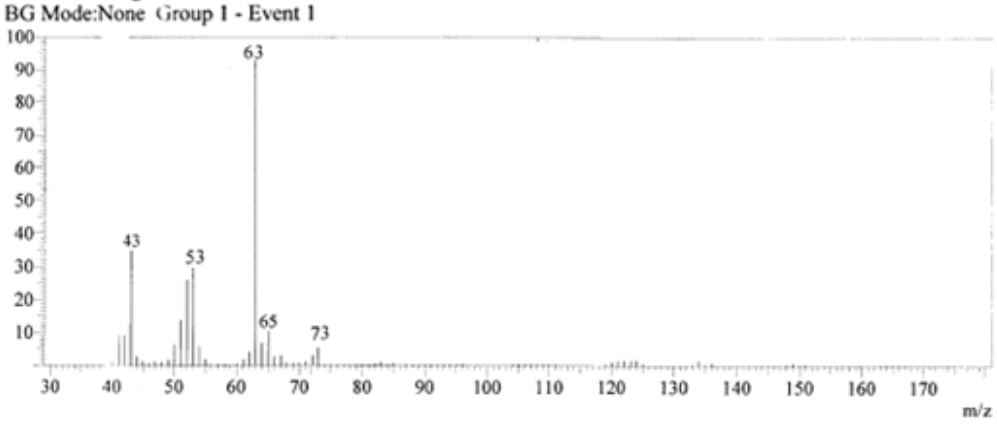


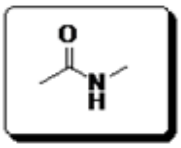
GCMS Spectra of *N*-Methylacetamide (Table No. 2, Entry No. 1)

KARNATAK UNIVERSITY
UNIVERSITY SCIENCE INSTRUMENTS CENTRE
DHARWAD

Sample Information

Analyzed by : Admin
Sample Type : Unknown
Level # : 1
Sample Name : SB 1A
Sample ID :
Line#:1 R.Time 0.1
MassPeaks:68
RawMode:Single 0.1
BG Mode:None Group 1 - Event 1

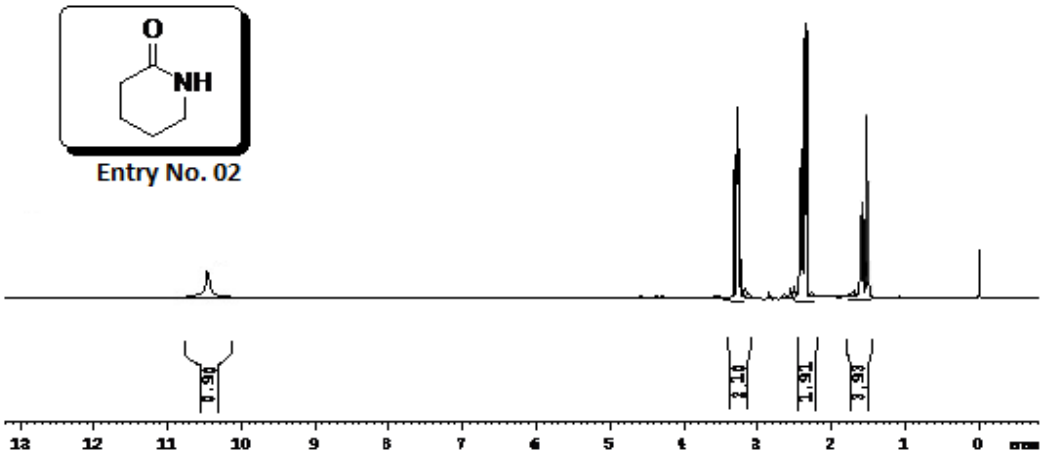




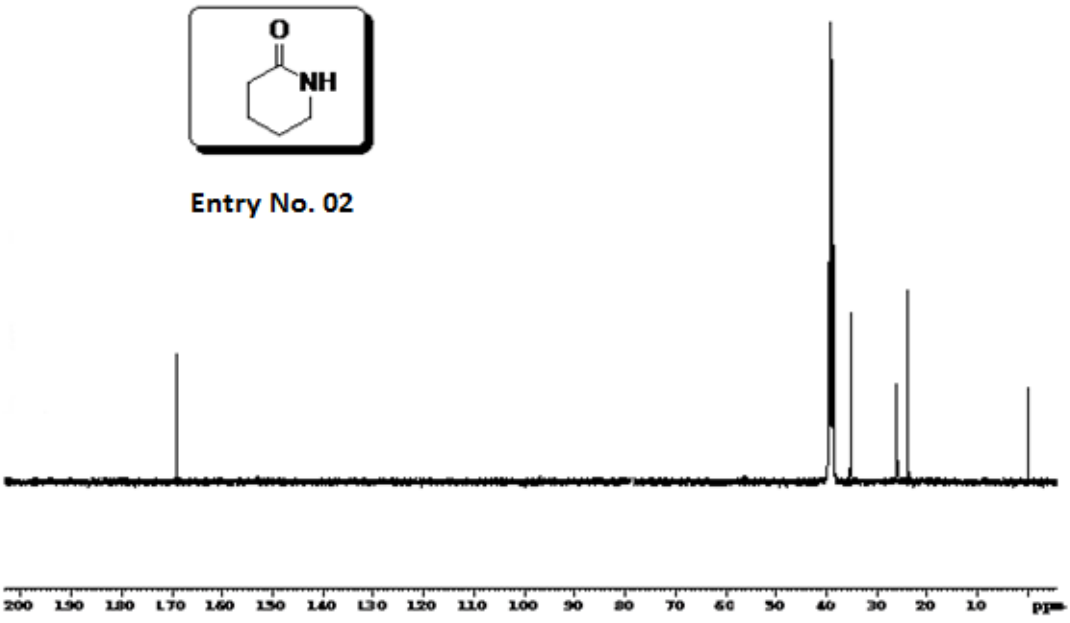
Mol. Weight = 73

Entry No. 01

^{1}H-NMR Spectra of Piperidin-2-one (Table No. 2, Entry No. 2)



^{13}C-NMR Spectra of Piperidin-2-one (Table No. 2, Entry No. 2)



GCMS Spectra of Piperidin-2-one (Table No. 2, Entry No. 2)

KARNATAK UNIVERSITY
UNIVERSITY SCIENCE INSTRUMENTS CENTRE
DHARWAD

Sample Information

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Sample Type : Unknown
Level # : 1
Sample Name : SB 2A
Sample ID :
IS Amount : [1]=1
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MassPeaks:77
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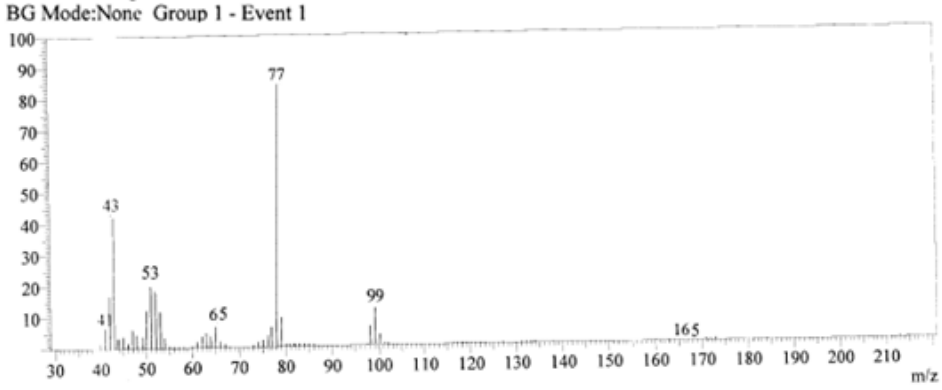
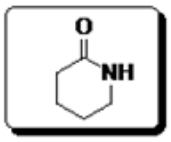
BG Mode:None Group 1 - Event 1

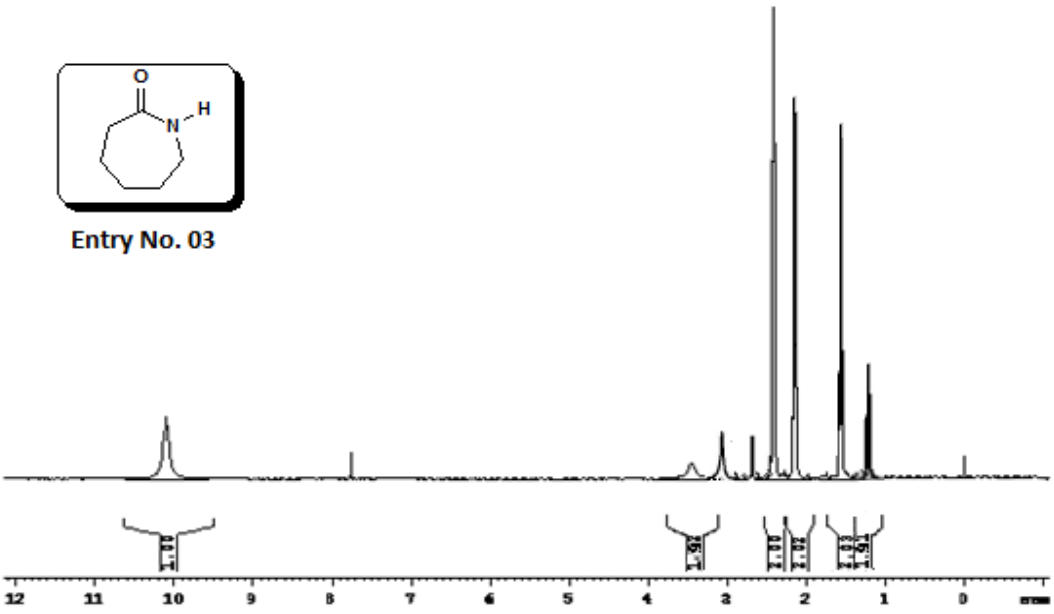




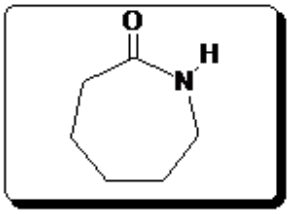
Mol. Weight = 99

Entry No. 02

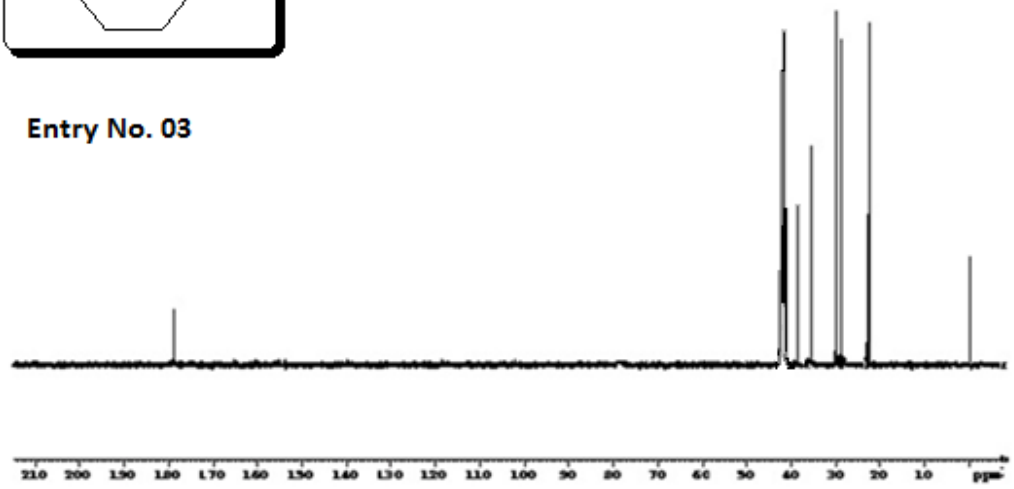
^{1}H-NMR Spectra of ɢ-Caprolactam (Table No. 2, Entry No.3)



^{13}C-NMR Spectra of ε-Caprolactam (Table No. 2, Entry No.3)



Entry No. 03



GCMS Spectra of ɢ-Caprolactam (Table No. 2, Entry No. 3)

KARNATAK UNIVERSITY
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Sample Information

Analyzed by : Admin
Sample Type : Unknown
Level # : 1
Sample Name : SB 2B
Sample ID :
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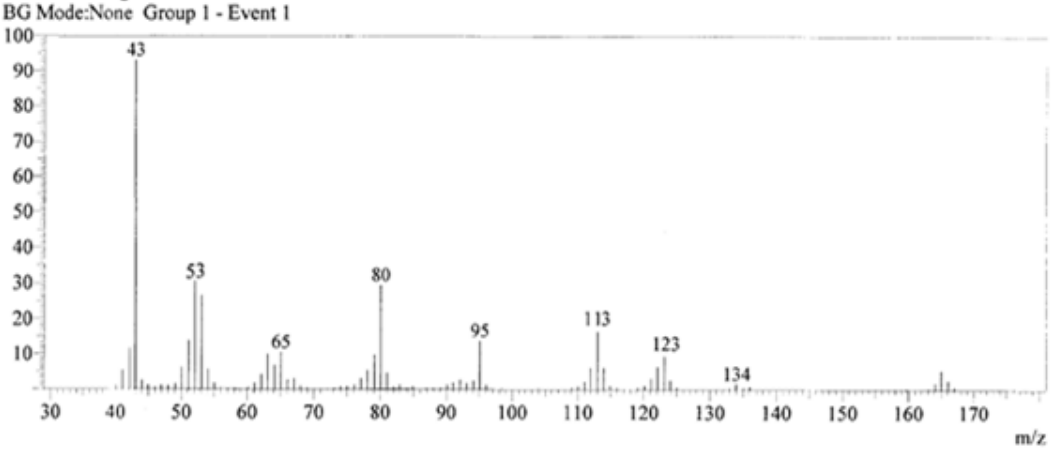
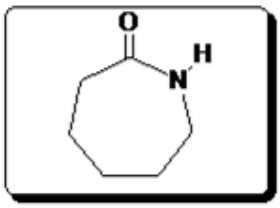
MassPeaks:
RawMode:Single 0.1
BG Mode:None Group 1 - Event 1

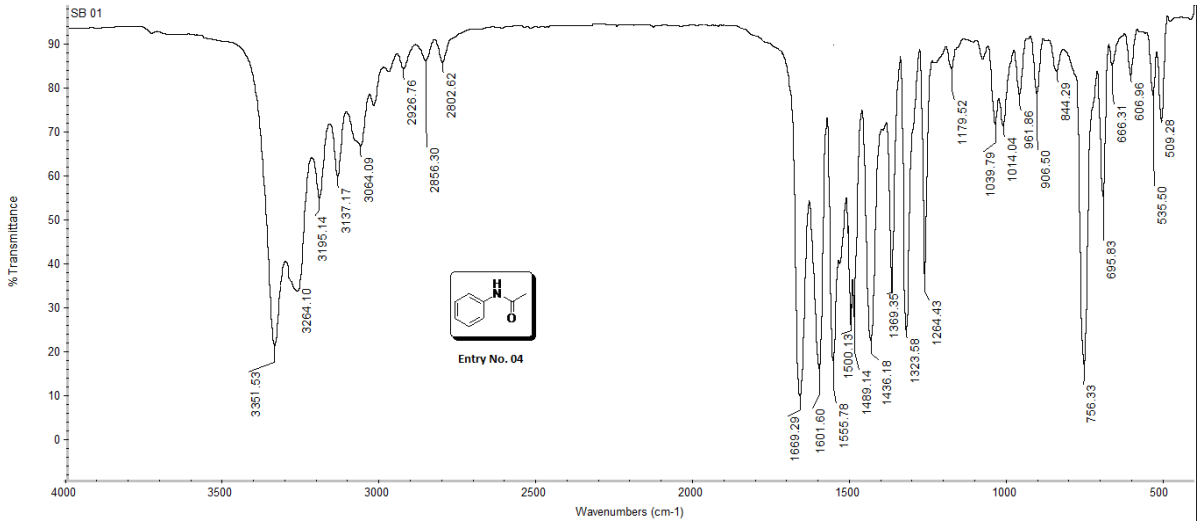




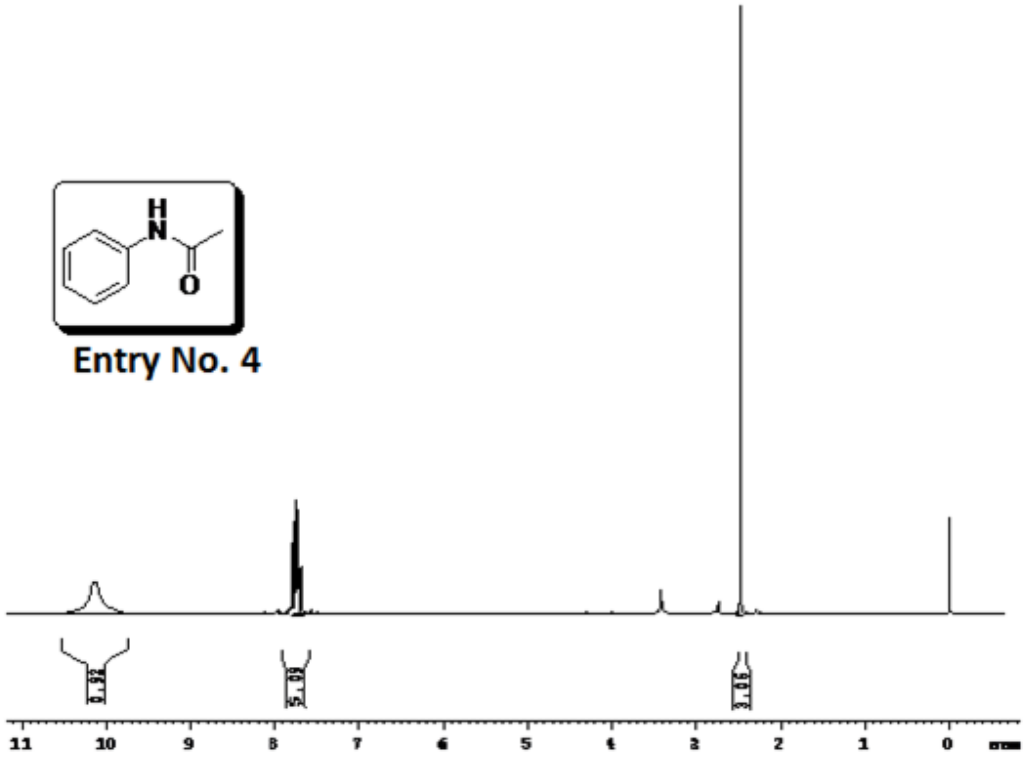
Mol Weight =113

Entry No. 03

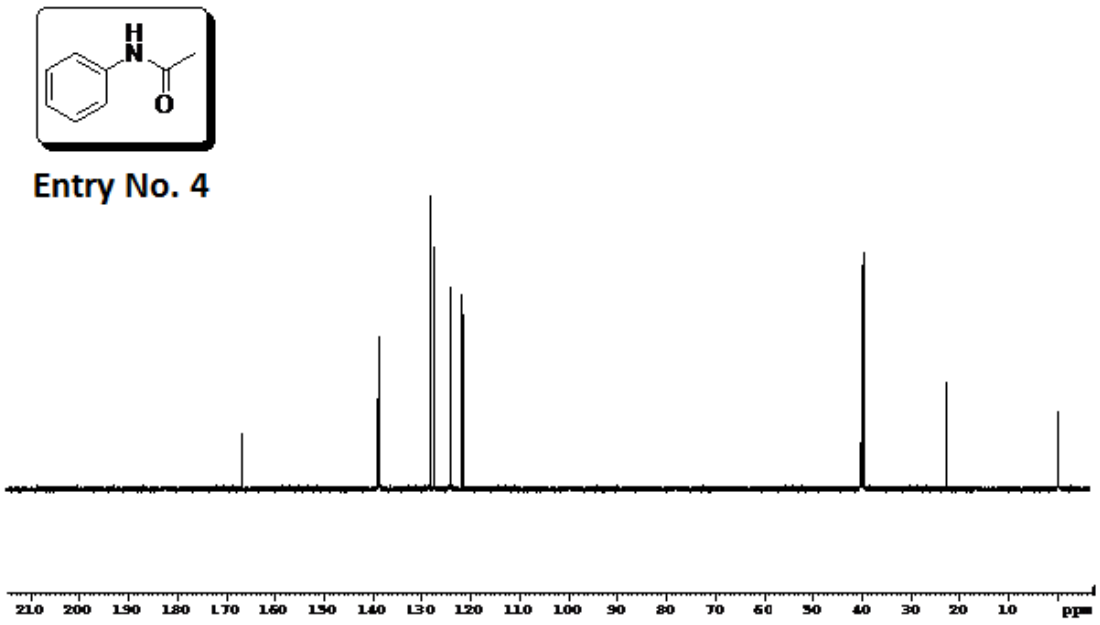
IR Spectra of *N*-Phenylacetamide (Table No. 2, Entry No. 4)



^{1}H-NMR Spectra of *N*-Phenylacetamide (Table No. 2, Entry No. 4)



^{13}C-NMR Spectra of *N*-Phenylacetamide (Table No. 2, Entry No.4)



GCMS Spectra of *N*-Phenylacetamide (Table No. 2, Entry No. 4)

KARNATAK UNIVERSITY
UNIVERSITY SCIENCE INSTRUMENTS CENTRE
DHARWAD

Sample Information

Analyzed by : Admin
Sample Type : Unknown
Level # : 1
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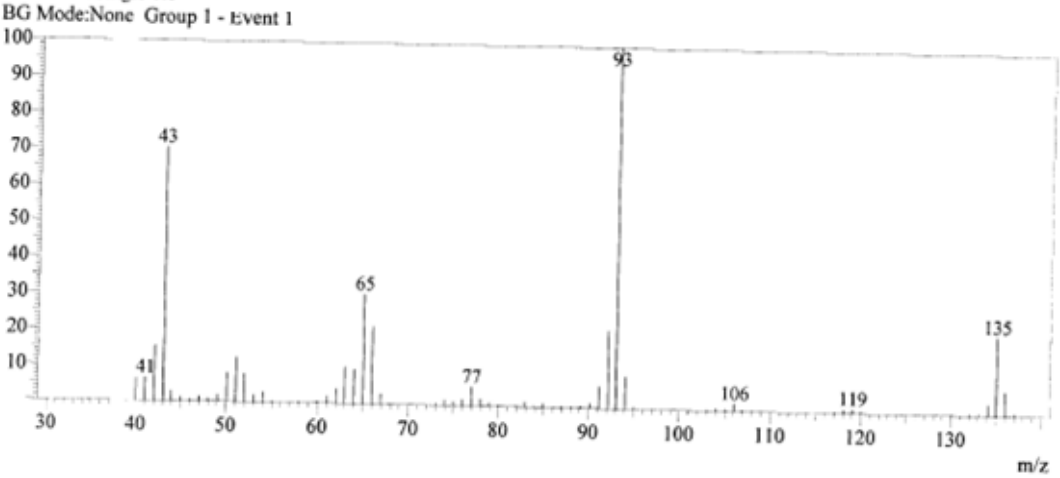
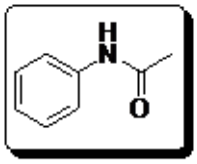
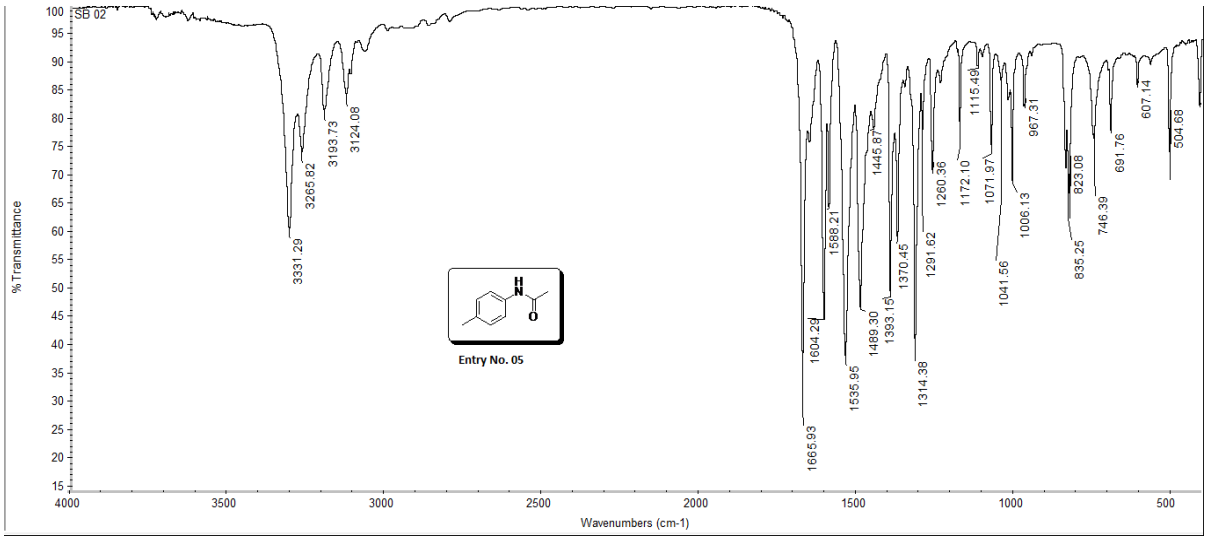




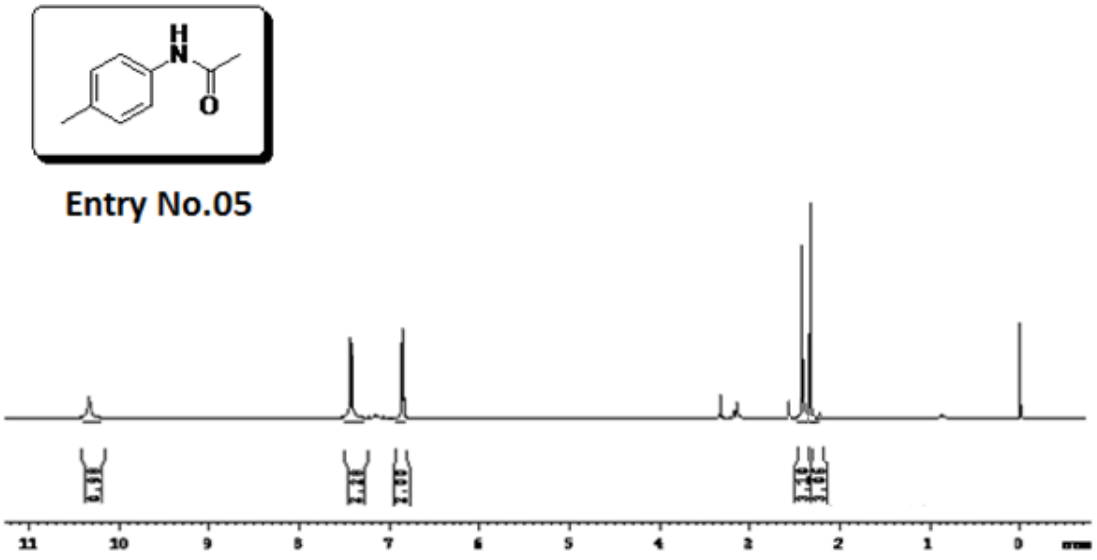
Mol Weight = 135

Entry No = 04

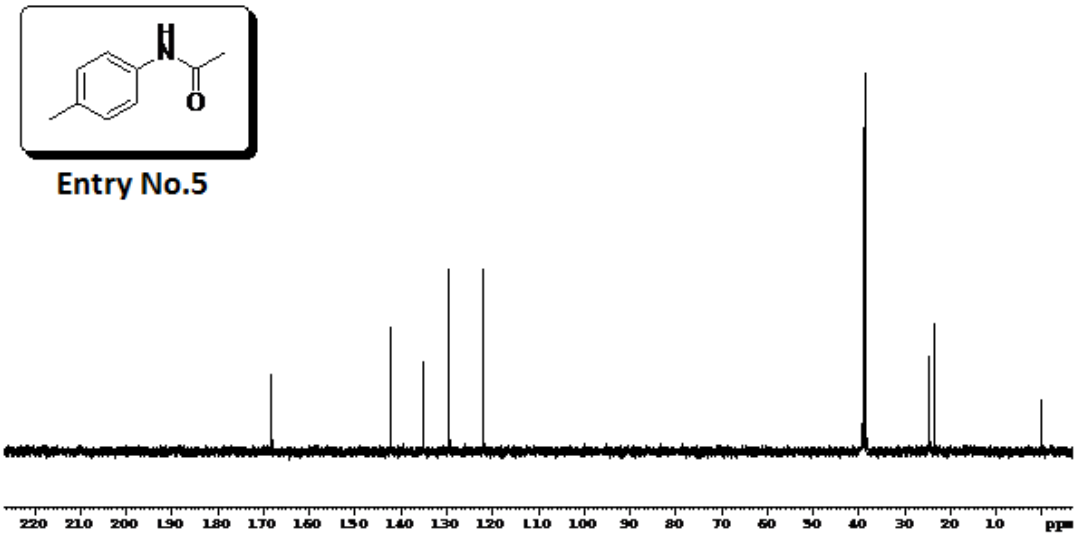
IR Spectra of *N-p*-tolylacetamide (Table No. 2, Entry No. 5)



^{1}H-NMR Spectra of *N-p*-tolylacetamide (Table No. 2, Entry No.5)



^{13}C-NMR Spectra of *N-p*-tolylacetamide (Table No. 2, Entry No.5)



GCMS Spectra of *N-p*-tolylacetamide (Table No. 2, Entry No. 5)

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Sample Information

Analyzed by : Admin
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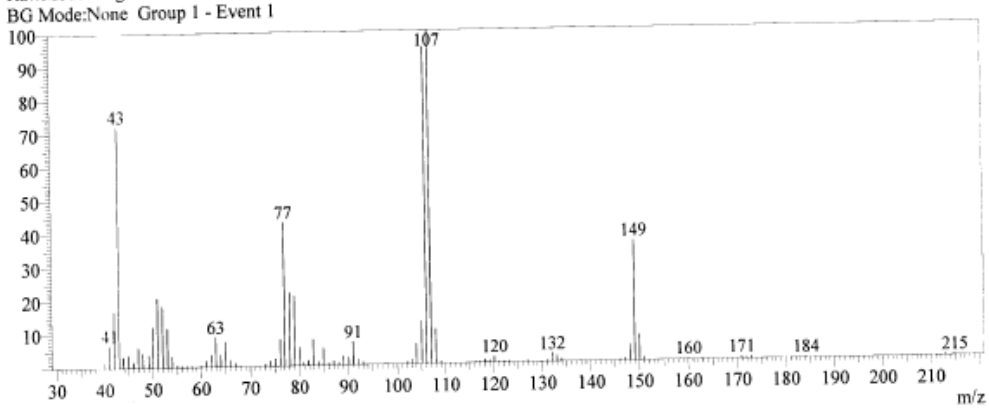
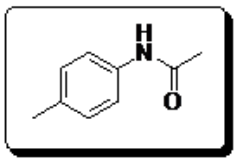
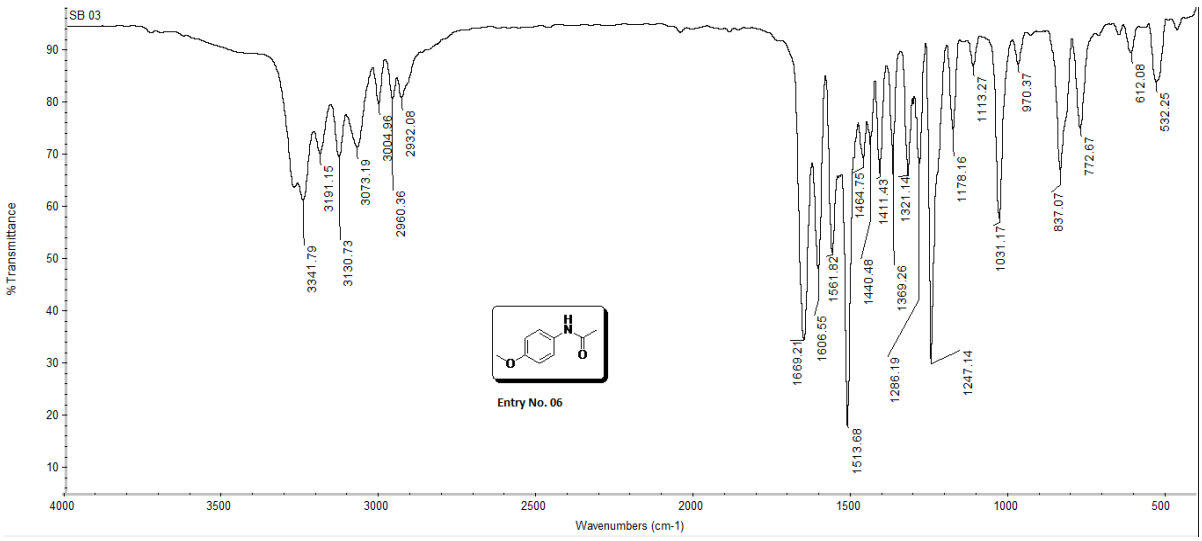




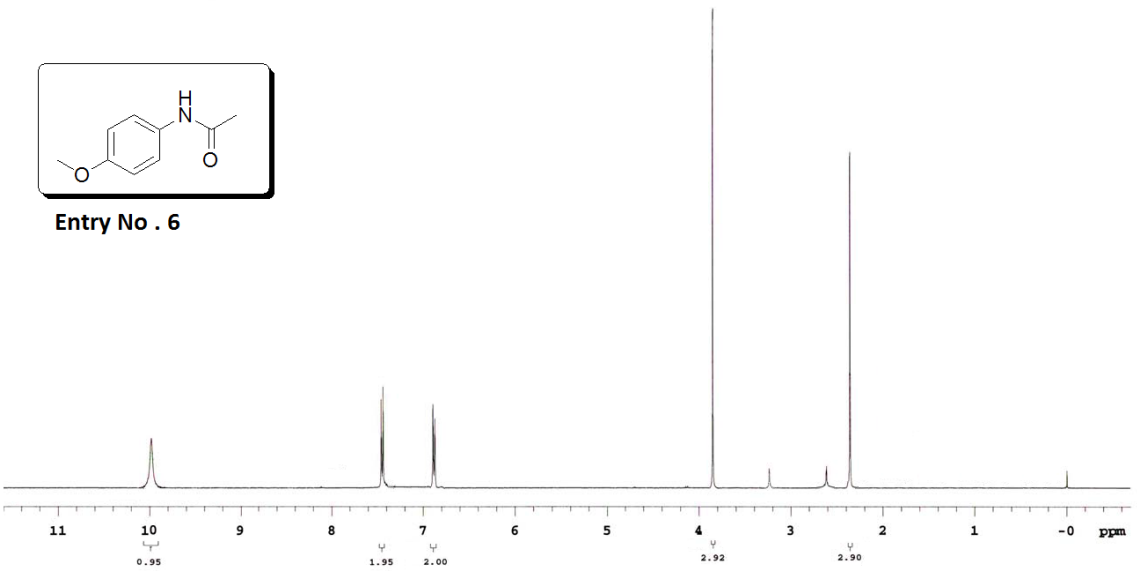
Mol Weight = 149.

Entry No. 05

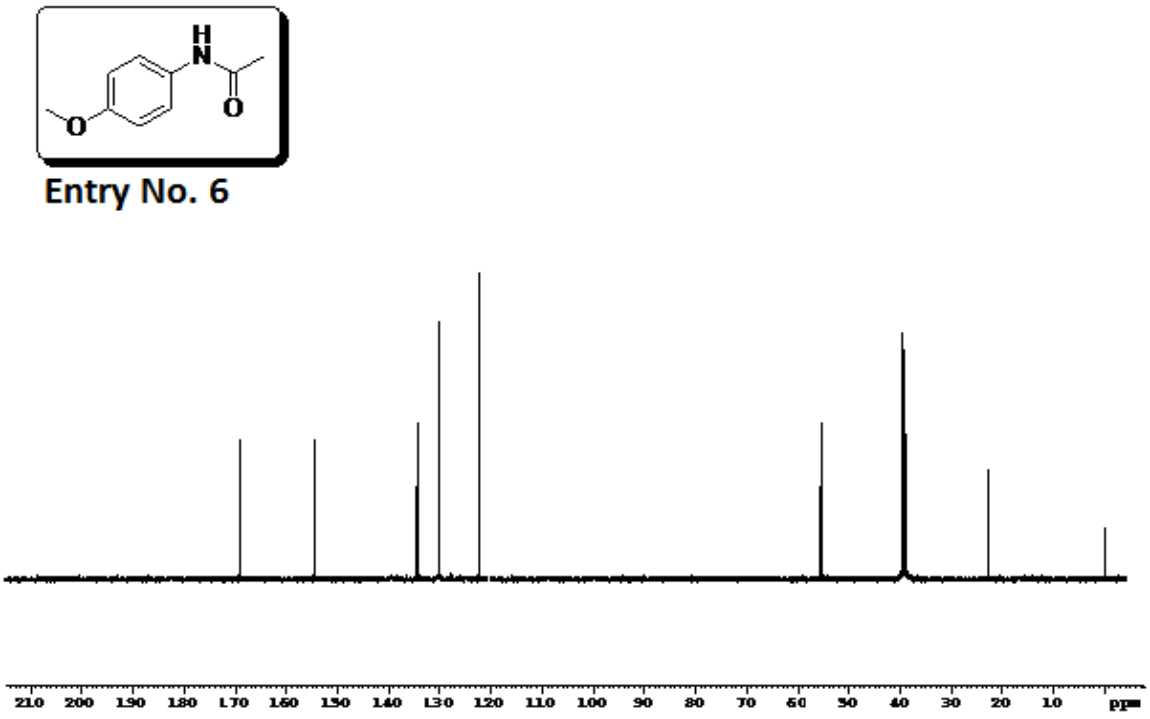
IR Spectra of *N*-(4-Methoxyphenyl)acetamide (Table No. 2, Entry No. 6)



^{1}H-NMR Spectra of *N*-(4-Methoxyphenyl)acetamide (Table No. 2, Entry No. 6)



^{13}C-NMR Spectra of *N*-(4-Methoxyphenyl)acetamide (Table No. 2, Entry No.6)



GCMS Spectra of *N*-(4-Methoxyphenyl)acetamide (Table No. 2, Entry No. 6)

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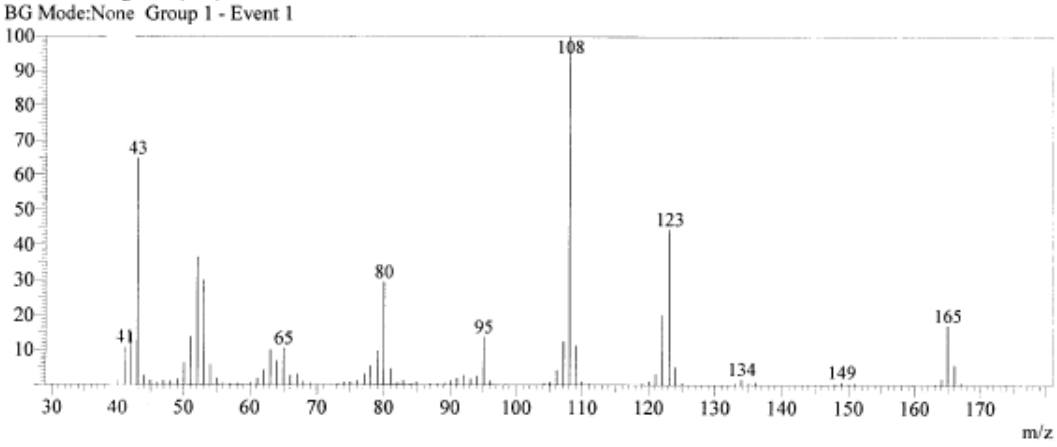
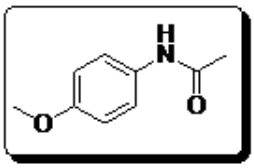
Sample Information

Analyzed by : Admin
Sample Type : Unknown
Level # : 1
Sample Name : SB 03
Sample ID :
IS Amount : [1]=1
Line#:1 R.Time:3.1
MassPeaks:80
RawMode:Single 3.1(372)
BG Mode:None Group 1 - Event 1

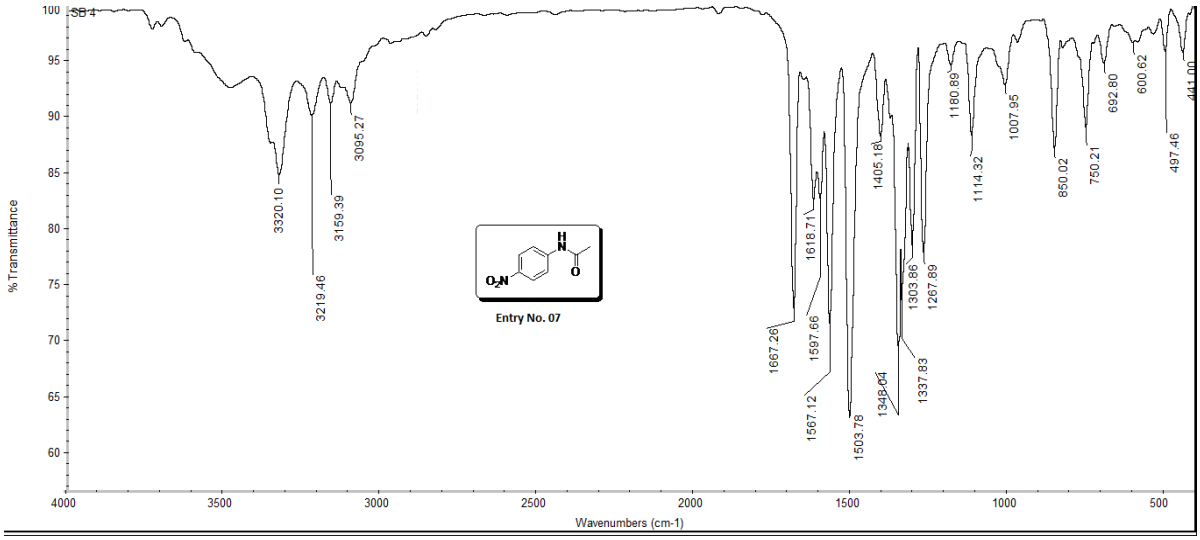




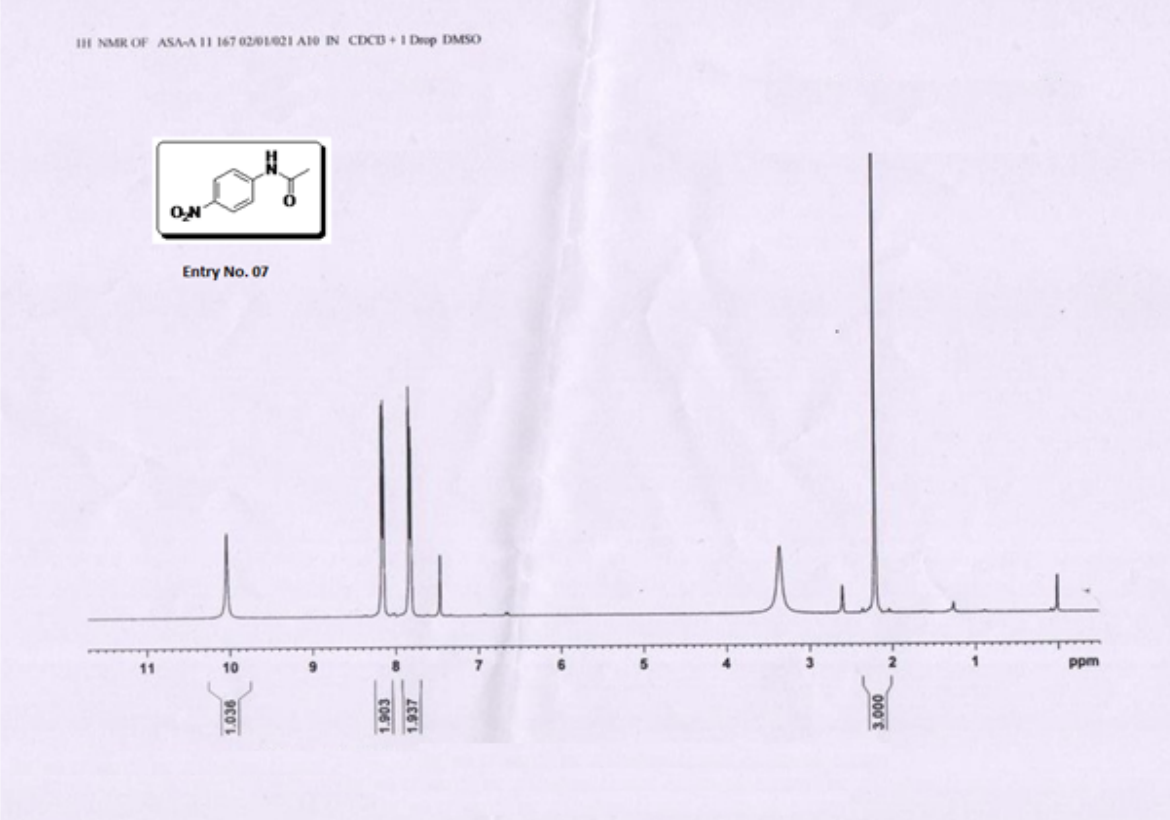
Mol Weight = 165

Entry No. 06

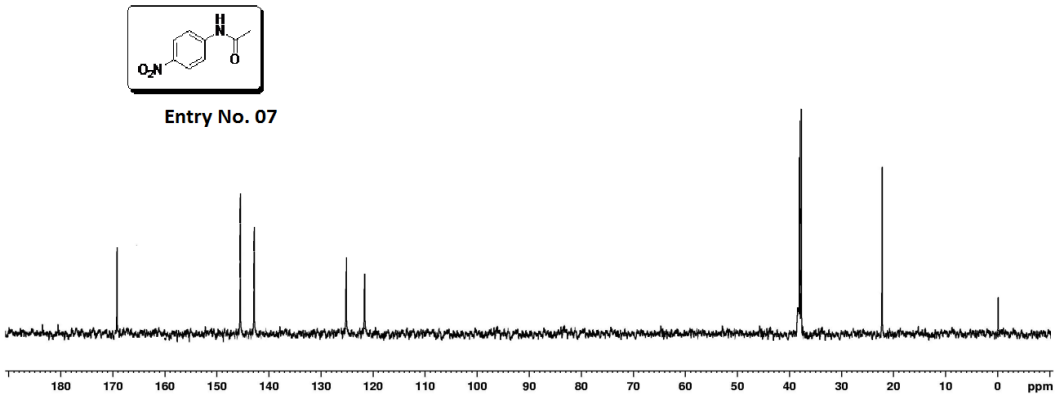
IR Spectra of *N*-(4-Nitrophenyl)acetamide (Table No. 2, Entry No. 7)



^{1}H-NMR Spectra of *N*-(4-Nitrophenyl)acetamide (Table No. 2, Entry No.7)



^{13}C-NMR Spectra of *N*-(4-Nitrophenyl)acetamide (Table No. 2, Entry No.7)



GCMS Spectra of *N*-(4-Nitrophenyl)acetamide (Table No. 2, Entry No.7)

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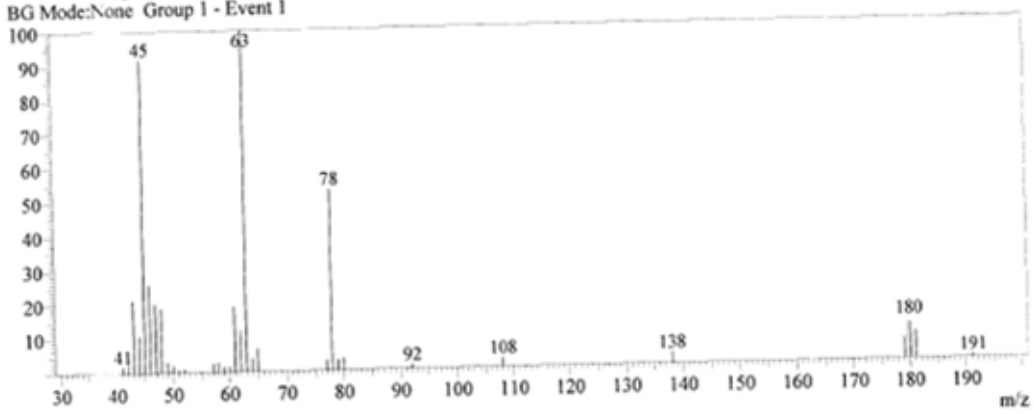
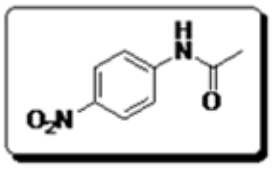
Sample Information

Analyzed by : Admin
Sample Type : Unknown
Level # : 1
Sample Name : SB 4
Sample ID :
IS Amount : [1]=1
Line#:1 R.Time:2.9
MassPeaks:30
RawMode:Single 2.9(347)
BG Mode:None Group 1 - Event 1

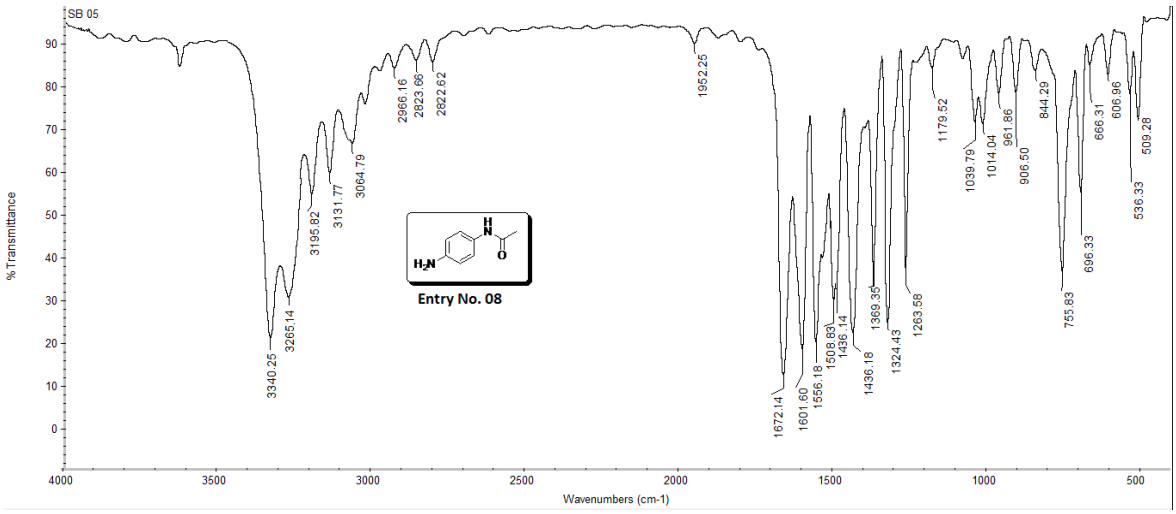




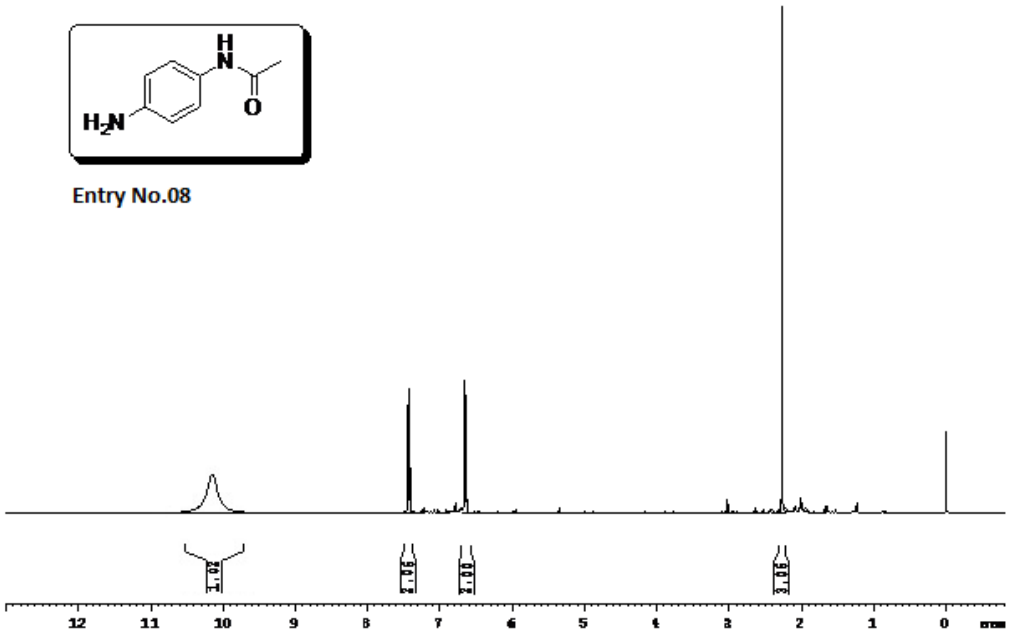
mol Weight = 180

Entry No. 07

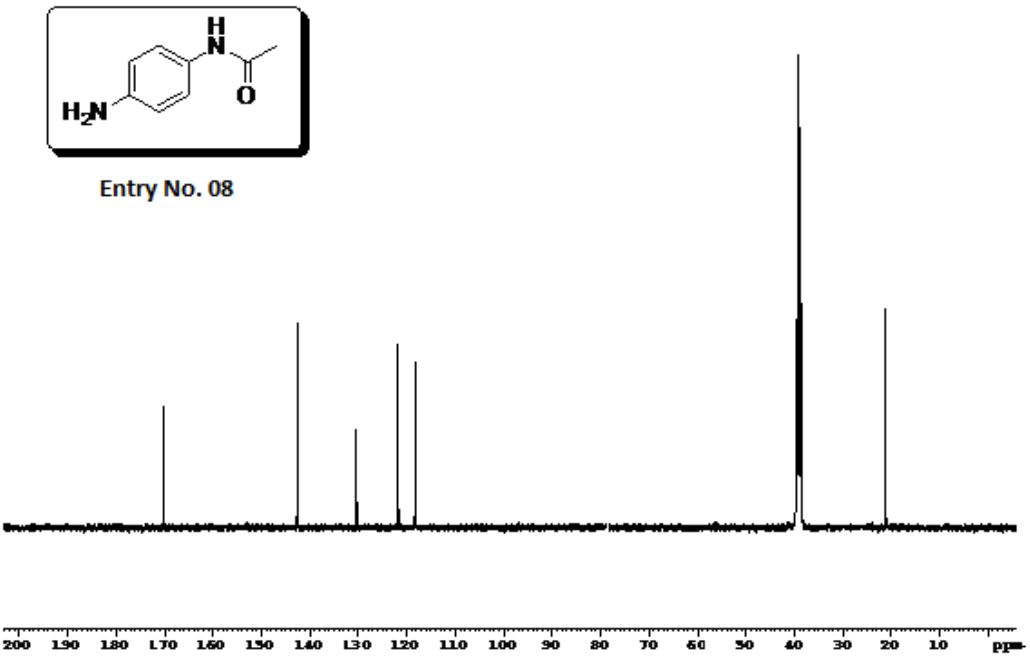
IR Spectra *N*-(4-Aminophenyl)acetamide (Table No. 2, Entry No.8)



^{1}H-NMR Spectra of *N*-(4-Aminophenyl)acetamide (Table No. 2, Entry No.8)



^{13}C-NMR Spectra of *N*-(4-Aminophenyl)acetamide (Table No. 2, Entry No.8)



GCMS Spectra of *N*-(4-Aminophenyl)acetamide (Table No. 2, Entry No.8)

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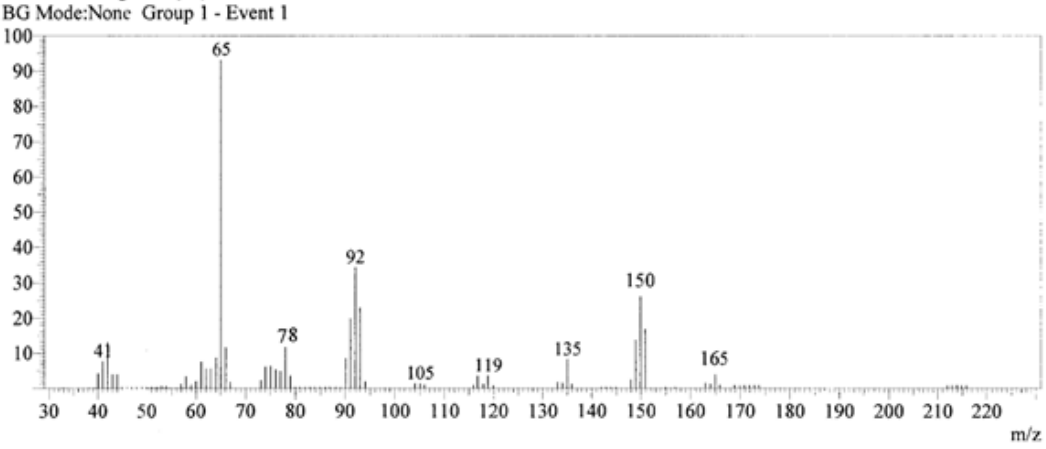
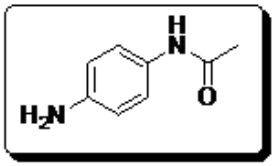
Sample Information

Analyzed by : Admin
Sample Type : Unknown
Level # : 1
Sample Name : SB 05
Sample ID :
IS Amount : [1]=1
Line#:1 R.Time:0.1
MassPeaks:77
RawMode:Single 0.1(10)
BG Mode:None Group 1 - Event 1

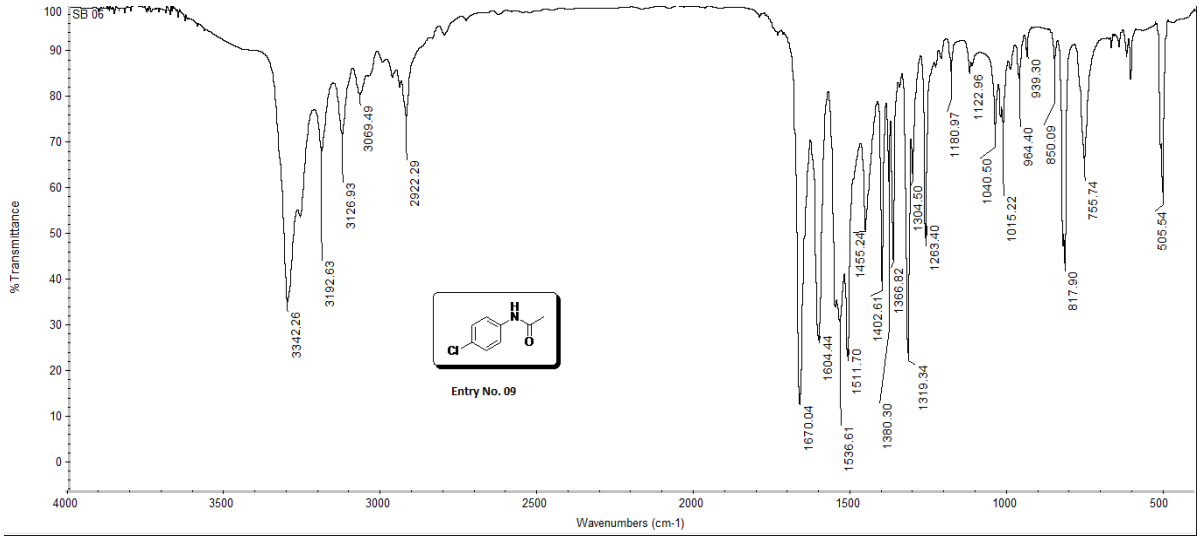




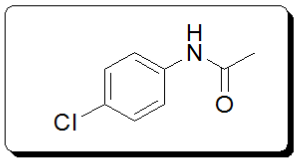
Mol Weight= 150

Entry No. 08

IR Spectra of *N*-(4-Chlorophenyl)acetamide (Table No. 2, Entry No. 9)

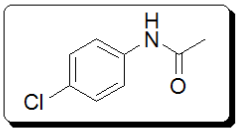


^{1}H-NMR Spectra of *N*-(4-Chlorophenyl)acetamide (Table No. 2, Entry No.9)

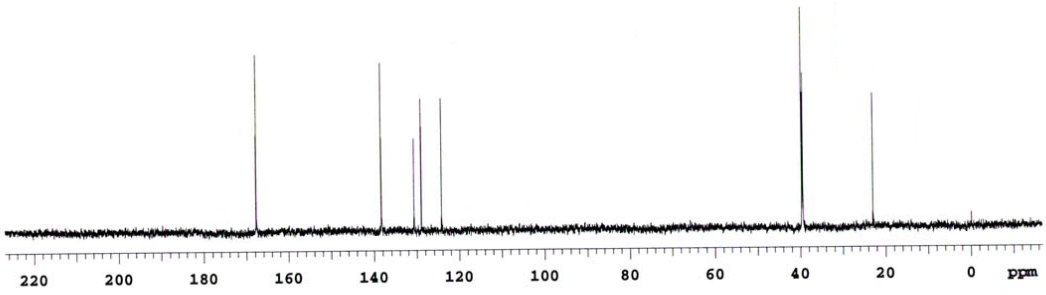


Entry No. 09

^{13}C-NMR Spectra *N*-(4-Chlorophenyl)acetamide (Table No. 2, Entry No.9)



Entry No. 9



GCMS Spectra of *N*-(4-Chlorophenyl)acetamide (Table No. 2, Entry No.9)

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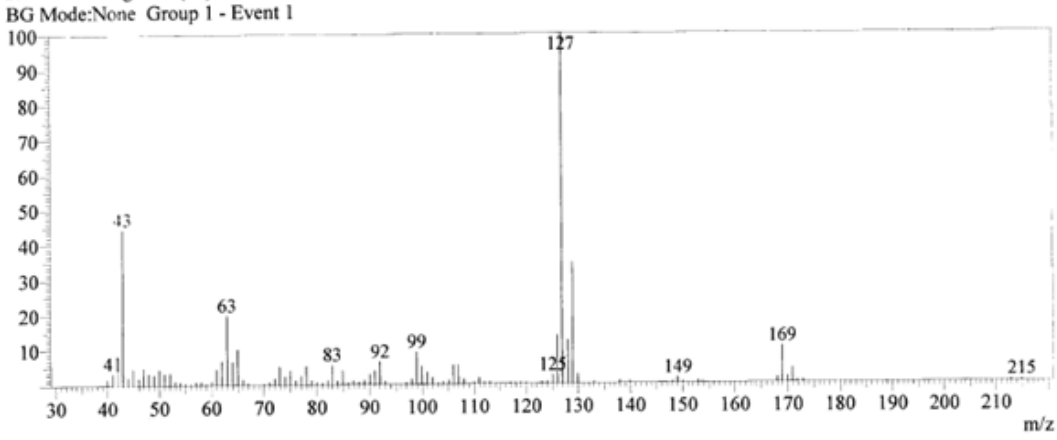
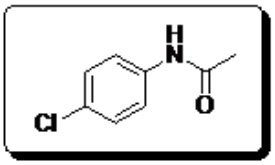
Sample Information

Analyzed by : Admin
Sample Type : Unknown
Level # : 1
Sample Name : SB 06
Sample ID :
IS Amount : [1]=1
Line#:1 R.Time:0.1
MassPeaks:96
RawMode:Single 0.1(11)
BG Mode:None Group 1 - Event 1

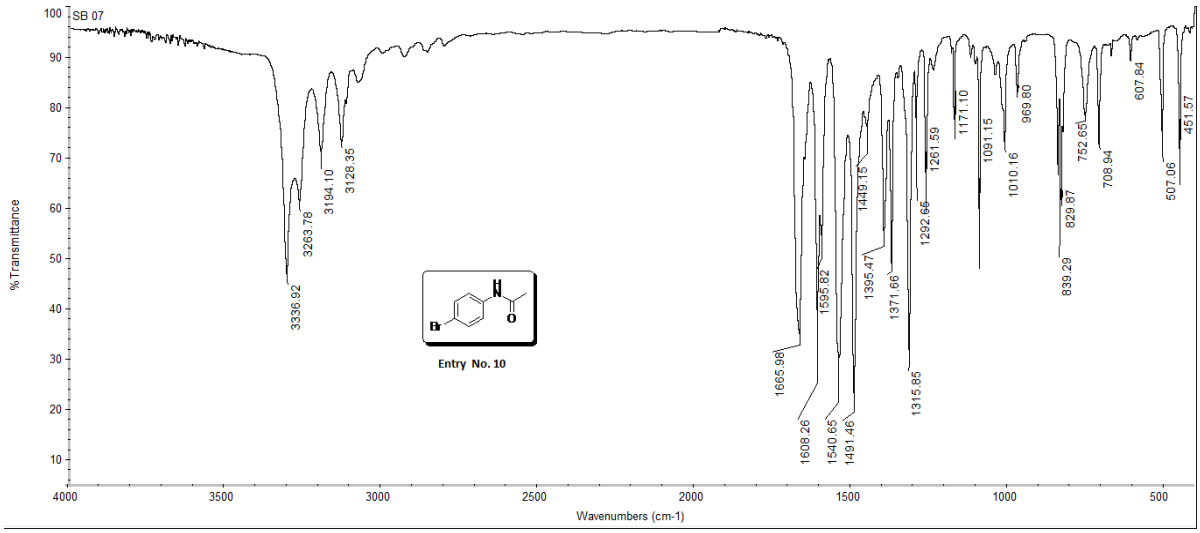




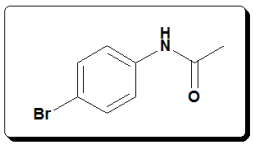
Mol. Weight = 169

Entry No 09

IR Spectra of *N*-(4-Bromophenyl)acetamide (Table No. 2, Entry No.10)

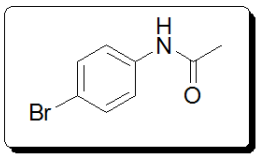


^{1}H-NMR Spectra of *N*-(4-Bromophenyl)acetamide (Table No. 2, Entry No.10)

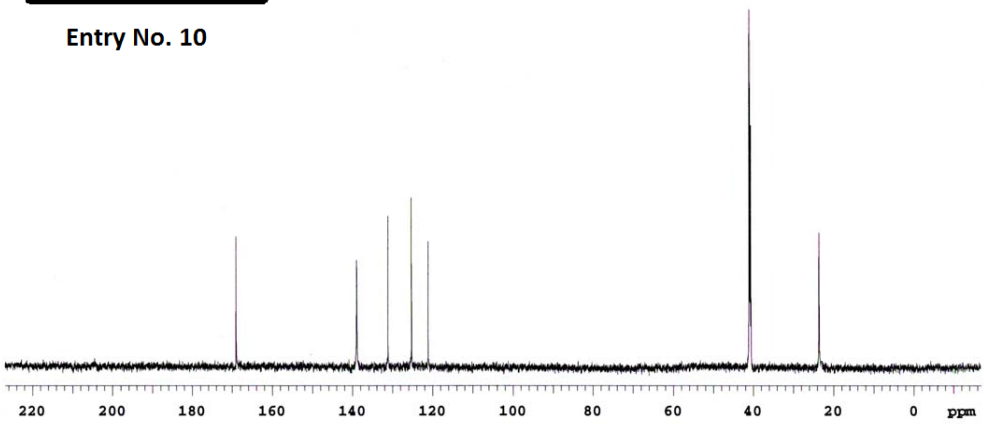


Entry No. 10

^{13}C-NMR Spectra *N*-(4-Bromophenyl)acetamide (Table No. 2, Entry No.10)



Entry No. 10



GCMS Spectra of *N*-(4-Bromophenyl)acetamide (Table No. 2, Entry No.10)

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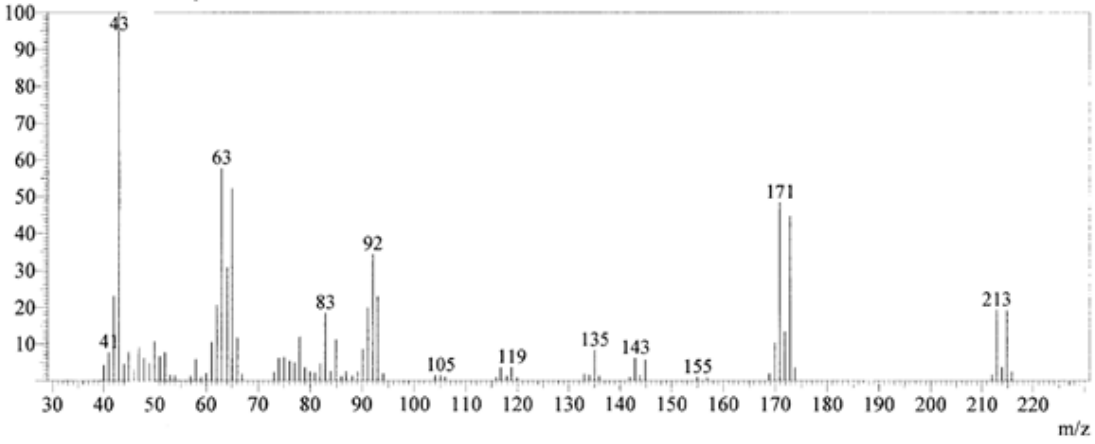
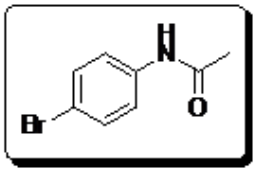
DHARWAD

Sample Information

Analyzed by : Admin
Sample Type : Unknown
Level # : 1
Sample Name : SB 07
Sample ID :
IS Amount : [1]=1
Line#:1 R.Time:0.1
MassPeaks:77
RawMode:Single 0.1(10)
BG Mode:None Group 1 - Event 1

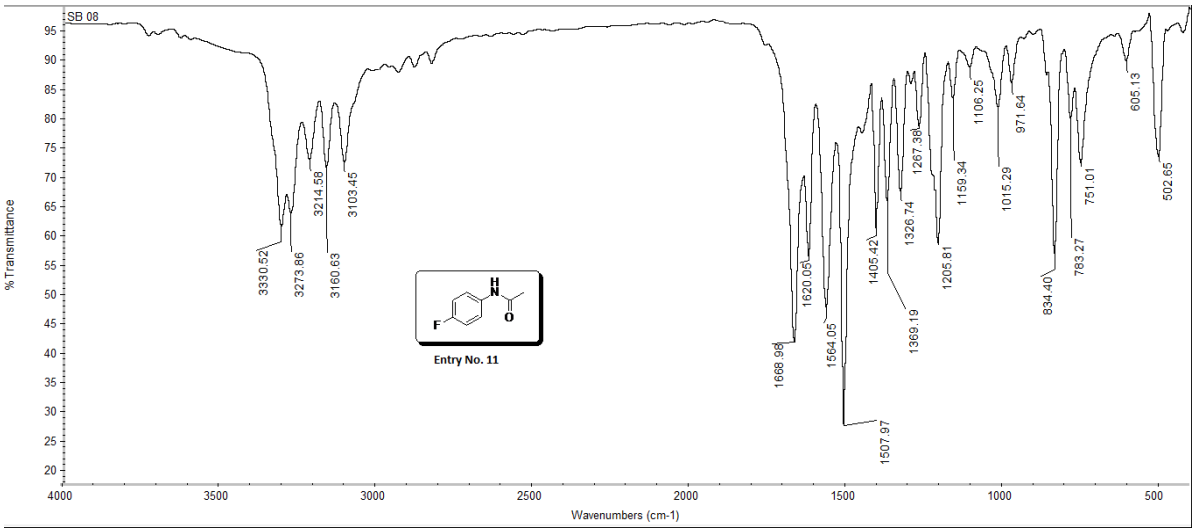




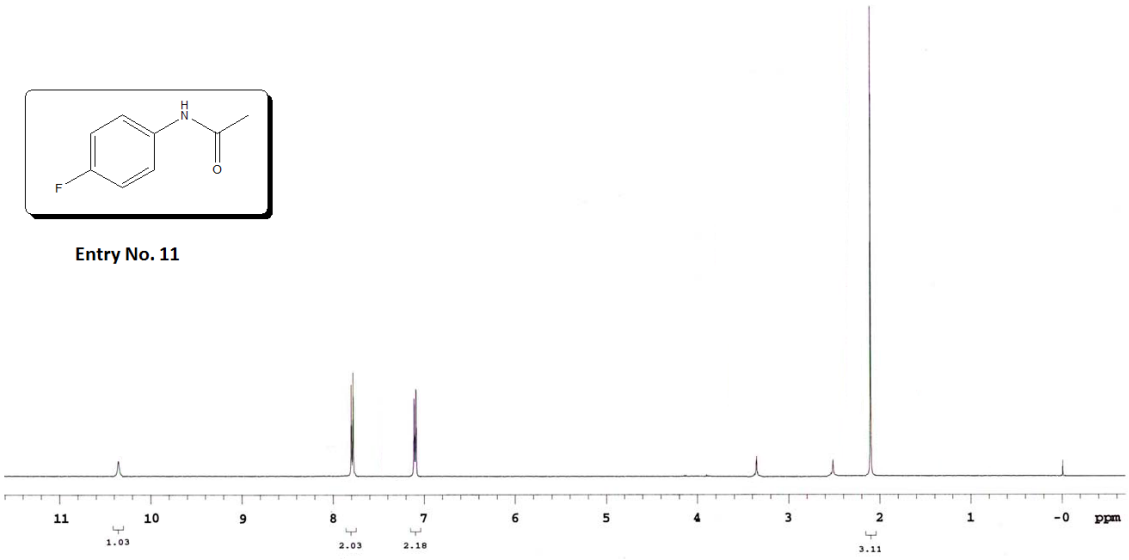
Mol Weight = 214

Entry No. 10

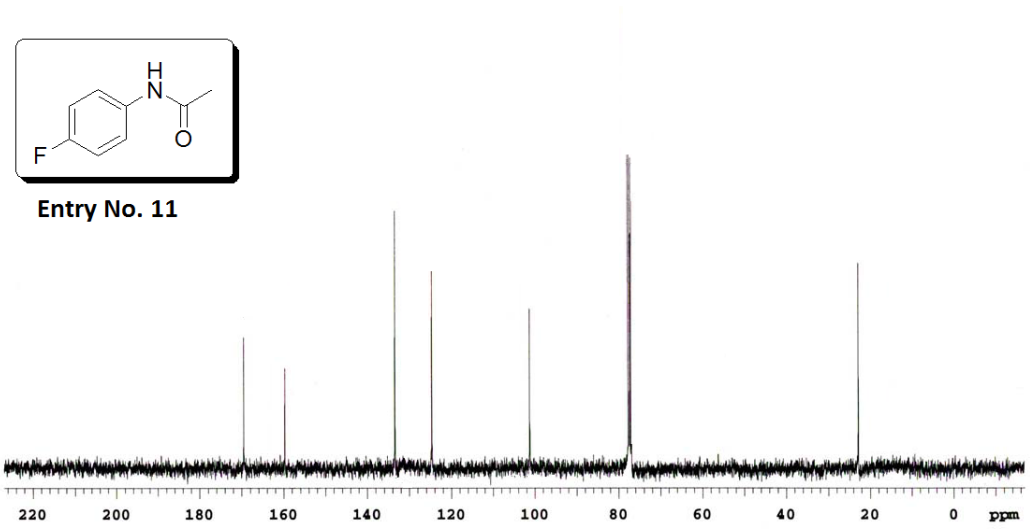
IR Spectra of *N*-(4-Fluorophenyl)acetamide (Table No. 2, Entry No.11)



^{1}H-NMR Spectra of *N*-(4-Fluorophenyl)acetamide (Table No. 2, Entry No11)



^{13}C-NMR Spectra *N*-(4-Fluorophenyl)acetamide (Table No. 2, Entry No.11)



GCMS Spectra of *N*-(4-Fluorophenyl)acetamide (Table No. 2, Entry No.11)

KARNATAK UNIVERSITY
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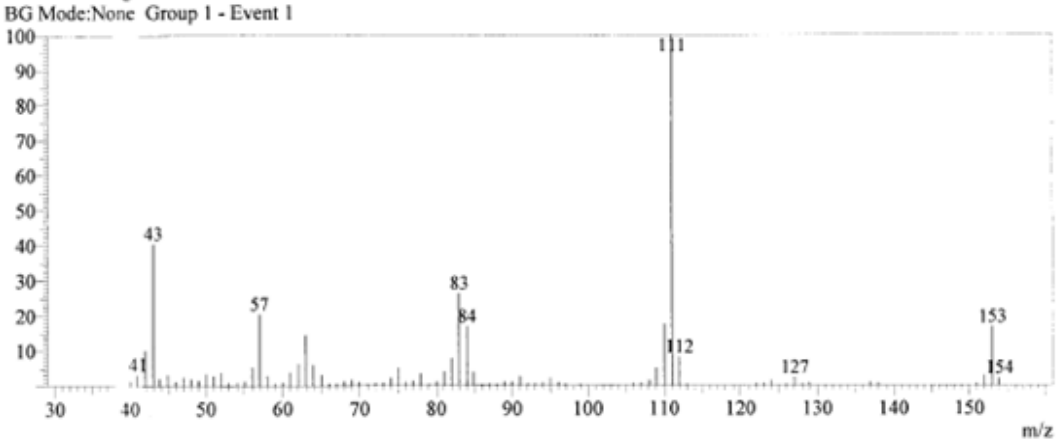
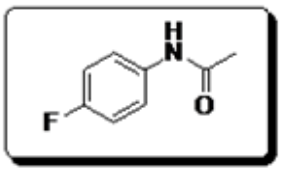
DHARWAD

Sample Information

Analyzed by : Admin
Sample Type : Unknown
Level # : 1
Sample Name : SB 08
Sample ID :
IS Amount : 111=1
Line#:1 R.Time:0.1
MassPeaks:80
RawMode:Single 0.1
BG Mode:None Group 1 - Event 1

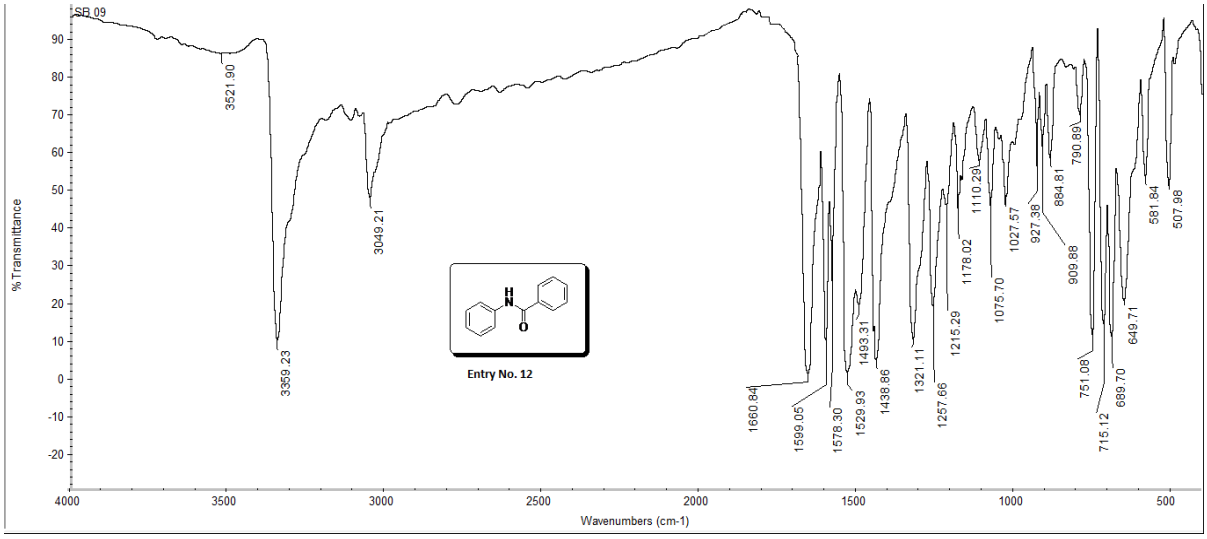




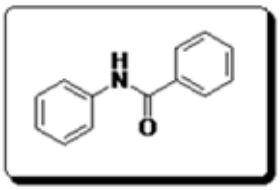
Mol Weight = 153

Entry No. 11

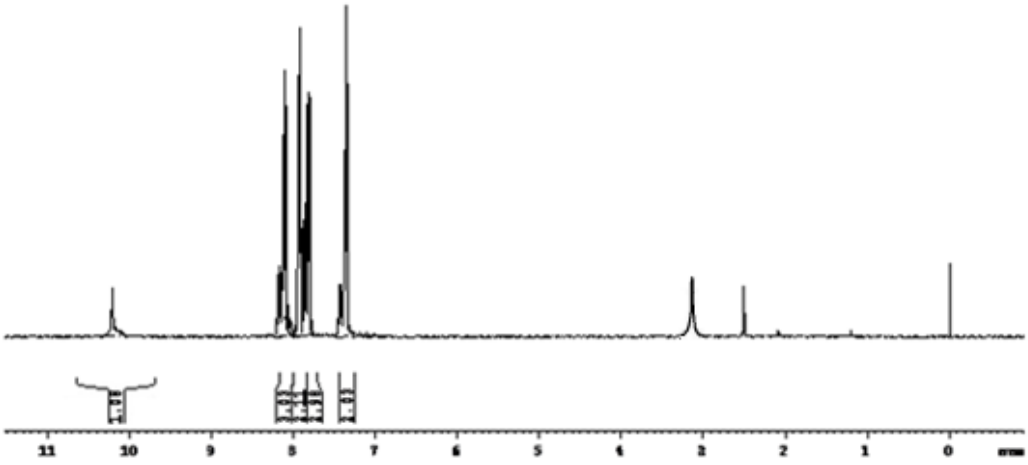
IR Spectra of *N*-phenylbenzamide (Table No. 2, Entry No.12)



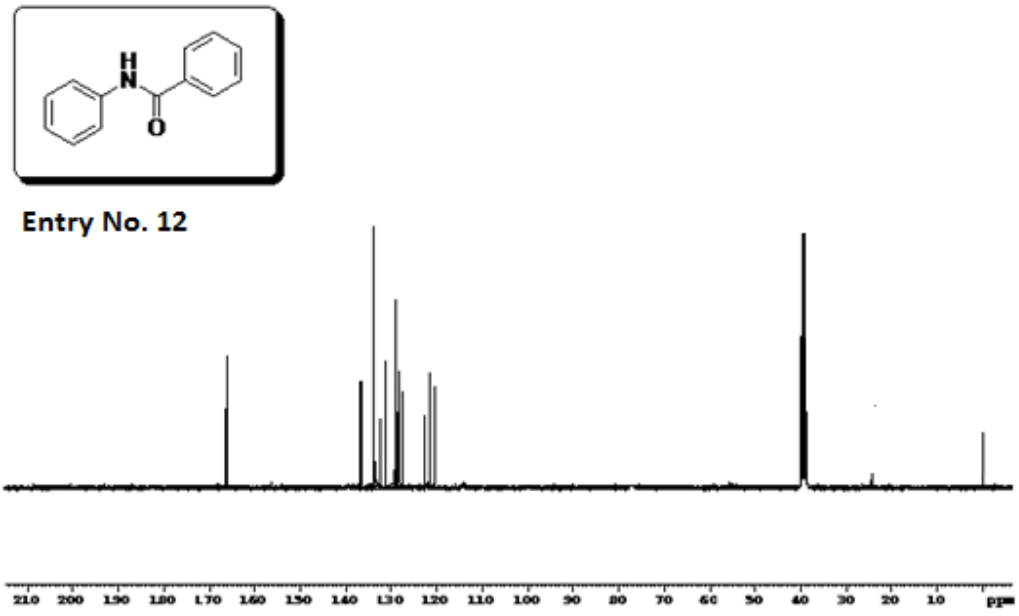
^{1}H-NMR Spectra of *N*-phenylbenzamide (Table No. 2, Entry No12)



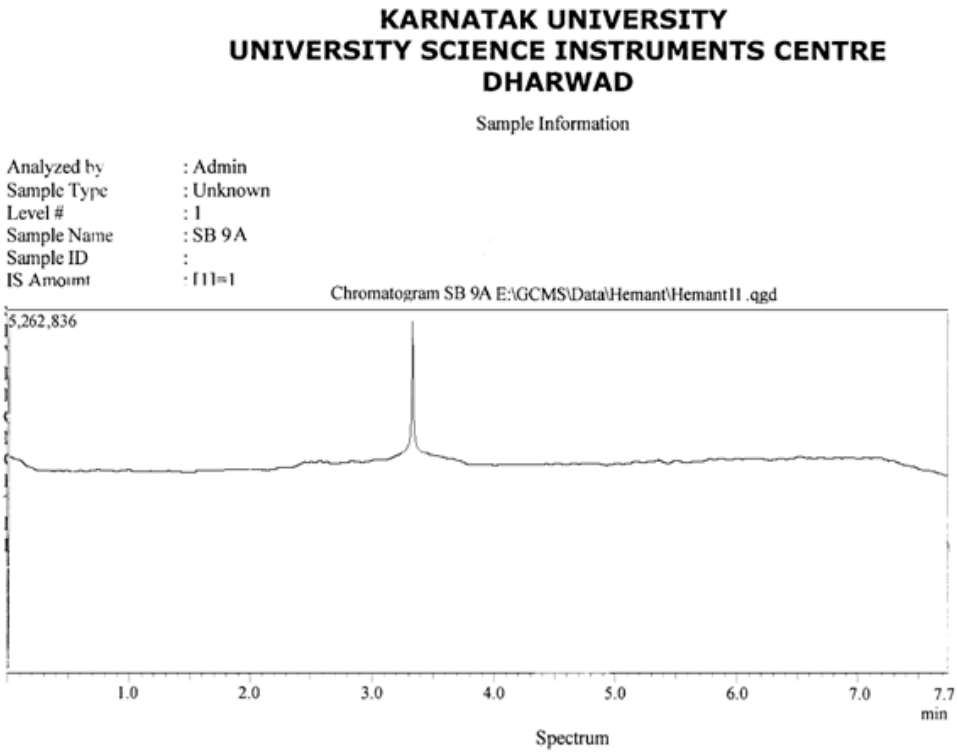
Entry No. 12

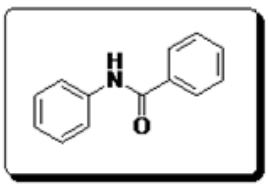


^{13}C-NMR Spectra *N*-phenylbenzamide (Table No. 2, Entry No.12)



GCMS Spectra of *N*-phenylbenzamide (Table No. 2, Entry No.12)





Mol Weight=197

Entry No. 12

GCMS Spectra of *N*-phenylbenzamide (Table No. 2, Entry No.12)

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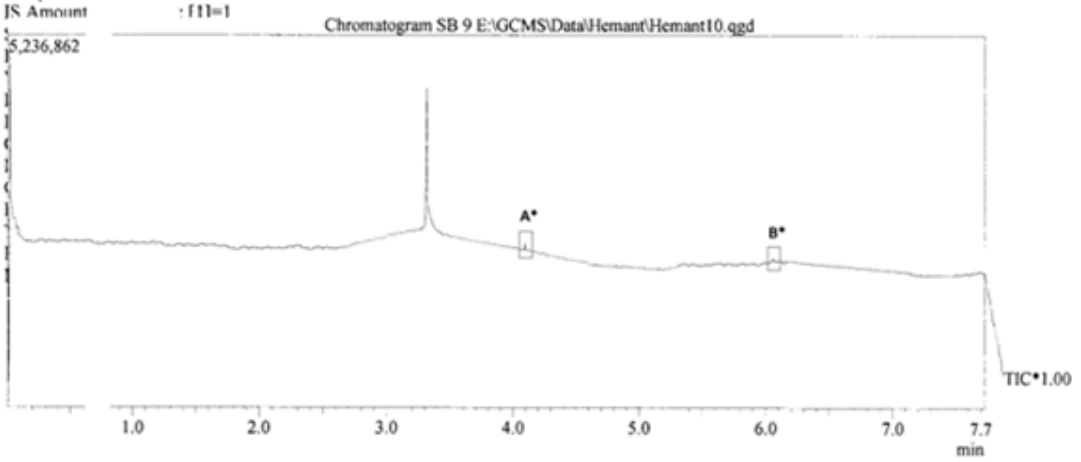
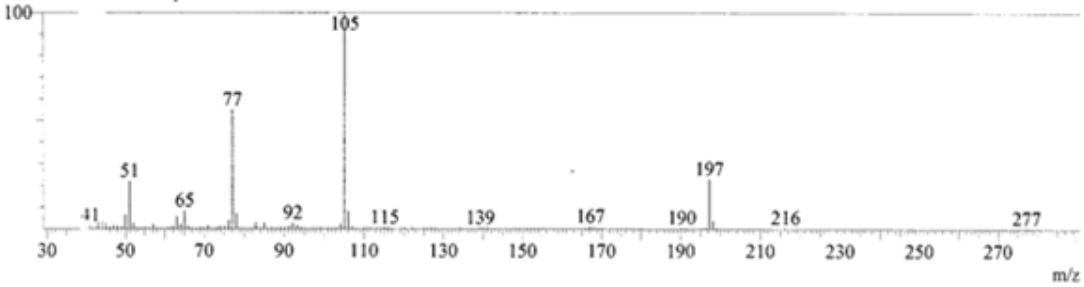
Sample Information

Analyzed by : Admin
Sample Type : Unknown
Level # : 1
Sample Name : SB 9
Sample ID :
IS Amount : f11=1

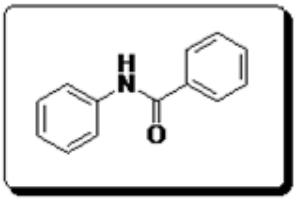


Spectrum

Line#:1 R.Time:3.3(Scan#:402)
MassPeaks:111
RawMode Single 3.3(402) BasePeak:105(1713646)
BG Mode:None Group 1 - Event 1



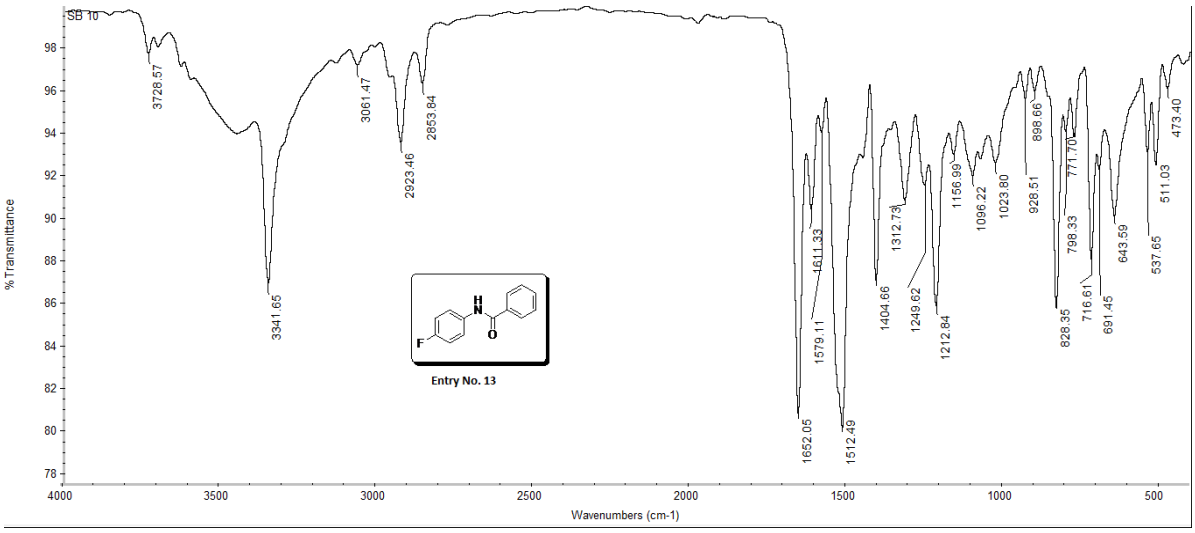
A* = Keto Impurity
B* = Dimer Impurity



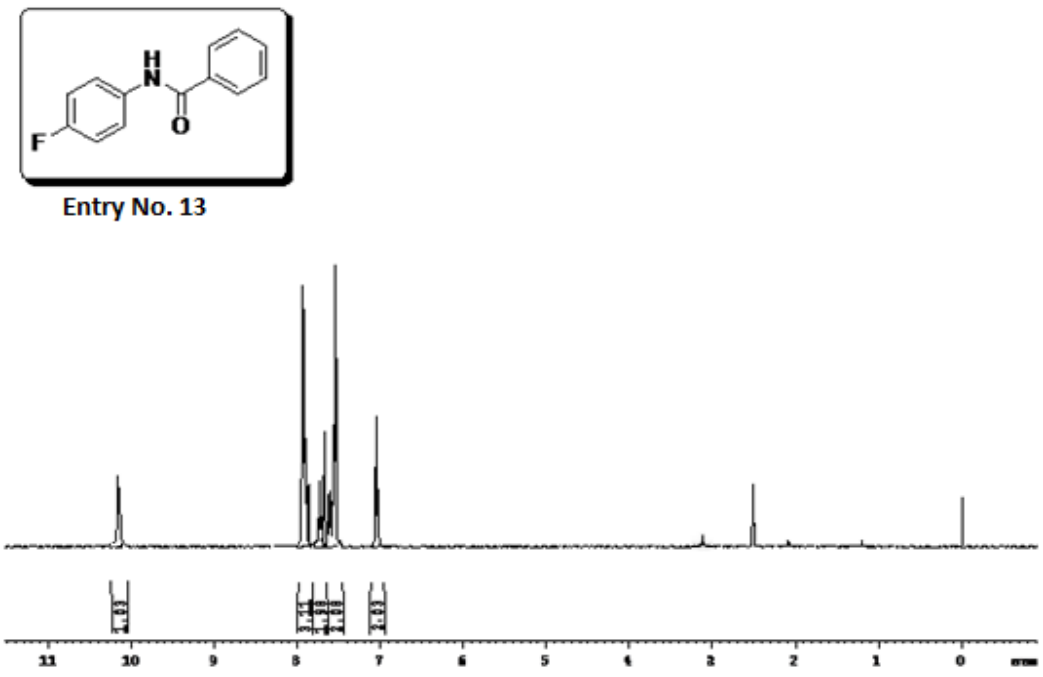
Mol Weight=197

Entry No. 12

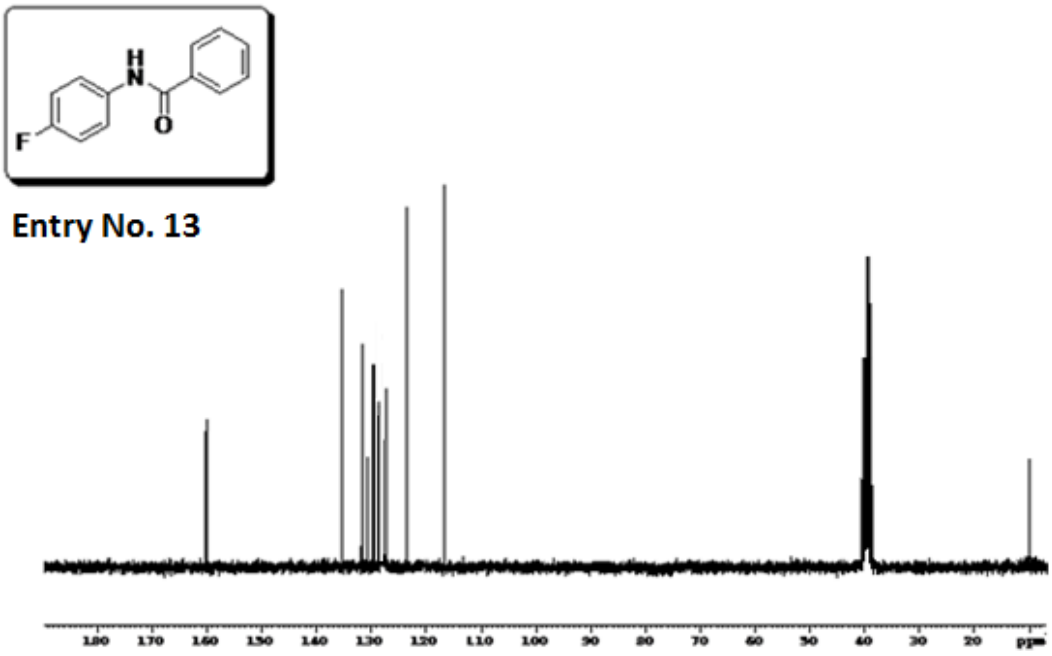
IR Spectra of *N*-(4-Fluorophenyl)benzamide (Table No. 2, Entry No.13)



^{1}H-NMR Spectra of *N*-(4-Fluorophenyl)benzamide (Table No. 2, Entry No13)



^{13}C-NMR Spectra *N*-(4-Fluorophenyl)benzamide (Table No. 2, Entry No.13)



GCMS Spectra of *N*-(4-Fluorophenyl)benzamide (Table No. 2, Entry No.13)

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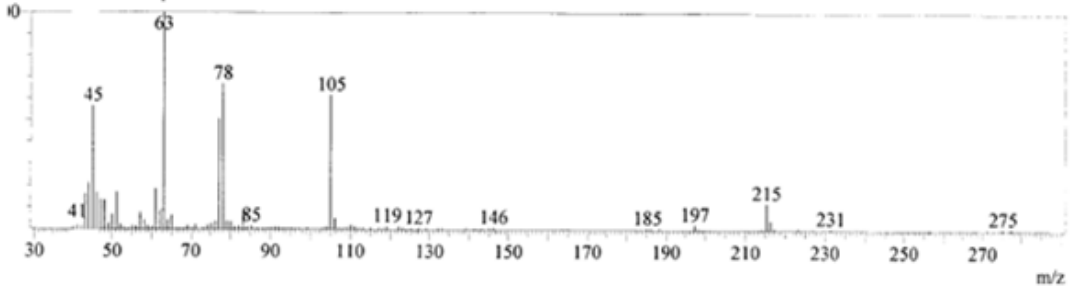
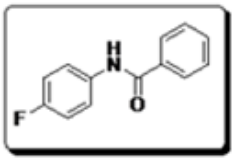
UNIVERSITY SCIENCE INSTRUMENTS CENTRE
DHARWAD

Sample Information

Analyzed by : Admin
Sample Type : Unknown
Level # : 1
Sample Name : SB 10
Sample ID :
IS Amount : 111=1
Line#:1 R Time:4.0
Processed Peaks:97
Raw Mode:Single 4.0(487)
MS Mode:None Group 1 - Event 1

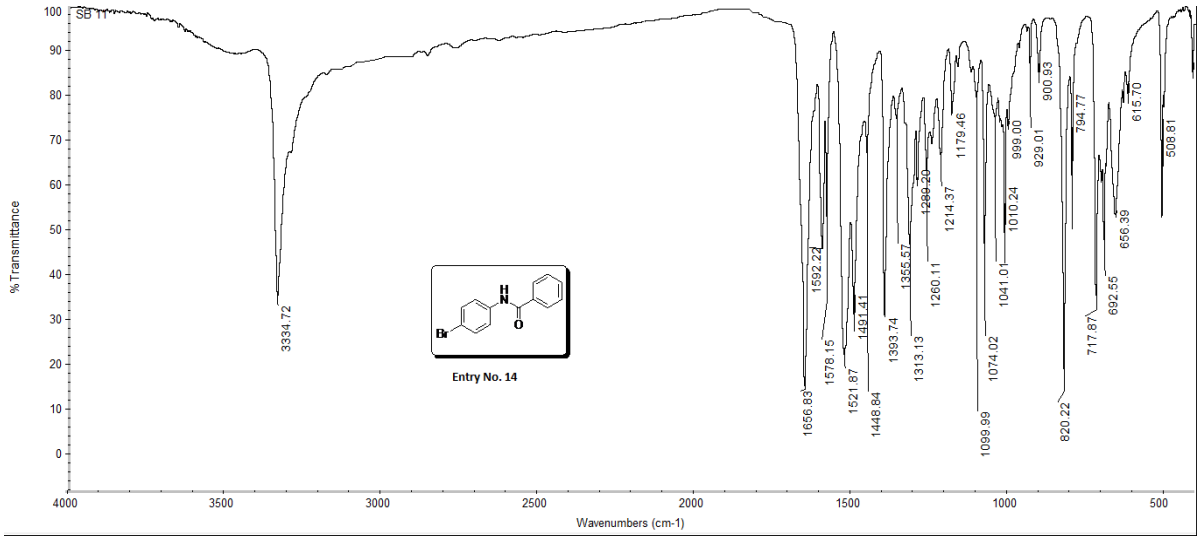




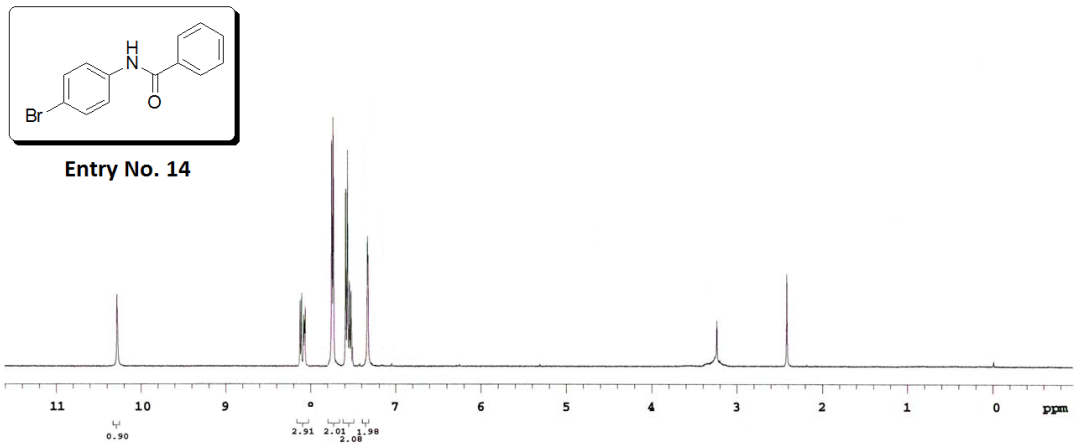
Entry No. =13

Mol Weight = 215

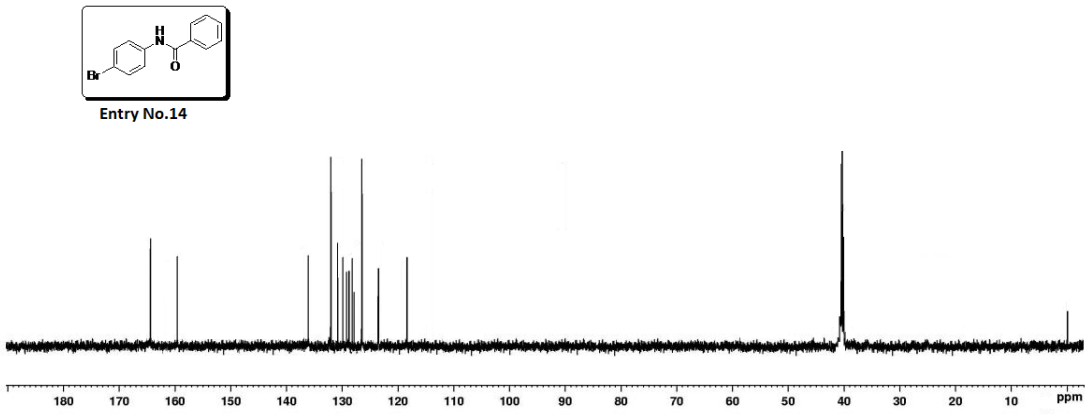
IR Spectra of *N*-(4-Bromophenyl)benzamide (Table No. 2, Entry No.14)



^{1}H-NMR Spectra of *N*-(4-Bromophenyl)benzamide (Table No. 2, Entry No14)



^{13}C-NMR Spectra *N*-(4-Bromophenyl)benzamide (Table No. 2, Entry No.14)



GCMS Spectra of *N*-(4-Bromophenyl)benzamide (Table No. 2, Entry No.14)

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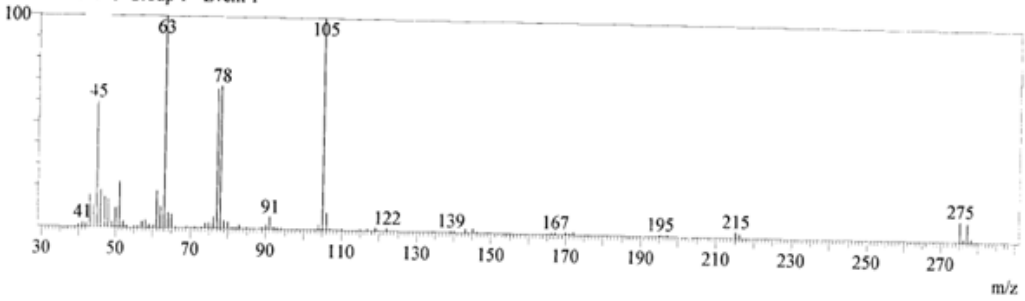
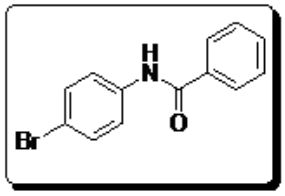
Sample Information

Analyzed by : Admin
Sample Type : Unknown
Level # : 1
Sample Name : SB 11
Sample ID :

Line#:1 R.Time:3.7
MassPeaks:74
RawMode:Single 3.7
BG Mode:None Group 1 - Event 1

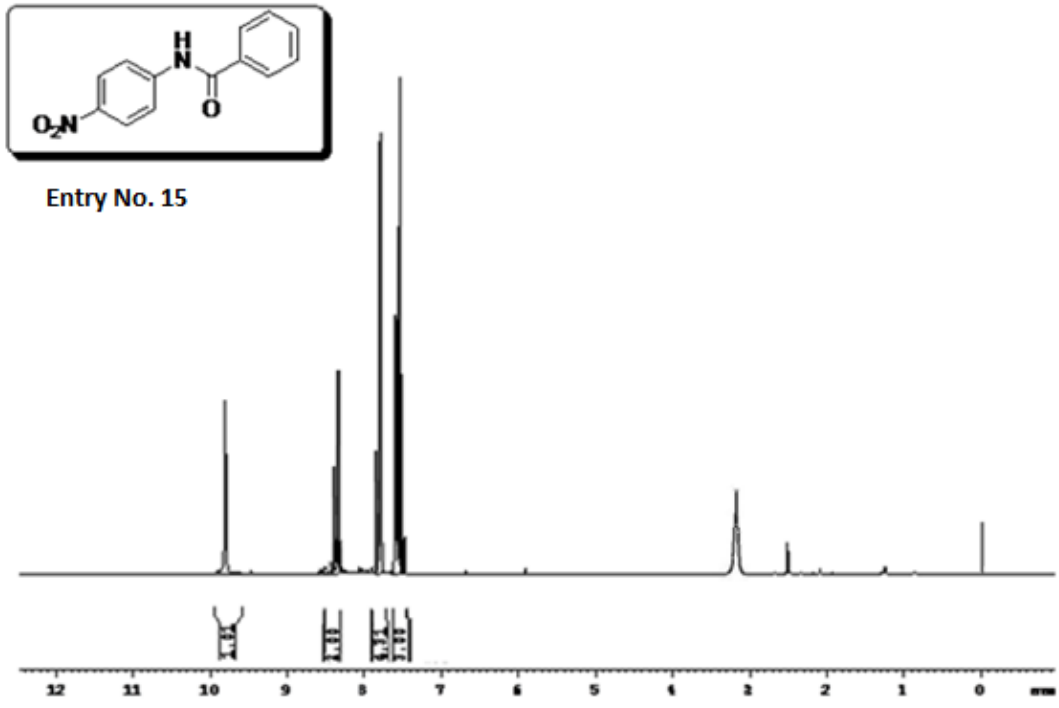




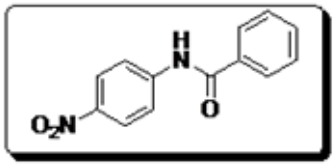
Mol Weight = 276

Entry No. 14

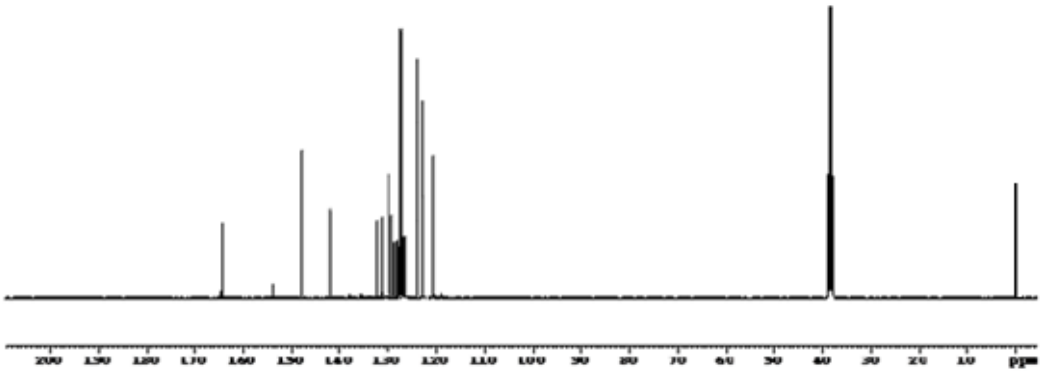
^{1}H-NMR Spectra of *N*-(4-Nitrorophenyl)benzamide (Table No. 2, Entry No15)



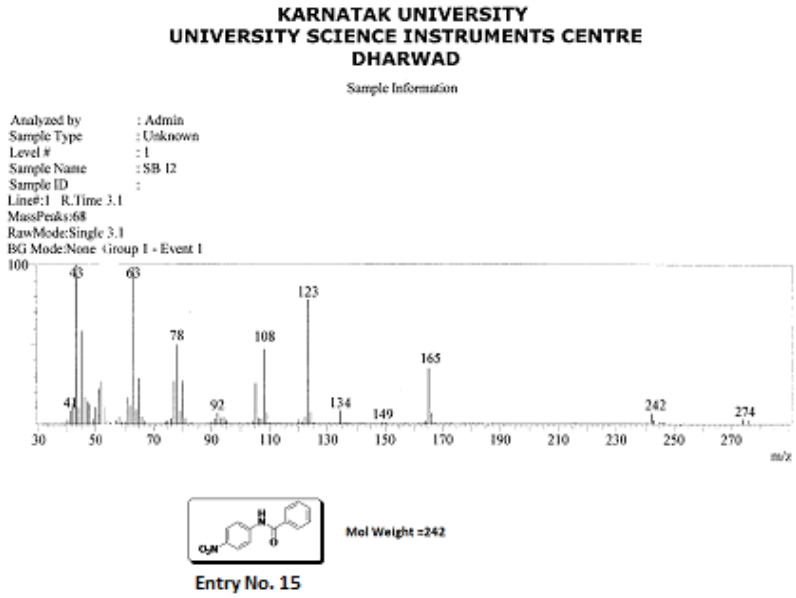
^{13}C-NMR Spectra *N*-(4-Nitrorophenyl)benzamide (Table No. 2, Entry No.15)



Entry No. 15

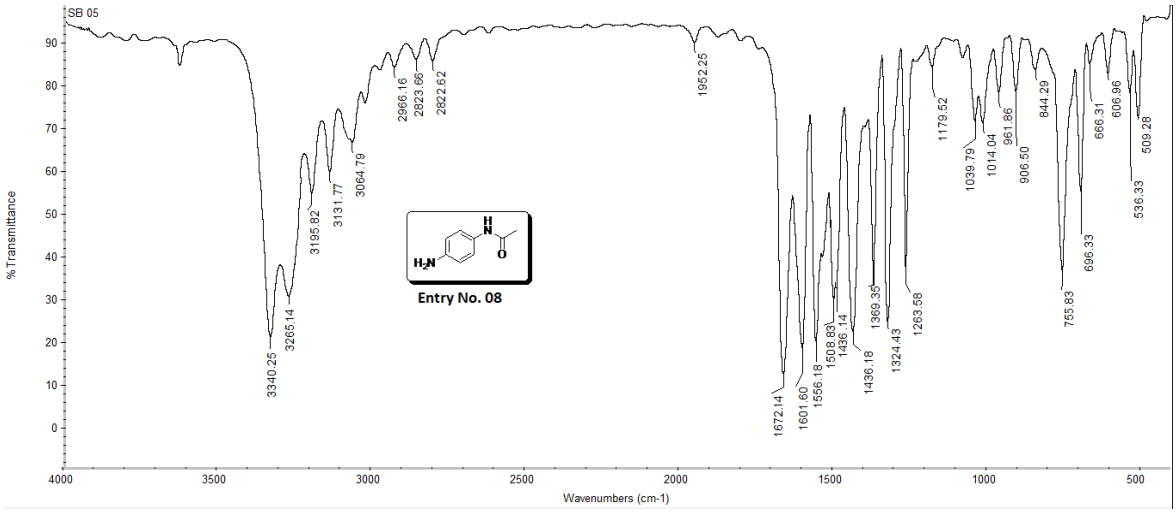


GCMS Spectra of *N*-(4-Nitrorophenyl)benzamide (Table No. 2, Entry No.15)

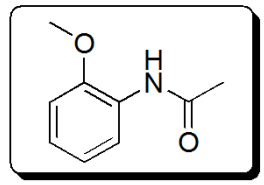


Entry No. 15

IR Spectra of *N*-(2-Methoxyphenyl)acetamide (Table No. 2, Entry No.16)

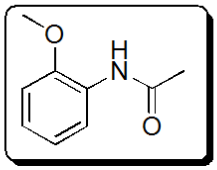


^{1}H-NMR Spectra of *N*-(2-Methoxyphenyl)acetamide (Table No. 2, Entry No16)



Entry No. 16

^{13}C NMR Spectra *N*-(2-Methoxyphenyl)acetamide (Table No. 2, Entry No.16)



Entry no.16

GCMS Spectra of *N*-(2-Methoxyphenyl)acetamide (Table No. 2, Entry No.16)

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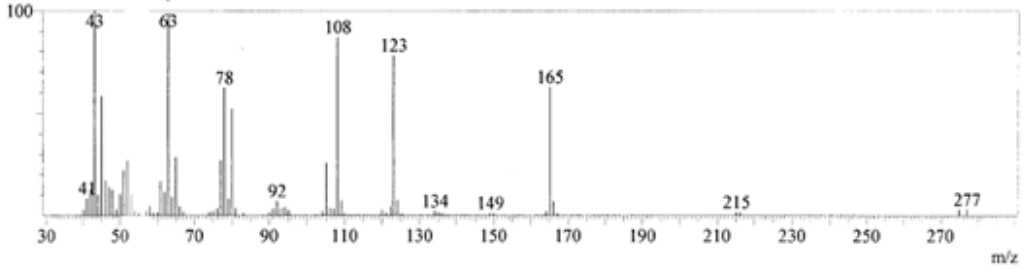
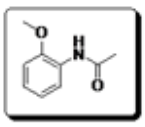
Sample Information

Analyzed by : Admin
Sample Type : Unknown
Level # : 1
Sample Name : SB 13
Sample ID :
Line#:1 R.Time 3.1
MassPeaks:68
RawMode:Single 3.1
BG Mode:None Group 1 - Event 1

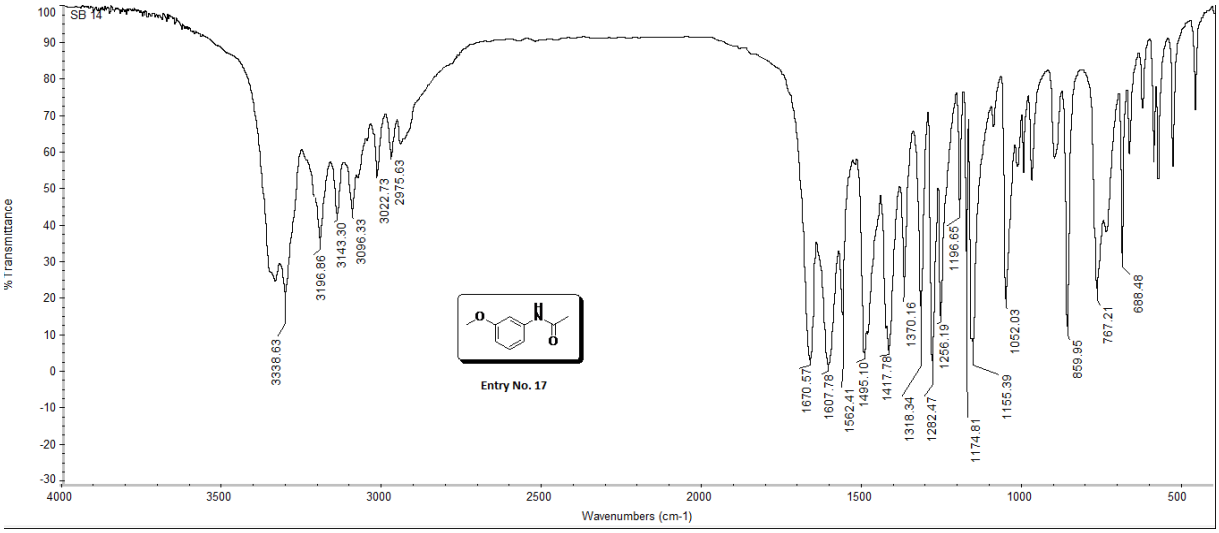




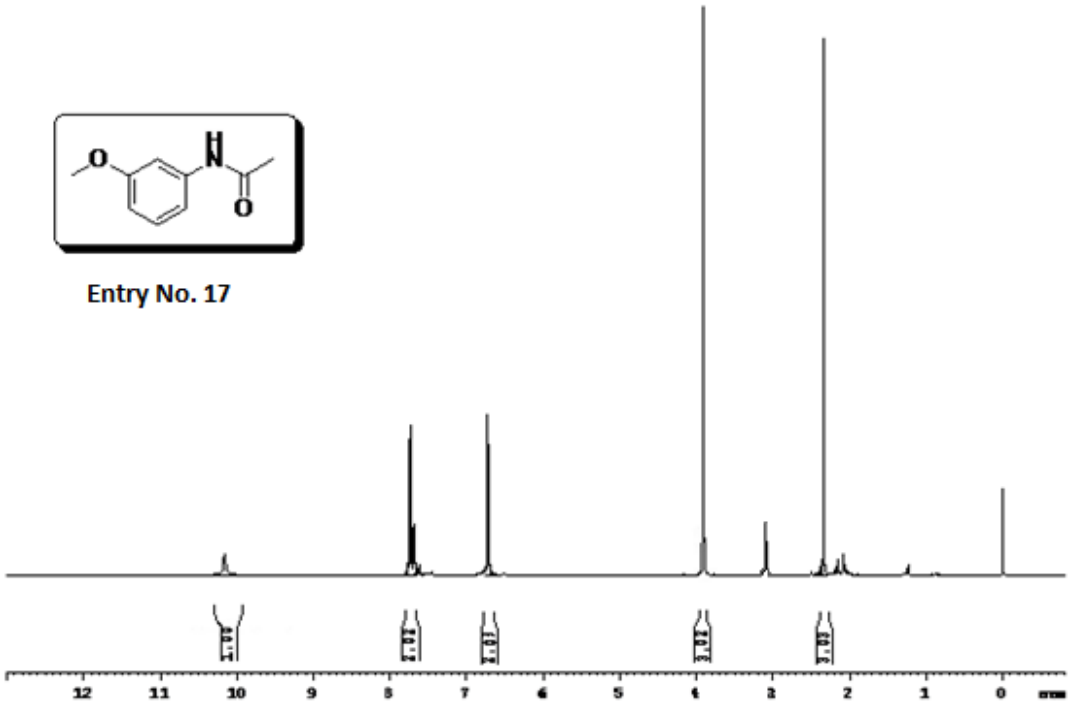
Mol Weight =165

Entry No. 16

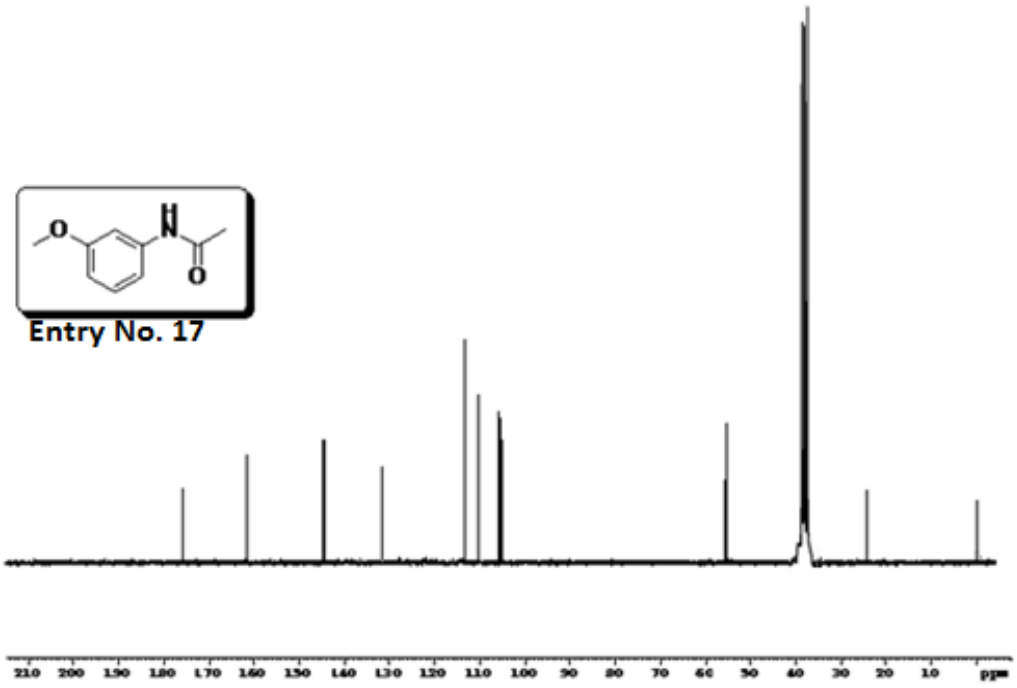
IR Spectra of *N*-(3-Methoxyphenyl)acetamide (Table No. 2, Entry No.17)



^{1}H-NMR Spectra of *N*-(3-Methoxyphenyl)acetamide (Table No. 2, Entry No17)



^{13}C-NMR Spectra *N*-(3-Methoxyphenyl)acetamide (Table No. 2, Entry No.17)



GCMS Spectra of *N*-(3-Methoxyphenyl)acetamide (Table No. 2, Entry No.17)

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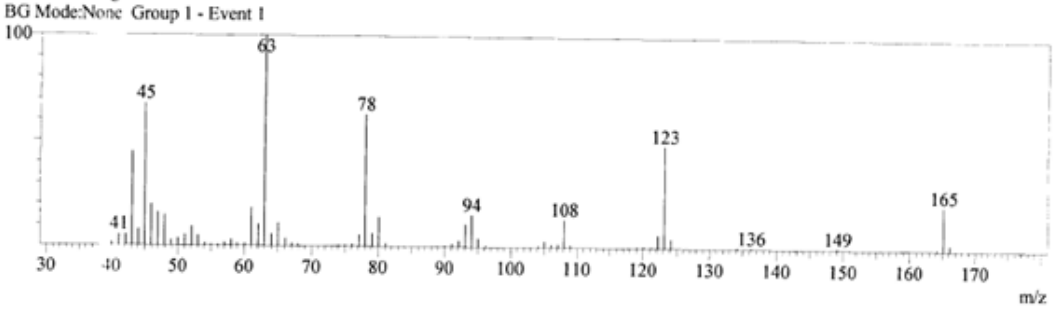
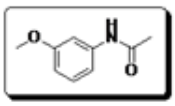
Sample Information

Analyzed by : Admin
Sample Type : Unknown
Level # : 1
Sample Name : SB 14
Sample ID :
Line#:1 R.Time:3.0
MassPeaks:62
RawMode:Single 3.0
BG Mode:None Group 1 - Event 1

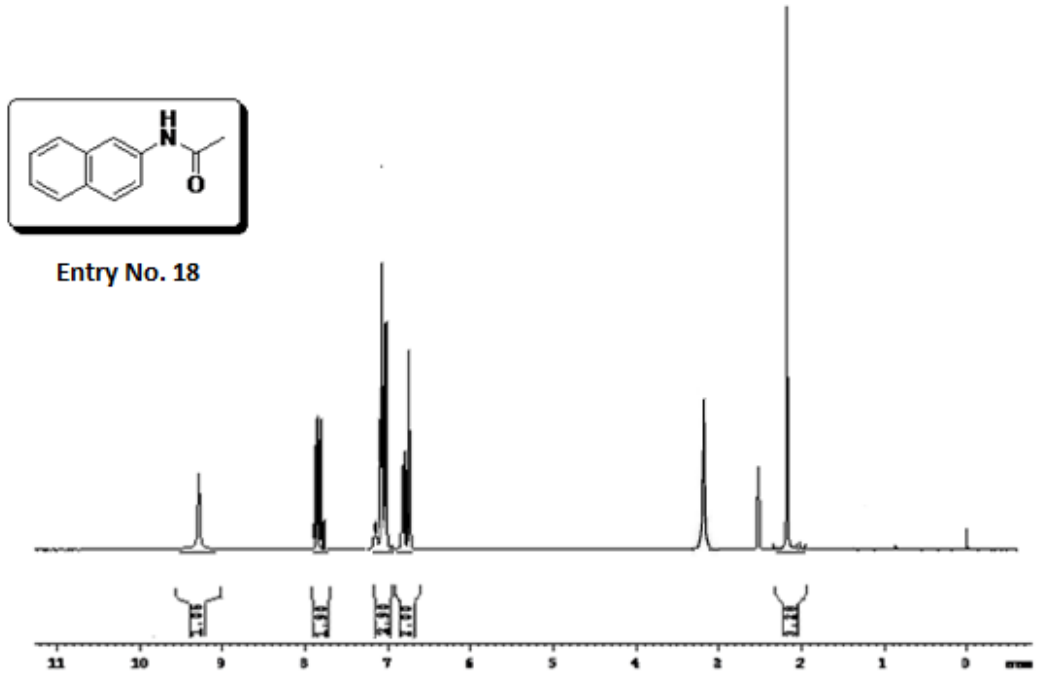




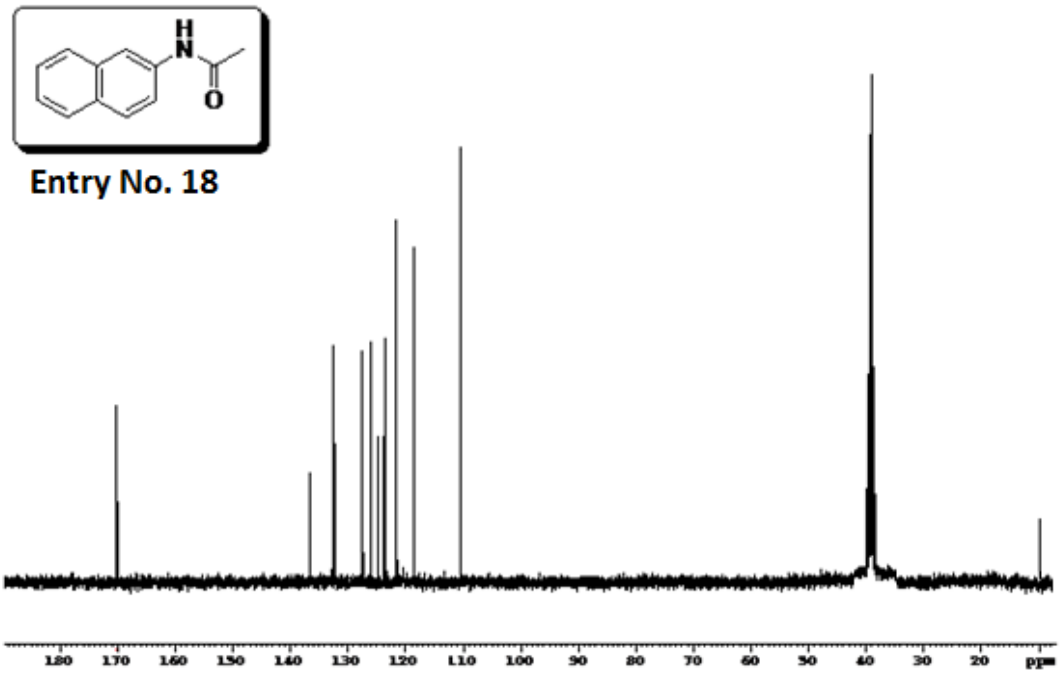
Mol Weight = 165

Entry No. 17

^{1}H-NMR Spectra *N*-(Naphthalene-2-yl)acetamide (Table No. 2, Entry No18)



^{13}C-NMR Spectra *N*-(Naphthalene-2-yl)acetamide (Table No. 2, Entry No.18)



GCMS Spectra of *N*-(Naphthalene-2-yl)acetamide (Table No. 2, Entry No.18)

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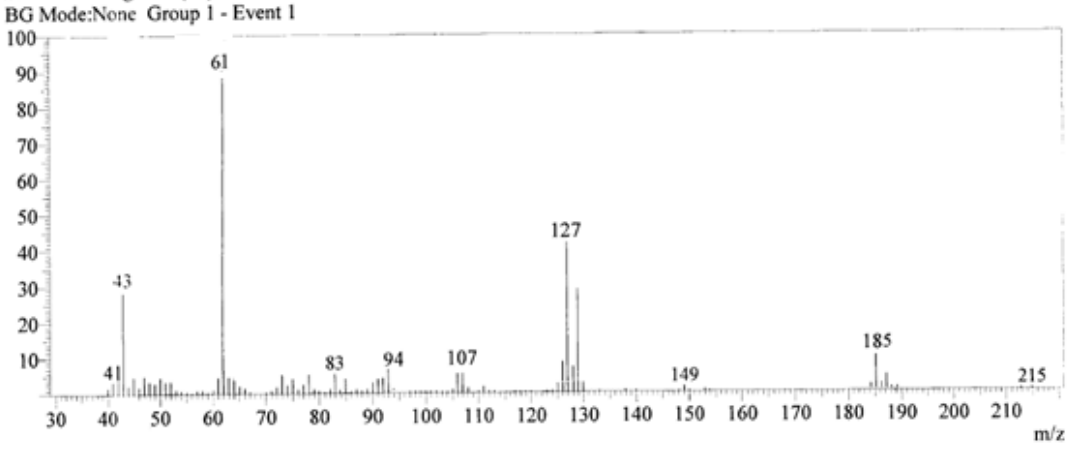
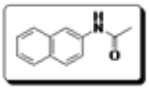
Sample Information

Analyzed by : Admin
Sample Type : Unknown
Level # : 1
Sample Name : SB 15
Sample ID :
IS Amount : [1]=1
Line#:1 R.Time:3.1
MassPeaks:
RawMode:Single 0.1(11)
BG Mode:None Group 1 - Event 1

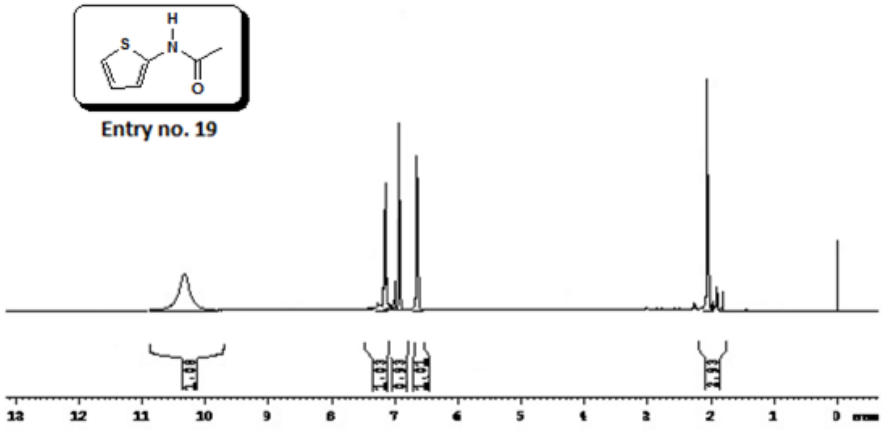




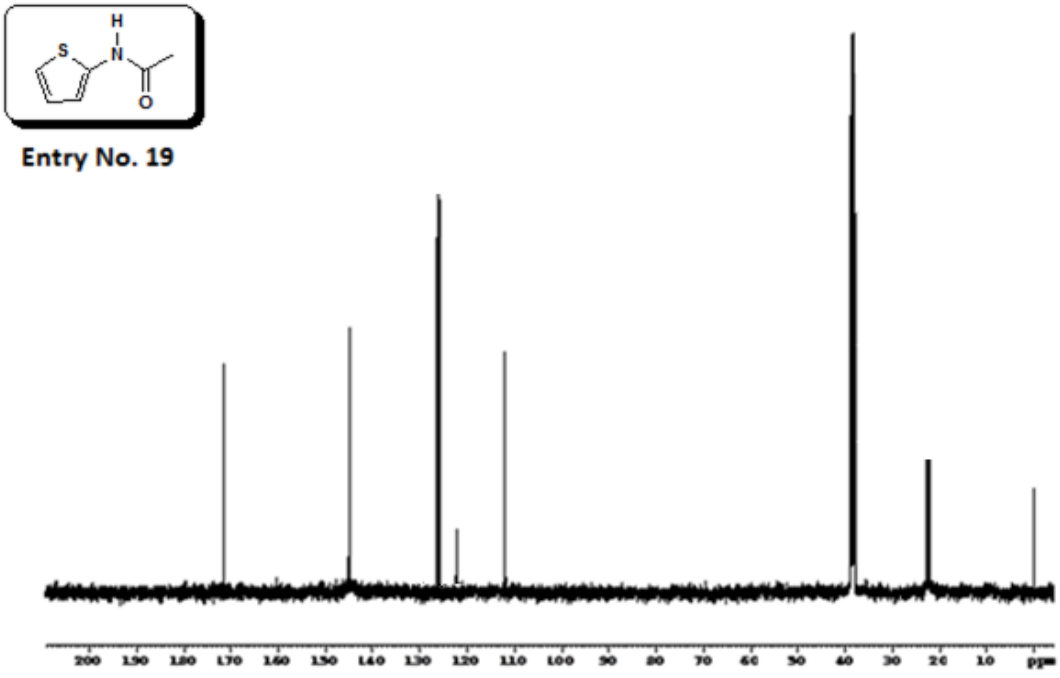
mol. Weight = 185

Entry No. 18

^{1}H-NMR Spectra *N*-(thiophen-2-yl)acetamide (Table No. 2, Entry No19)



^{13}C-NMR Spectra *N*-(thiophen-2-yl)acetamide (Table No. 2, Entry No.19)



GCMS Spectra of *N*-(thiophen-2-yl)acetamide (Table No. 2, Entry No.19)

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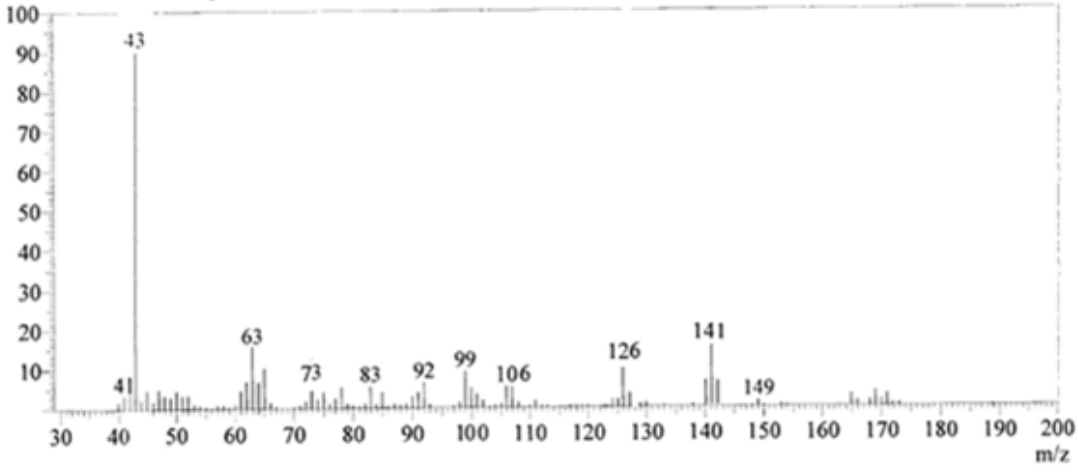
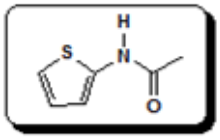
Sample Information

Analyzed by : Admin
Sample Type : Unknown
Level # : 1
Sample Name : SB 1B
Sample ID :
IS Amount : [1]=1
Line#:1 R.Time:0.1
MassPeaks:96
RawMode:Single 0.1(11)
BG Mode:None Group 1 - Event 1





Mol. Weight = 141

Entry No. 19