

Palladium-Catalyzed Fluorocarbonylation Using *N*-Formylsaccharin as CO Source: General Access to Carboxylic Acid Derivatives

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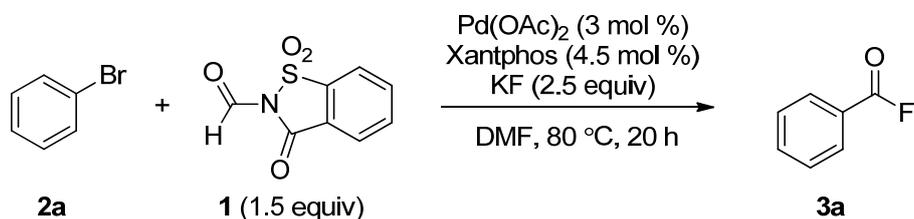
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1. General method and materials

General. All reactions were performed in oven-dried or flame-dried glassware under N₂ atmosphere. Reactions were monitored by TLC on Merck silica gel 60 F254 plates visualized by UV lamp at 254 nm. Column chromatography was performed on Merck silica gel 60 and preparative TLC was performed on Merck silica gel 60 F254 1.0 mm plates. Gas chromatography (GC) analyses were conducted on Agilent 6890N. DB-1 was used as a GC column. High performance liquid chromatography (HPLC) analyses were conducted on Shimazu LC-10Avp. YMC Pack Pro C18 (4.6 × 100 mm) was used as a HPLC column. A mobile phase was a mixed solution of acetonitrile and 0.2% aq. TFA (40/60–90/10). NMR spectra were measured on a JEOL JNM-ECA500 NMR spectrometer or JEOL JNM-LA400 NMR spectrometer at 500 or 400 MHz for ¹H spectra and 125 or 100 MHz for ¹³C spectra, and for ¹H NMR, tetramethylsilane (TMS) (δ = 0) in CDCl₃ served as an internal standard. For ¹³C NMR, CDCl₃ (δ = 77.0) served as an internal standard. Infrared spectra were measured on a JASCO FT/IR-6100 type A or S.T.Japan Durascope (ATR). High-resolution mass spectra (HRMS) were measured on a JEOL JMS-T100GC mass spectrometer and Waters Xevo Q-Tof ESI mass spectrometer.

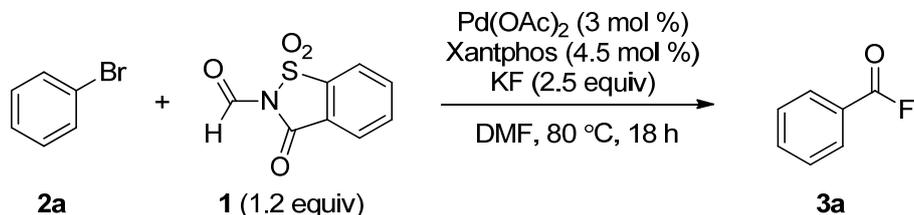
Materials. Commercially available chemicals including Pd catalysts, ligands, and *N*-formylsaccharin were purchased from Tokyo Chemical Industry Co., Ltd. and used as received. Super dehydrated DMF and KF powder (>95%) were purchased from Wako Pure Chemical Industries, Ltd. and used as received.

2. Experimental procedures for fluorocarbonylation (Table 1, entry 12)



$\text{Pd}(\text{OAc})_2$ (4.3 mg, 0.019 mmol, 3.0 mol %), Xantphos (16.6 mg, 0.029 mmol, 4.5 mol %), **1** (202 mg, 0.96 mmol, 1.2 equiv), and KF (93 mg, 1.59 mmol, 2.5 equiv) were added to a 10-mL test tube. The test tube was evacuated and backfilled with N_2 three times. Then, a degassed solution of **2a** (67 μL , 0.64 mmol, 1.0 equiv) in DMF (2 mL) was added to the test tube under flowing N_2 . After that, the test tube was sealed by a plastic screw cap. The mixture was warmed to 80 °C and stirred for 20 h. The reaction mixture was transferred to a 10-mL volumetric flask, diluted with CH_3CN to make exactly 10mL, and filtered through syringe filter. This solution was analyzed by GC to determine the conversion and yield. The concentration of **3a** in the solution was calculated from the ratio between GC peak area of **3a** in the solution and that in standard solution (concentration of **3a** in the standard solution : ca.10 mg/mL). GC yield (65.8 mg, 83.3%) was derived from the concentration of **3a**. The conversion (85.5%) of the reaction was derived from the concentration of **2a** in the solution calculated by the same procedure as above.

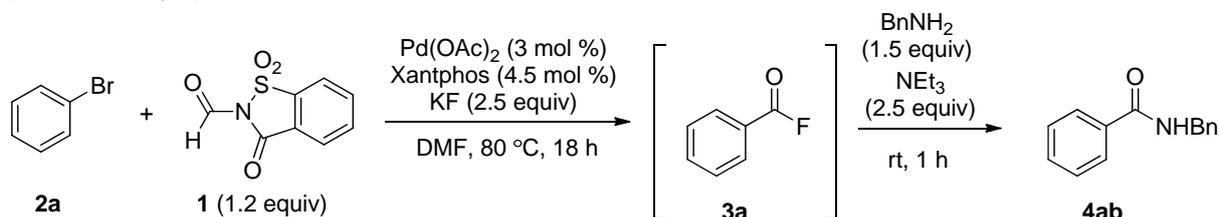
(Table 2, entry 6)



$\text{Pd}(\text{OAc})_2$ (4.3 mg, 0.019 mmol, 3.0 mol %), Xantphos (16.6 mg, 0.029 mmol, 4.5 mol %), **1** (161 mg, 0.76 mmol, 1.2 equiv), and KF (93 mg, 1.59 mmol, 2.5 equiv) were added to a 10-mL test tube. The test tube was evacuated and backfilled with N_2 three times. Then, a degassed solution of **2a** (67 μL , 0.64 mmol, 1.0 equiv) in DMF (3 mL) was added to the test tube under flowing N_2 . After that, the test tube was sealed by a plastic screw cap. The mixture was warmed to 80 °C and stirred for 18 h. The reaction mixture was directly purified by flash column chromatography on silica gel (ether/*n*-pentane = 1/50) to afford **3a** (40.5 mg, 51%) as a colorless oil.

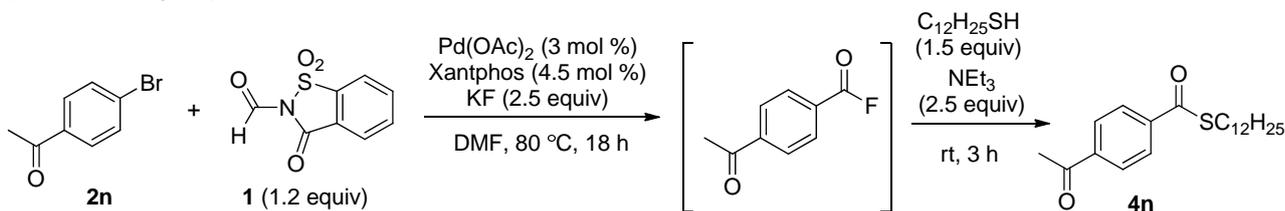
3. Representative procedures for fluorocarbonylation and the one-pot transformation

(Table 3, entry 1)



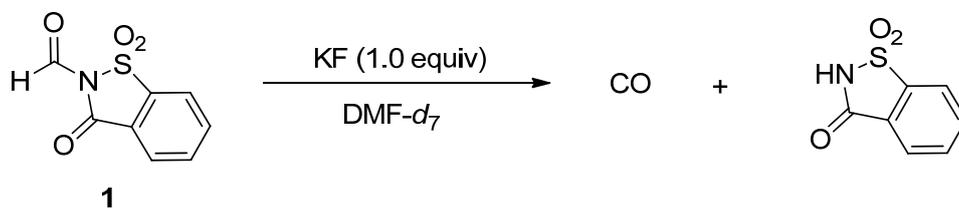
Pd(OAc)₂ (4.0 mg, 0.018 mmol, 3.0 mol %), Xantphos (15.6 mg, 0.027 mmol, 4.5 mol %), **1** (152 mg, 0.72 mmol, 1.2 equiv), and KF (87 mg, 1.50 mmol, 2.5 equiv) were added to a 10-mL test tube. The test tube was evacuated and backfilled with N₂ three times. Then, a degassed solution of **2a** (63 μL, 0.60 mmol, 1.0 equiv) in DMF (3 mL) was added to the test tube under flowing N₂. After that, the test tube was sealed by a plastic screw cap. The mixture was warmed to 80 °C and stirred for 18 h. After the reaction, the reaction mixture was cooled to rt. NEt₃ (208 μL, 1.50 mmol, 2.5 equiv) and BnNH₂ (98 μL, 0.90 mmol, 1.5 equiv) were added and the mixture was stirred for 1 h at rt. 10 mL of EtOAc and 10 mL of H₂O was added, the organic layer was separated, and the aqueous layer was extracted with 10 mL of EtOAc. The combined organic layer was dried over MgSO₄, filtered, and concentrated. The residue was purified by PTLC (SiO₂, EtOAc/hexane = 1/1) to afford **4ab** (107.8 mg, 85%) as a white crystal.

(Table 3, entry 13)



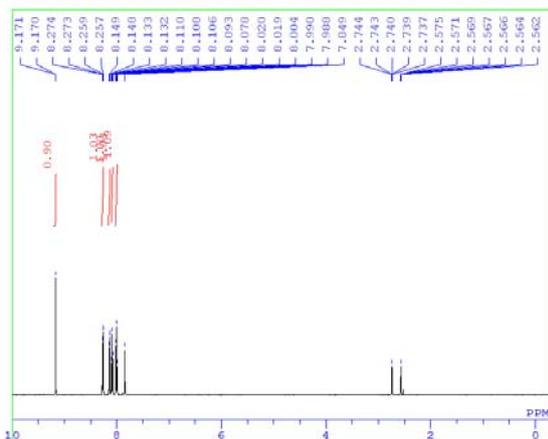
Pd(OAc)₂ (4.0 mg, 0.018 mmol, 3.0 mol %), Xantphos (15.6 mg, 0.027 mmol, 4.5 mol %), **1** (152 mg, 0.72 mmol, 1.2 equiv), **2n** (119 mg, 0.60 mmol, 1.0 equiv), and KF (87 mg, 1.50 mmol, 2.5 equiv) were added to a 10-mL test tube. The test tube was evacuated and backfilled with N₂ three times. Then, a degassed DMF (3 mL) was added to the test tube under flowing N₂. After that, the test tube was sealed by a plastic screw cap. The mixture was warmed to 80 °C and stirred for 18 h. After the reaction, the reaction mixture was cooled to rt. NEt₃ (208 μL, 1.50 mmol, 2.5 equiv) and C₁₂H₂₅SH (217 μL, 0.90 mmol, 1.5 equiv) were added and the mixture was stirred for 3 h at rt. 10 mL of EtOAc and 10 mL of H₂O was added, the organic layer was separated, and the aqueous layer was extracted with 10 mL of EtOAc. The combined organic layer was dried over MgSO₄, filtered, and concentrated. The residue was purified by PTLC (SiO₂, EtOAc/hexane = 10/1) to afford **4n** (160.2 mg, 79%) as a white crystal.

4. Experimental procedure for decarboxylation of *N*-formylsaccharin (**1**)



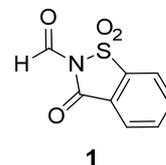
KF (10.5 mg, 0.18 mmol, 1.0 equiv) was added to the solution of *N*-formylsaccharin (38 mg, 0.18 mmol) in *DMF-d*₇ (0.75 mL). The mixture was stirred at 30 or 60 °C. A small aliquot (200 μL) was taken out from the reaction mixture at a suitable interval and the conversion of **1** was analyzed by ¹H-NMR. Meanwhile, CO generation from the solution was observed and CO was detected by a gas detector (GOT-110A-2, Gastec Corporation).

¹H-NMR of **1** (DMF-d₇)

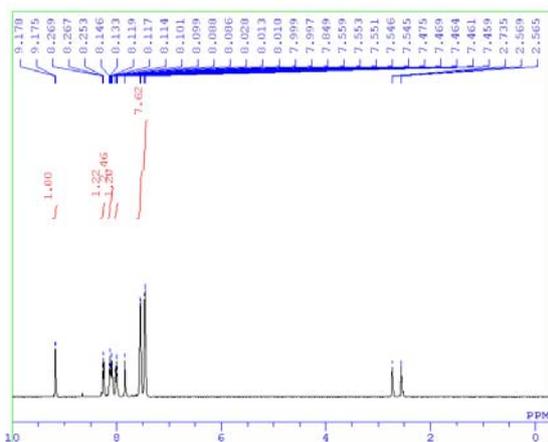


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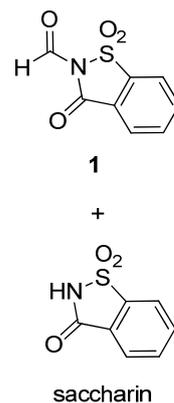


¹H-NMR at 2 h (30 °C) (DMF-d₇)



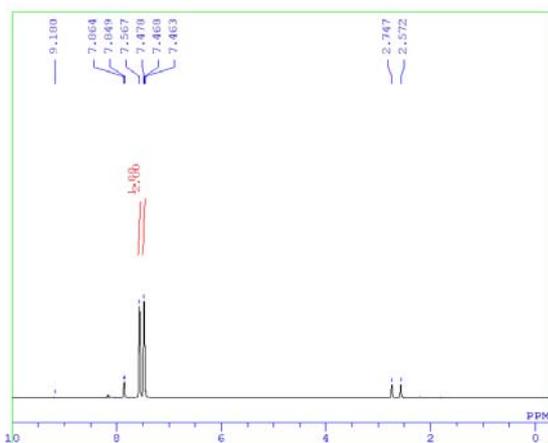
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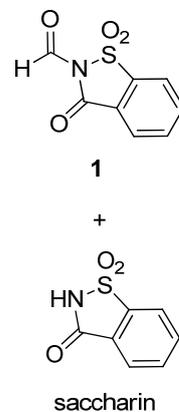
1/saccharin
= 34/66

¹H-NMR at 4 h (30 °C) (DMF-d₇)



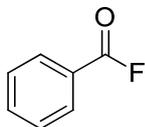
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1/saccharin
= trace/>99

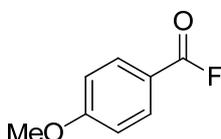
5. Analytical data of products



Benzoyl fluoride (3a)

3a was obtained from **1** and **2a** as a colorless oil. Yield: 51%.

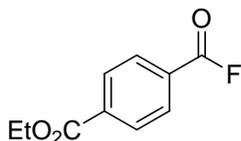
^1H NMR (400 MHz, CDCl_3) δ : 8.05 (d, 2H, $J = 6.8$ Hz), 7.71 (t, 1H, $J = 8.0$ Hz), 7.53 (dd, 2H, $J = 6.8, 8.0$ Hz); ^{13}C NMR (100 MHz, CDCl_3) δ : 157.4 (d, $^1J_{\text{C-F}} = 343.1$ Hz), 135.3, 131.4 (d, $^3J_{\text{C-F}} = 4.2$ Hz), 129.0 (d, $^4J_{\text{C-F}} = 1.7$ Hz), and 124.9 (d, $^2J_{\text{C-F}} = 60.1$ Hz); IR (ATR) 1784, 1724, 1687, 1452, 1211, 1173, 1038, 1014, 995, and 702 cm^{-1} ; HRMS (CI^+) $[\text{M}+\text{H}]^+$ calcd for $\text{C}_7\text{H}_6\text{FO}$: 125.0403; found 125.0401.



4-Methoxybenzoyl fluoride (3b)

3b was obtained from **1** and **2b** as a colorless oil. Yield: 73%.

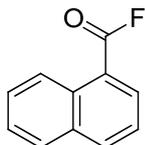
^1H -NMR (500 MHz, CDCl_3) δ : 8.00 (d, 2H, $J = 9.0$ Hz), 6.98 (dd, 2H, $J = 9.0, 1.0$ Hz), 3.90 (s, 3H). ^{13}C -NMR (125 MHz, CDCl_3) δ : 165.2, 157.3 (d, $^1J_{\text{C-F}} = 339.8$ Hz), 133.7 (d, $^3J_{\text{C-F}} = 4.0$ Hz), 116.8 (d, $^2J_{\text{C-F}} = 61.9$ Hz), 114.4 (d, $^4J_{\text{C-F}} = 1.8$ Hz), 55.6; IR (ATR) 1791, 1604, 1511, 1246, 1167, 1016, 998, 845, 758, 688, and 599 cm^{-1} ; HRMS (CI^+) $[\text{M}+\text{H}]^+$ calcd for $\text{C}_8\text{H}_8\text{FO}_2$: 155.0502; found 155.0508.



4-Ethoxycarbonylbenzoyl fluoride (3c)

3c was obtained from **1** and **2c** as a colorless oil. Yield: 68%.

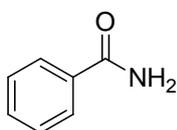
^1H -NMR (500 MHz, CDCl_3) δ : 8.19 (d, 2H, $J = 8.0$ Hz), 8.12 (d, 2H, $J = 8.0$ Hz), 4.43 (q, 2H, $J = 7.5$ Hz), 1.43 (t, 3H, $J = 7.5$ Hz). ^{13}C -NMR (125 MHz, CDCl_3) δ : 165.1, 157.6 (d, $^1J_{\text{C-F}} = 346.2$ Hz), 136.4, 131.3 (d, $^3J_{\text{C-F}} = 4.0$ Hz), 130.0 (d, $^4J_{\text{C-F}} = 1.1$ Hz), 128.5 (d, $^2J_{\text{C-F}} = 61.9$ Hz), 61.8, 14.2; IR (ATR) 1811, 1718, 1273, 1233, 1103, 1008, and 716 cm^{-1} ; HRMS (CI^+) $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{10}\text{H}_{10}\text{FO}_3$: 197.0611; found 197.0614.



1-Naphthoyl fluoride (3d)

3d was obtained from **1** and **2d** as a white crystal (m.p. 66 °C). Yield: 74%.

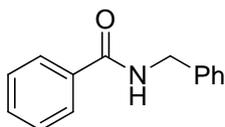
$^1\text{H-NMR}$ (500 MHz, CDCl_3) δ : 8.97 (d, 1H, $J = 8.5$ Hz), 8.29 (dd, 1H, $J = 7.0, 1.5$ Hz), 8.11 (d, 1H, $J = 8.5$ Hz), 7.88 (d, 1H, $J = 8.0$ Hz), 7.67 (ddd, 1H, $J = 8.0, 6.5, 1.0$ Hz), 7.57 (ddd, 1H, $J = 8.5, 7.0, 1.0$ Hz), 7.50 (t, 1H, $J = 8.0$ Hz). $^{13}\text{C-NMR}$ (125 MHz, CDCl_3) δ : 156.3 (d, $^1J_{\text{C-F}} = 344.5$ Hz), 136.6, 133.7 (d, $^3J_{\text{C-F}} = 4.0$ Hz), 133.6 (d, $^4J_{\text{C-F}} = 1.6$ Hz), 132.0 (d, $^3J_{\text{C-F}} = 7.0$ Hz), 129.1, 128.9, 126.9, 125.0 (d, $^4J_{\text{C-F}} = 1.1$ Hz), 124.4, 120.1 (d, $^2J_{\text{C-F}} = 55.5$ Hz); IR (ATR) 1786, 1237, 1181, 1081, 949, 770, 638, and 495 cm^{-1} ; HRMS (CI^+) $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{11}\text{H}_8\text{FO}$: 175.0562; found 175.0559.



Benzamide (4aa)

4aa was obtained from **1** and **2a** by using 10 equiv of 28% aq. NH_3 as a white crystal (m.p. 125 °C). Yield: 80%.

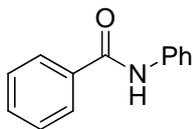
$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.82 (dd, 2H, $J = 8.8, 2.0$ Hz), 7.53 (t, 1H, $J = 6.8$ Hz), 7.44 (dd, 2H, $J = 8.8, 6.8$ Hz, 2H), 6.35 (brs, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 169.7, 133.3, 131.9, 128.6, and 127.3; IR (KBr pellet) 3368, 3171, 1659, 1625, 1578, 1403, 1122, 792, 770, 705, 685, 635, and 529 cm^{-1} ; HRMS (CI^+) $[\text{M}+\text{H}]^+$ calcd for $\text{C}_7\text{H}_8\text{NO}$: 122.0606; found 122.0603.



N-Benzylbenzamide (4ab)

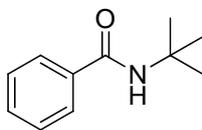
4ab was obtained from **1** and **2a**, **2b**, or **2c** by using 1.5 equiv of BnNH_2 as a white crystal (m.p. 104 °C). Yield: 85% from **2a**, 79% from **2b**, 79% from **2c**.

$^1\text{H NMR}$ (400 Hz, CDCl_3) δ : 7.77 (d, 2H, $J = 7.2$ Hz), 7.45 (t, 1H, $J = 7.2$ Hz), 7.38-7.24 (m, 7H), 6.87 (brs, 1H), 4.57 (d, 2H, $J = 6.0$ Hz); $^{13}\text{C NMR}$ (100 Hz, CDCl_3) δ : 167.4, 138.2, 134.2, 131.4, 128.6, 128.4, 127.7, 127.4, 126.9, and 43.9; IR (KBr pellet) 3324, 3296, 1640, 1546, 1314, 727, 693, and 666 cm^{-1} ; HRMS (CI^+) $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{14}\text{H}_{14}\text{NO}$: 212.1075; found 212.1072.



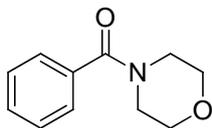
N-Phenylbenzamide (4ac)

4ac was obtained from **1** and **2a** by using 1.5 equiv of aniline as a white crystal (m.p. 161 °C). Yield: 82%. ¹H NMR (400 MHz, CDCl₃) δ: 8.00 (brs, 1H), 7.86 (d, 2H, *J* = 6.8 Hz), 7.64 (d, 2H, *J* = 8.0 Hz), 7.53 (t, 1H, *J* = 6.8 Hz), 7.46 (dd, 2H, *J* = 8.0, 6.8 Hz), 7.36 (dd, 2H, *J* = 8.0, 6.8 Hz), 7.15 (t, 1H, *J* = 8.0 Hz); ¹³C NMR (100 MHz, CDCl₃) δ: 165.8, 137.9, 134.9, 131.8, 129.0, 128.7, 127.0, 124.5, and 120.2; IR (KBr pellet) 3344, 1656, 1600, 1533, 1439, 1322, 1260, 750, 716, 690, and 650 cm⁻¹; HRMS (CI⁺) [M+H]⁺ calcd for C₁₃H₁₂NO: 198.0919; found 198.0922.



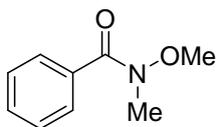
N-tert-Butylbenzamide (4ad)

4ad was obtained from **1** and **2a** by using 1.5 equiv of *tert*-butylamine as a white crystal (m.p. 136 °C). Yield: 89%. ¹H NMR (400 MHz, CDCl₃) δ 7.71 (d, 2H, *J* = 6.8 Hz), 7.44 (t, 1H, *J* = 8.0 Hz), 7.38 (dd, 2H, *J* = 6.8, 8.0 Hz), 6.09 (brs, 1H), 1.46 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 166.8, 135.7, 130.9, 128.3, 126.6, 51.4, and 28.7; IR (KBr pellet) 3322, 2979, 2965, 1635, 1541, 1492, 1312, 1228, 1218, 719, and 695 cm⁻¹; HRMS (CI⁺) [M+H]⁺ calcd for C₁₁H₁₆NO: 178.1232; found 178.1230.



Morpholino(phenyl)methanone (4ae)

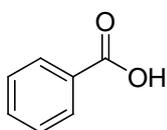
4ae was obtained from **1** and **2a** by using 1.5 equiv of morpholine as a white crystal (m.p. 73 °C). Yield: 80%. ¹H NMR (400 MHz, CDCl₃) δ 7.45-7.39 (m, 5H), 3.95-3.35 (m, 8H); ¹³C NMR (100 MHz, CDCl₃) δ 170.3, 135.2, 129.8, 128.4, 127.0, and 66.8 (one carbon signal is missing); IR (ATR) 1625, 1424, 1276, 1255, 1111, 1012, 708, 698, and 547 cm⁻¹; HRMS (CI⁺) [M+H]⁺ calcd for C₁₁H₁₄NO₂: 192.1025; found 192.1028.



N-Methoxy-N-methylbenzamide (4af)

4af was obtained from **1** and **2a** by using 1.5 equiv of *N,O*-dimethylhydroxylamine hydrochloride as a colorless oil. Yield: 89%.

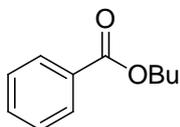
^1H NMR (400 MHz, CDCl_3) δ 7.66 (d, 2H, $J = 7.6$ Hz), 7.46-7.35 (m, 3H), 3.54 (s, 3H), 3.35 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 169.8, 133.9, 130.4, 128.0, 127.8, 60.9 and 33.7; IR (ATR) 1635, 1378, 1214, 977, 787, 703, 558, and 443 cm^{-1} ; HRMS (CI^+) $[\text{M}+\text{H}]^+$ calcd for $\text{C}_9\text{H}_{12}\text{NO}_2$: 166.0868; found 166.0862.



Benzoic acid (4ag)

4ag was obtained from **1** and **2a** by using 10 equiv of H_2O as a white crystal (m.p. 122 $^\circ\text{C}$). Yield: 95%.

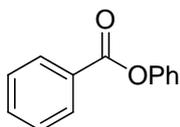
^1H NMR (400 MHz, CDCl_3) δ 11.88 (brs, 1H), 8.13 (d, 2H, $J = 7.6$ Hz), 7.62 (t, 1H, $J = 6.8$ Hz), 7.48 (dd, 2H, $J = 7.6, 6.8$ Hz); ^{13}C NMR (100 MHz, CDCl_3) δ 172.5, 133.8, 130.2, 129.3, and 128.5; IR (KBr pellet) 2835, 1686, 1454, 1425, 1327, 1292, 934, 708, 667, and 552 cm^{-1} ; HRMS (CI^+) $[\text{M}+\text{H}]^+$ calcd for $\text{C}_7\text{H}_7\text{O}_2$: 123.0446; found 123.0442.



Butyl benzoate (4ah)

4ah was obtained from **1** and **2a** by using 1.5 equiv of BuOH as a colorless oil. Yield: 80%.

^1H NMR (400 MHz, CDCl_3) δ 8.04 (d, 2H, $J = 8.8$ Hz), 7.54 (t, 1H, $J = 6.8$ Hz), 7.43 (dd, 2H, $J = 8.8, 6.8$ Hz), 4.32 (t, 2H, $J = 6.8$ Hz), 1.79-1.71 (m, 2H), 1.51-1.44 (m, 2H), 0.98 (t, 3H, $J = 8.0$ Hz); ^{13}C NMR (100 MHz, CDCl_3) δ 166.6, 132.7, 130.4, 129.4, 128.2, 64.7, 30.7, 19.2, and 13.7; IR (ATR) 1715, 1270, 1108, 1069, 1026, 707, and 687 cm^{-1} ; HRMS (CI^+) $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{11}\text{H}_{15}\text{O}_2$: 179.1072; found 179.1068.

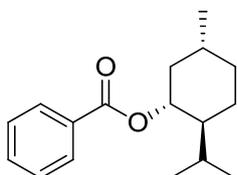


Phenyl benzoate (4ai)

4ai was obtained from **1** and **2a** by using 1.5 equiv of phenol as a white crystal (m.p. 68 $^\circ\text{C}$). Yield: 90%.

^1H NMR (400 MHz, CDCl_3) δ 8.21 (d, 2H, $J = 6.8$ Hz), 7.62 (t, 1H, $J = 7.3$ Hz), 7.48 (t, 2H, $J = 7.8$ Hz), 7.41 (t, 2H, $J = 7.3$ Hz), 7.26 (t, 1H, $J = 7.8$ Hz), 7.21 (d, 2H, $J = 7.8$ Hz); ^{13}C NMR (100 MHz, CDCl_3) δ

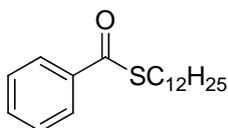
165.1, 150.9, 133.5, 130.1, 129.4, 128.5, 125.8, and 121.6 (one aromatic carbon signal is missing); IR (KBr pellet) 1730, 1487, 1262, 1200, 1063, 751, 704, and 694 cm^{-1} ; HRMS (CI^+) $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{13}\text{H}_{11}\text{O}_2$: 199.0759; found 199.0755.



L-Menthyl benzoate (4aj)

4aj was obtained from **1** and **2a** using 1.5 equiv of L-menthol as a colorless oil. Yield: 61%.

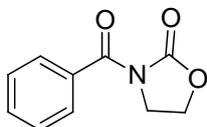
^1H NMR (400 Hz, CDCl_3) δ : 8.06-8.03 (m, 2H), 7.57-7.52 (m, 1H), 7.43 (t, 2H, $J = 7.6$ Hz), 4.98-4.90 (m, 1H), 2.15-2.10 (m, 1H), 1.99-1.95 (m, 1H), 1.76-1.71 (m, 2H), 1.60-1.52 (m, 2H), 1.18-1.05 (m, 2H), 0.93-0.89 (m, 7H), 0.79 (d, 3H, $J = 7.6$ Hz); ^{13}C NMR (100 Hz, CDCl_3) δ : 166.0, 132.6, 130.8, 129.5, 128.2, 74.7, 47.2, 40.9, 34.3, 31.4, 26.4, 23.5, 22.0, 20.7, and 16.4; IR (ATR) 2953, 1713, 1450, 1270, 1112, 1096, 1026, 962, and 709 cm^{-1} ; HRMS (CI^+) $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{17}\text{H}_{25}\text{O}_2$: 261.1855; found 261.1847; $[\alpha]_{\text{D}}^{25} = -86$ ($c = 0.4$, CHCl_3).



Dodecyl benzothioate (4ak)

4ak was obtained from **1** and **2a** by using 1.5 equiv of $\text{C}_{12}\text{H}_{25}\text{SH}$ as a yellow oil. Yield: 85%.

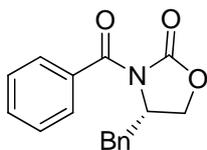
^1H NMR (400 Hz, CDCl_3) δ : 7.97 (d, 2H, $J = 8.8$ Hz), 7.54 (t, 1H, $J = 8.0$ Hz), 7.43 (dd, 2H, $J = 8.0, 8.8$ Hz), 3.06 (t, 2H, $J = 7.6$ Hz), 1.71-1.62 (m, 2H), 1.49-1.25 (m, 18H), 0.88 (t, 3H, $J = 6.0$ Hz); ^{13}C NMR (100 Hz, CDCl_3) δ : 192.0, 137.2, 133.1, 128.5, 127.1, 31.9, 29.60, 29.59, 29.55, 29.52, 29.46, 29.3, 29.1, 29.0, 28.9, 22.7, and 14.1; IR (ATR) 2922, 2852, 1663, 1204, 1174, 910, 771, 688, and 646 cm^{-1} ; HRMS (CI^+) $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{19}\text{H}_{31}\text{OS}$: 307.2096; found 307.2094.



N-Benzoyloxazolidin-2-one (4am)

4am was obtained from **1** and **2a** by using 1.5 equiv of oxazolidin-2-one as a white crystal (m.p. 168 $^{\circ}\text{C}$). Yield: 78%.

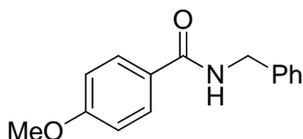
^1H NMR (500 MHz, CDCl_3) δ 7.65 (dd, 2H, $J = 6.8, 1.2$ Hz), 7.55 (tt, 1H, $J = 6.0, 1.2$ Hz), 7.43 (dd, 2H, $J = 6.8, 6.0$ Hz), 4.47 (t, 2H, $J = 6.4$ Hz), 4.16 (t, 2H, $J = 6.4$ Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 169.7,, 153.2, 132.5, 132.4, 129.0, 127.8, 62.2, and 43.6; IR (KBr pellet) 1776, 1677, 1380, 1339, 1214, 1201, 1108, 1039, 989, 912, 757, 721, 699, and 663 cm^{-1} ; HRMS (TOF MS ES^+) $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{10}\text{H}_{10}\text{NO}_3$: 192.0661; found 192.0659.



(S)-3-Benzoyl-4-benzyloxazolidin-2-one (4an)

4an was obtained from **1** and **2a** by using 1.5 equiv of (S)-4-benzyloxazolidin-2-one as a white crystal (m.p. 140 °C). Yield: 40%.

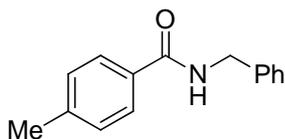
¹H NMR (400 MHz, CDCl₃) δ 7.65 (d, 2H, *J* = 7.6 Hz), 7.55 (t, 1H, *J* = 7.2 Hz), 7.43 (dd, 2H, *J* = 7.6, 7.2 Hz), 7.36-7.26 (m, 3H), 7.23 (d, 2H, *J* = 7.6 Hz), 4.90-4.83 (m, 1H), 4.31 (t, 1H, *J* = 8.0 Hz), 4.21 (dd, 1H, *J* = 4.8, 9.6 Hz), 3.42 (dd, 1H, *J* = 9.6, 2.8 Hz), 2.95 (dd, 1H, *J* = 8.8, 13.6 Hz); ¹³C NMR (100 MHz, CDCl₃) δ 169.8, 153.2, 134.9, 133.0, 132.4, 129.5, 129.0, 128.9, 127.8, 127.4, 66.3, 55.7, and 37.3; IR (KBr pellet) 1788, 1687, 1390, 1356, 1307, 1201, 1168, 1112, 1030, 717, 702, 693, and 505 cm⁻¹; HRMS (TOF MS ES⁺) [M+H]⁺ calcd for C₁₇H₁₆NO₃: 282.1130; found 282.1138; [α]_D²⁵ = +120 (*c* = 0.5, CHCl₃).



N-Benzyl-4-methoxybenzamide (4b)

4b was obtained from **1** and **2b** by using 1.5 equiv of BnNH₂ as a white crystal (m.p. 130 °C). Yield: 91%.

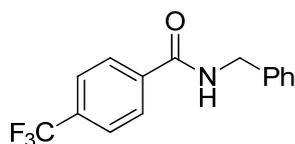
¹H NMR (400 MHz, CDCl₃) δ 7.75 (d, 2H, *J* = 8.8 Hz), 7.32-7.22 (m, 5H), 6.86 (d, 2H, *J* = 8.8 Hz), 6.67 (brs, 1H), 4.57 (d, 2H, *J* = 4.8 Hz), 3.81 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 166.9, 162.1, 138.4, 128.8, 128.6, 127.7, 127.4, 126.5, 113.6, 55.3, and 43.9; IR (KBr pellet) 3265, 1632, 1607, 1559, 1509, 1330, 1254, 1179, 1030, 843, 723, 696, and 687 cm⁻¹; HRMS (TOF MS ES⁺) [M+H]⁺ calcd for C₁₅H₁₆NO₂: 242.1181; found 242.1182.



N-Benzyl-4-methylbenzamide (4g)

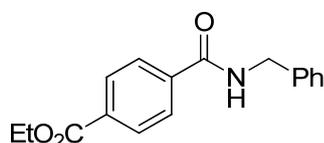
4g was obtained from **1** and **2g** by using 1.5 equiv of BnNH₂ as a white crystal (m.p. 132 °C). Yield: 91%.

¹H NMR (400 MHz, CDCl₃) δ 7.69 (d, 2H, *J* = 8.0 Hz), 7.33-7.24 (m, 5H), 7.18 (d, 2H, *J* = 8.0 Hz), 6.65 (brs, 1H), 4.59 (d, 2H, *J* = 6.0 Hz), 2.37 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 167.3, 141.8, 138.3, 131.4, 129.1, 128.6, 127.8, 127.4, 126.9, 43.9, and 21.4; IR (KBr pellet) 3311, 1640, 1548, 1324, 842, 721, 694, and 675 cm⁻¹; HRMS (CI⁺) [M+H]⁺ calcd for C₁₅H₁₆NO: 226.1232; found 226.1230.



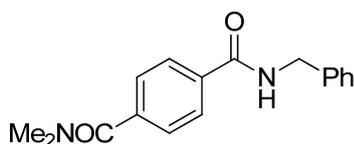
***N*-Benzyl-4-trifluoromethylbenzamide (4h)**

4h was obtained from **1** and **2h** by using 1.5 equiv of BnNH₂ as a white crystal (m.p. 168 °C). Yield: 72%. ¹H NMR (400 MHz, CDCl₃) δ 7.88 (d, 2H, *J* = 8.0 Hz), 7.66 (d, 2H, *J* = 8.0 Hz), 7.38-7.25 (m, 5H), 6.63 (brs, 1H), 4.63 (d, 2H, *J* = 5.6 Hz); ¹³C NMR (100 MHz, CDCl₃) δ 166.2, 137.7, 137.6, 133.2 (q, ²*J*_{C-F} = 32.1 Hz), 128.8, 127.9, 127.8, 127.4, 125.6 (q, ³*J*_{C-F} = 4.0 Hz), 123.6 (q, ¹*J*_{C-F} = 273.1 Hz), and 44.3; IR (KBr pellet) 3330, 1644, 1551, 1332, 1311, 1168, 1157, 1126, 1111, 1070, 862, 729, 696, and 672 cm⁻¹; HRMS (CI⁺) [M+H]⁺ calcd for C₁₅H₁₃F₃NO: 280.0949; found 280.0952.



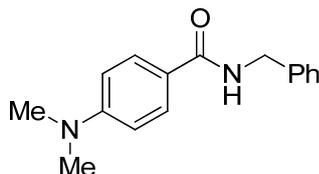
Ethyl 4-(benzylcarbamoyl)benzoate (4c)

4c was obtained from **1** and **2c** by using 1.5 equiv of BnNH₂ as a white crystal (m.p. 130 °C). Yield: 90%. ¹H NMR (400 MHz, CDCl₃) δ 8.02 (d, 2H, *J* = 8.8 Hz), 7.82 (d, 2H, *J* = 8.8 Hz), 7.33-7.26 (m, 5H), 7.00 (brs, 1H), 4.59 (d, 2H, *J* = 5.2 Hz), 4.36 (q, 2H, *J* = 6.8 Hz), 1.39 (t, 3H, *J* = 6.8 Hz); ¹³C NMR (100 MHz, CDCl₃) δ 166.6, 165.8, 138.1, 137.9, 132.9, 129.6, 128.7, 127.8, 127.5, 127.0, 61.3, 44.1, and 14.2; IR (KBr pellet) 3260, 1715, 1636, 1559, 1419, 1275, 1109, 1021, 871, 728, 702, and 679 cm⁻¹; HRMS (TOF MS ES⁺) [M+H]⁺ calcd for C₁₇H₁₈NO₃: 284.1287; found 284.1290.



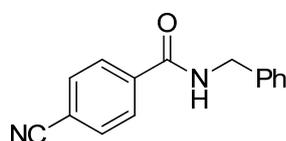
***N*¹-Benzyl-*N*⁴,*N*⁴-dimethylterephthalamide (4i)**

4i was obtained from **1** and **2i** by using 1.5 equiv of BnNH₂ as a white gum. Yield: 83%. ¹H NMR (400 MHz, CDCl₃) δ 7.75 (d, 2H, *J* = 8.0 Hz), 7.66 (brs, 1H), 7.32-7.22 (m, 7H), 4.57 (d, 2H, *J* = 5.6 Hz), 3.02 (s, 3H), 2.87 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 170.6, 166.7, 138.5, 138.4, 135.3, 128.4, 127.7, 127.6, 127.2, 126.7, 43.8, 39.2, and 35.1; IR (KBr pellet) 3346, 2922, 1662, 1617, 1608, 1538, 1492, 1408, 1393, 1085, 870, 742, 702, and 609 cm⁻¹; HRMS (TOF MS ES⁺) [M+H]⁺ calcd for C₁₇H₁₉N₂O₂: 283.1447; found 283.1442.



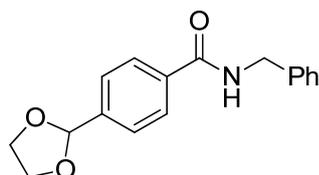
***N*-Benzyl-[4-(*N,N*-dimethylamino)]benzamide (**4j**)**

4j was obtained from **1** and **2j** by using 1.5 equiv of BnNH₂ as a white crystal (m.p. 166 °C). Yield: 72% ¹H NMR (400 Hz, CDCl₃) δ: 7.70 (d, 2H, *J* = 8.8 Hz), 7.31-7.22 (m, 5H), 6.62 (d, 2H, *J* = 8.8 Hz), 6.54 (brs, 1H), 4.57 (d, 2H, *J* = 6.0 Hz), 2.98 (s, 6H); ¹³C NMR (100 Hz, CDCl₃) δ: 167.3, 152.3, 138.8, 128.5, 128.4, 127.7, 127.2, 120.9, 110.9, 43.7, and 40.0; IR (KBr pellet) 3292, 1623, 1608, 1553, 1520, 1335, 1310, 1216, 830, and 716 cm⁻¹; HRMS (TOF MS ES⁺) [M+H]⁺ calcd for C₁₆H₁₉N₂O: 255.1497; found 255.1491.



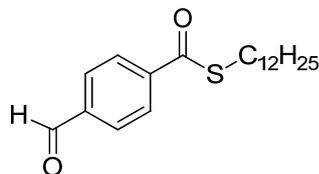
***N*-Benzyl-4-cyanobenzamide (**4k**)**

4k was obtained from **1** and **2k** by using 1.5 equiv of BnNH₂ as a white crystal (m.p. 151 °C). Yield: 82% ¹H NMR (400 Hz, CDCl₃) δ: 7.86 (d, 2H, *J* = 8.0 Hz), 7.66 (d, 2H, *J* = 8.0 Hz), 7.36-7.26 (m, 5H), 6.94 (brs, 1H), 4.60 (d, 2H, *J* = 6.0 Hz); ¹³C NMR (100 Hz, CDCl₃) δ: 165.6, 138.2, 137.5, 132.3, 128.8, 127.8, 127.73, 127.69, 117.9, 114.9, and 44.2; IR (KBr pellet) 3316, 2233, 1644, 1553, 1498, 1322, 1312, 865, 721, 694, and 675 cm⁻¹; HRMS (CI⁺) [M+H]⁺ calcd for C₁₅H₁₃N₂O: 237.1028; found 237.1026.



***N*-Benzyl-4-[1,3]dioxolan-2-ylbenzamide (**4l**)**

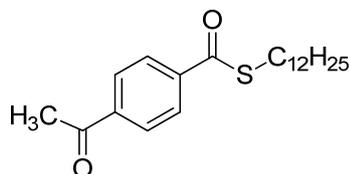
4l was obtained from **1** and **2l** by using 1.5 equiv of BnNH₂ as a white crystal (m.p. 131 °C). Yield: 88% ¹H NMR (400 Hz, CDCl₃) δ: 7.76 (d, 2H, *J* = 8.8 Hz), 7.45 (d, 2H, *J* = 8.8 Hz), 7.32-7.19 (m, 5H), 6.97 (brs, 1H), 5.78 (s, 1H), 4.54 (d, 2H, *J* = 6.0 Hz), 4.08-3.97 (m, 4H); ¹³C NMR (100 Hz, CDCl₃) δ: 167.0, 141.1, 138.1, 134.9, 128.5, 127.6, 127.3, 127.0, 126.5, 102.8, 65.2, 44.1, and 43.8; IR (KBr pellet) 3327, 1638, 1538, 1310, 1082, 976, 847, 741, and 699 cm⁻¹; HRMS (TOF MS ES⁺) [M+H]⁺ calcd for C₁₇H₁₈NO₃: 284.1287; found 284.1282.



S-Dodecyl-4-formylbenzenecarbothioate (4m)

4m was obtained from **1** and **2m** by using 1.5 equiv of C₁₂H₂₅SH as a white crystal (m.p. 55 °C). Yield: 76%

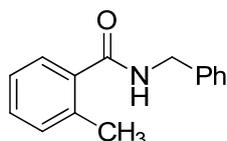
¹H NMR (400 Hz, CDCl₃) δ: 10.10 (s, 1H), 8.10 (d, 2H, *J* = 7.6 Hz), 7.96 (d, 2H, *J* = 7.6 Hz), 3.10 (t, 2H, *J* = 8.0 Hz), 1.73-1.64 (m, 2H), 1.47-1.25 (m, 18H), 0.88 (t, 3H, *J* = 6.8 Hz); ¹³C NMR (100 Hz, CDCl₃) δ: 191.4, 141.5, 139.1, 129.7, 127.7, 31.9, 29.6, 29.5, 29.43, 29.35, 29.32, 29.29, 29.1, 28.9, 22.6, and 14.1 (one carbon signal is missing); IR (KBr pellet) 2915, 2850, 1691, 1656, 1472, 1203, 924, 837, 826, 717, and 638 cm⁻¹; HRMS (CI⁺) [M+H]⁺ calcd for C₂₀H₃₁O₂S: 335.2045; found 335.2044



S-Dodecyl-4-acetylbenzenecarbothioate (4n)

4n was obtained from **1** and **2n** by using 1.5 equiv of C₁₂H₂₅SH as a white crystal (m.p. 68 °C). Yield: 79%

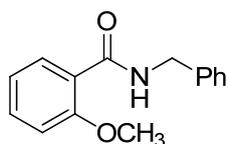
¹H NMR (400 Hz, CDCl₃) δ: 8.05-7.99 (m, 4H), 3.09 (t, 2H, *J* = 8.0 Hz), 2.64 (s, 3H), 1.72-1.64 (m, 2H), 1.45-1.24 (m, 18H), 0.87 (t, 3H, *J* = 6.8 Hz); ¹³C NMR (100 Hz, CDCl₃) δ: 197.2, 191.4, 140.4, 140.2, 128.4, 127.3, 31.8, 29.6, 29.5, 29.4, 29.33, 29.27, 29.24, 29.1, 28.9, 26.8, 22.6, and 14.1 (one carbon signal is missing); IR (KBr pellet) 2915, 2849, 1684, 1653, 1471, 1272, 1205, 923, 752, and 640 cm⁻¹; HRMS (CI⁺) [M+H]⁺ calcd for C₂₁H₃₃O₂S: 349.2201; found 349.2200.



N-Benzyl-2-methylbenzamide (4o)

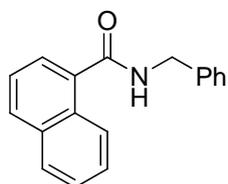
4o was obtained from **1** and **2o** by using 1.5 equiv of BnNH₂ as a white crystal (m.p. 105 °C). Yield: 87%

¹H NMR (400 Hz, CDCl₃) δ: 7.35-7.23 (m, 7H), 7.19-7.10 (m, 2H), 6.34 (brs, 1H), 4.53 (d, 2H, *J* = 5.6 Hz), 2.39 (s, 3H); ¹³C NMR (100 Hz, CDCl₃) δ: 169.9, 138.2, 136.1, 136.0, 130.9, 129.7, 128.6, 127.7, 127.4, 126.6, 125.5, 43.6, and 19.7; IR (KBr pellet) 3279, 1629, 1525, 1456, 1273, 746, 739, and 697 cm⁻¹; HRMS (CI⁺) [M+H]⁺ calcd for C₁₅H₁₆NO: 226.1232; found 226.1230.



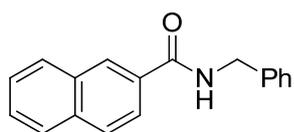
***N*-Benzyl-2-methoxybenzamide (4p)**

4p was obtained from **1** and **2p** by using 1.5 equiv of BnNH₂ as a white crystal (m.p. 85 °C). Yield: 33%
¹H NMR (400 Hz, CDCl₃) δ: 8.25 (dd, 1H, *J* = 7.6, 2.0 Hz), 8.24 (brs, 1H), 7.45-7.40 (m, 1H), 7.38-7.24 (m, 5H), 7.09 (t, 1H, *J* = 7.6 Hz), 6.97 (d, 1H, *J* = 8.8 Hz), 4.69 (d, 2H, *J* = 5.6 Hz), 3.91 (s, 3H); ¹³C NMR (100 Hz, CDCl₃) δ: 165.3, 157.4, 138.7, 132.8, 132.4, 128.6, 127.4, 127.2, 121.30, 121.28, 111.2, 55.9, and 43.7; IR (ATR) 3399, 1642, 1599, 1525, 1482, 1453, 1293, 1236, 1018, 753, 696, and 603 cm⁻¹; HRMS (CI⁺) [M+H]⁺ calcd for C₁₅H₁₆NO₂: 242.1181; found 242.1178.



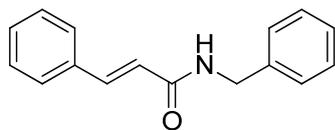
***N*-Benzyl-1-naphthamide (4d)**

4d was obtained from **1** and **2d** by using 1.5 equiv of BnNH₂ as a white crystal (m.p. 120 °C). Yield: 90%
¹H NMR (400 Hz, CDCl₃) δ: 8.24-8.21 (m, 1H), 7.81-7.76 (m, 2H), 7.47-7.42 (m, 3H), 7.30-7.22 (m, 6H), 6.68 (brs, 1H), 4.53 (d, 2H, *J* = 6.0 Hz); ¹³C NMR (100 Hz, CDCl₃) δ: 169.3, 138.1, 134.0, 133.4, 130.4, 130.0, 128.6, 128.1, 127.6, 127.3, 126.9, 126.2, 125.3, 124.8, 124.5, and 43.7; IR (KBr pellet) 3266, 1633, 1525, 1311, 1287, 1256, 783, 770, and 695 cm⁻¹; HRMS (CI⁺) [M+H]⁺ calcd for C₁₈H₁₆NO: 262.1232; found 262.1240.



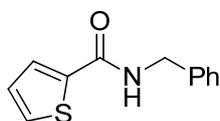
***N*-Benzyl-2-naphthamide (4q)**

4q was obtained from **1** and **2q** by using 1.5 equiv of BnNH₂ as a white crystal (m.p. 136 °C). Yield: 87%
¹H NMR (400 Hz, CDCl₃) δ: 8.29 (s, 1H), 7.85-7.83 (m, 4H), 7.56-7.47 (m, 2H), 7.38-7.21 (m, 5H), 6.82 (brs, 1H), 4.66 (d, 2H, *J* = 6.0 Hz); ¹³C NMR (100 Hz, CDCl₃) δ: 167.4, 138.2, 134.7, 132.5, 131.5, 128.8, 128.7, 128.4, 127.9, 127.7, 127.6, 127.5, 127.4, 126.7, 123.6, and 44.1; IR (KBr pellet) 3299, 1641, 1552, 1313, 829, 779, 719, 695, and 472 cm⁻¹; HRMS (CI⁺) [M+H]⁺ calcd for C₁₈H₁₆NO: 262.1232; found 262.1230.



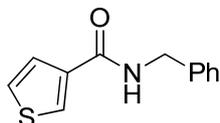
(E)-N-Benzyl-3-phenyl-2-propenamide (4r)

4r was obtained from **1** and **2r** by using 1.5 equiv of BnNH₂ as a white crystal (m.p. 108 °C). Yield: 83%
¹H NMR (500 Hz, CDCl₃) δ: 7.61 (d, 1H, *J* = 12.4 Hz), 7.42-7.39 (m, 2H), 7.29-7.20 (m, 8H), 6.85 (brs, 1H), 6.50 (d, 1H, *J* = 12.4 Hz), 4.46 (d, 1H, *J* = 4.8 Hz); ¹³C NMR (125 Hz, CDCl₃) δ: 166.0, 141.0, 138.1, 134.7, 129.5, 128.6, 128.5, 127.7, 127.6, 127.3, 120.6, and 43.6; IR (KBr pellet) 3263, 1652, 1614, 1542, 1449, 1328, 1221, 980, 757, 698, 674, and 535 cm⁻¹; HRMS (TOF MS ES⁺) [M+H]⁺ calcd for C₁₆H₁₆NO: 238.1232; found 238.1236.



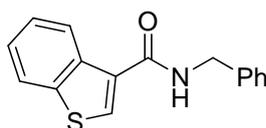
N-Benzylthiophene-2-carboxamide (4s)

4s was obtained from **1** and **2s** by using 1.5 equiv of BnNH₂ as a white crystal (m.p. 119 °C). Yield: 76%
¹H NMR (400 Hz, CDCl₃) δ: 7.53 (d, 1H, *J* = 4.0 Hz), 7.45 (d, 1H, *J* = 4.8 Hz), 7.34-7.25 (m, 5H), 7.03 (dd, 1H, *J* = 4.8, 4.0 Hz), 6.69 (brs, 1H), 4.56 (d, 2H, *J* = 6.0 Hz); ¹³C NMR (100 Hz, CDCl₃) δ: 161.8, 138.8, 138.0, 130.0, 128.6, 128.1, 127.8, 127.6, 127.5, and 43.8; IR (KBr pellet) 3352, 1622, 1544, 1423, 1304, 1247, 962, 861, 730, 717, 697, and 643 cm⁻¹; HRMS (TOF MS ES⁺) [M+H]⁺ calcd for C₁₂H₁₂NOS: 218.0640; found 218.0644.



N-Benzylthiophene-3-carboxamide (4t)

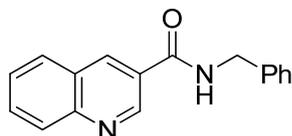
4t was obtained from **1** and **2t** by using 1.5 equiv of BnNH₂ as a white crystal (m.p. 100 °C). Yield: 81%
¹H NMR (400 Hz, CDCl₃) δ: 7.87 (d, 1H, *J* = 2.8 Hz), 7.40 (d, 1H, *J* = 4.8 Hz), 7.33-7.25 (m, 6H), 6.67 (brs, 1H), 4.55 (d, 2H, *J* = 6.0 Hz); ¹³C NMR (100 Hz, CDCl₃) δ: 163.0, 138.2, 137.2, 128.6, 128.4, 127.8, 127.4, 126.4, 126.1, and 43.7; IR (KBr pellet) 3304, 1637, 1552, 1309, 821, 718, and 693 cm⁻¹; HRMS (TOF MS ES⁺) [M+H]⁺ calcd for C₁₂H₁₂NOS: 218.0640; found 218.0635.



N-Benzylbenzo[b]thiophene-3-carboxamide (4u)

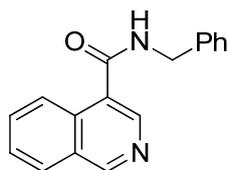
4u was obtained from **1** and **2u** by using 1.5 equiv of BnNH₂ as a white crystal (m.p. 146 °C). Yield: 88%
¹H NMR (500 Hz, CDCl₃) δ: 8.39 (d, 1H, *J* = 6.4 Hz), 7.84 (d, 1H, *J* = 6.8 Hz), 7.83 (s, 1H), 7.44-7.28 (m, 7H), 6.51 (brs, 1H), 4.64 (d, 2H, *J* = 4.4 Hz); ¹³C NMR (125 Hz, CDCl₃) δ: 163.9, 140.2, 138.1, 136.7,

131.8, 129.2, 128.8, 127.8, 127.6, 125.10, 125.09, 124.3, 122.5, and 43.8; IR (KBr pellet) 3277, 1626, 1542, 1433, 1425, 1270, 1253, 1233, 1032, 757, 744, 715, and 697 cm^{-1} ; HRMS (TOF MS ES^+) $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{16}\text{H}_{14}\text{NOS}$: 268.0796; found 268.0797.



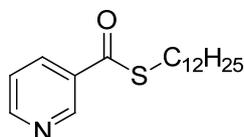
***N*-Benzylquinoline-3-carboxamide (4v)**

4v was obtained from **1** and **2v** by using 1.5 equiv of BnNH_2 as a white crystal (m.p. 140 $^\circ\text{C}$). Yield: 83% ^1H NMR (400 Hz, CDCl_3) δ : 9.24 (s, 1H), 8.55 (s, 1H), 8.00 (d, 1H, $J = 7.6$ Hz), 7.78-7.67 (m, 3H), 7.49 (t, 1H, $J = 6.8$ Hz), 7.30-7.18 (m, 5H), 4.60 (d, 2H, $J = 4.8$ Hz); ^{13}C NMR (100 Hz, CDCl_3) δ : 165.6, 148.8, 148.2, 137.8, 135.7, 131.1, 128.9, 128.6, 128.5, 127.7, 127.4, 127.3, 126.74, 126.66, and 44.0; IR (KBr pellet) 3268, 3060, 1662, 1546, 1501, 1304, 784, 743, and 694 cm^{-1} ; HRMS (TOF MS ES^+) $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{17}\text{H}_{15}\text{N}_2\text{O}$: 263.1184; found 263.1182.



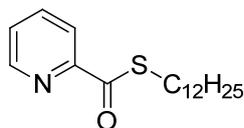
***N*-Benzylisoquinoline-4-carboxamide (4w)**

4w was obtained from **1** and **2w** by using 1.5 equiv of BnNH_2 as a white crystal (m.p. 168 $^\circ\text{C}$). Yield: 84% ^1H NMR (400 Hz, CDCl_3) δ : 8.87 (s, 1H), 8.38 (s, 1H), 8.26 (d, 1H, $J = 8.8$ Hz), 7.79 (d, 1H, $J = 7.6$ Hz), 7.64 (dd, 1H, $J = 7.6, 8.8$ Hz), 7.59-7.56 (m, 2H), 7.35-7.25 (m, 5H), 4.62 (d, 2H, $J = 6.0$ Hz); ^{13}C NMR (100 Hz, CDCl_3) δ : 167.1, 154.3, 140.9, 137.9, 132.7, 131.4, 128.6, 128.0, 127.73, 127.68, 127.63, 127.4, 127.2, 124.5, and 43.8; IR (KBr pellet) 3270, 1633, 1532, 1454, 1221, 797, 751, 696, and 599 cm^{-1} ; HRMS (TOF MS ES^+) $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{17}\text{H}_{15}\text{N}_2\text{O}$: 263.1184; found 263.1185.



***S*-Dodecyl pyridine-3-carbothioate (4x)**

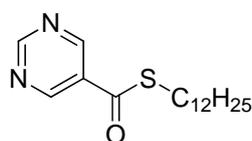
4x was obtained from **1** and **2x** by using 1.5 equiv of $\text{C}_{12}\text{H}_{25}\text{SH}$ as a colorless oil. Yield: 76% ^1H NMR (400 Hz, CDCl_3) δ : 9.18 (d, 1H, $J = 2.8$ Hz), 8.78 (dd, 1H, $J = 5.1, 2.0$ Hz), 8.22 (ddd, 1H, $J = 8.0, 2.8, 2.0$ Hz), 7.40 (dd, 1H, $J = 8.0, 5.1$ Hz), 3.10 (t, 2H, $J = 6.8$ Hz), 1.73-1.64 (m, 2H), 1.45-1.25 (m, 18H), 0.88 (t, 2H, $J = 6.8$ Hz); ^{13}C NMR (100 Hz, CDCl_3) δ : 190.6, 153.6, 148.4, 134.4, 132.7, 123.4, 31.9, 29.58, 29.56, 29.51, 29.42, 29.36, 29.29, 29.09, 29.07, 28.8, 22.6, and 14.1; IR (KBr pellet) 2914, 2849, 1659, 1472, 1417, 1227, 922, 716, and 698 cm^{-1} ; HRMS (TOF MS ES^+) $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{18}\text{H}_{30}\text{NOS}$: 308.2048; found 308.2050.



S-Dodecyl pyridine-2-carbothioate (4y)

4y was obtained from **1** and **2y** by using 1.5 equiv of C₁₂H₂₅SH as a colorless oil. Yield: 43%

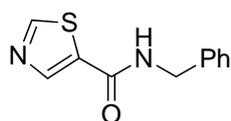
¹H NMR (400 Hz, CDCl₃) δ: 8.70 (d, 1H, *J* = 4.8 Hz), 7.97 (d, 1H, *J* = 8.0 Hz), 7.88-7.83 (m, 1H), 7.52-7.49 (m, 1H), 3.05 (t, 2H, *J* = 6.8 Hz), 1.73-1.65 (m, 2H), 1.48-1.25 (m, 18H), 0.88 (t, 2H, *J* = 6.8 Hz); ¹³C NMR (100 Hz, CDCl₃) δ: 193.6, 152.1, 149.1, 137.1, 127.6, 120.3, 31.9, 29.59, 29.57, 29.54, 29.46, 29.29, 29.25, 29.1, 28.9, 28.7, 22.6, and 14.1; IR (KBr pellet) 2916, 2849, 1662, 1471, 1224, 930, 791, 716, and 646 cm⁻¹; HRMS (TOF MS ES⁺) [M+H]⁺ calcd for C₁₈H₃₀NOS: 308.2048; found 308.2049.



S-Dodecyl pyrimidine-5-carbothioate (4z)

4z was obtained from **1** and **2z** by using 1.5 equiv of C₁₂H₂₅SH as a white crystal (m.p. 41 °C). Yield: 67%

¹H NMR (500 Hz, CDCl₃) δ: 9.37 (s, 1H), 9.22 (s, 2H), 3.15 (t, 2H, *J* = 6.0 Hz), 1.73-1.68 (m, 2H), 1.45-1.40 (m, 2H), 1.35-1.25 (m, 16H), 0.88 (t, 3H, *J* = 5.6 Hz); ¹³C NMR (125 Hz, CDCl₃) δ: 188.7, 161.5, 155.3, 130.4, 31.8, 29.55, 29.53, 29.48, 29.38, 29.26, 29.21, 29.18, 29.0, 28.8, 22.6, and 14.0; IR (KBr pellet) 2918, 2849, 1661, 1573, 1562, 1417, 1238, 1193, 919, 719, and 635 cm⁻¹; HRMS (TOF MS ES⁺) [M+H]⁺ calcd for C₁₇H₂₉N₂OS: 309.1993; found 309.2001.

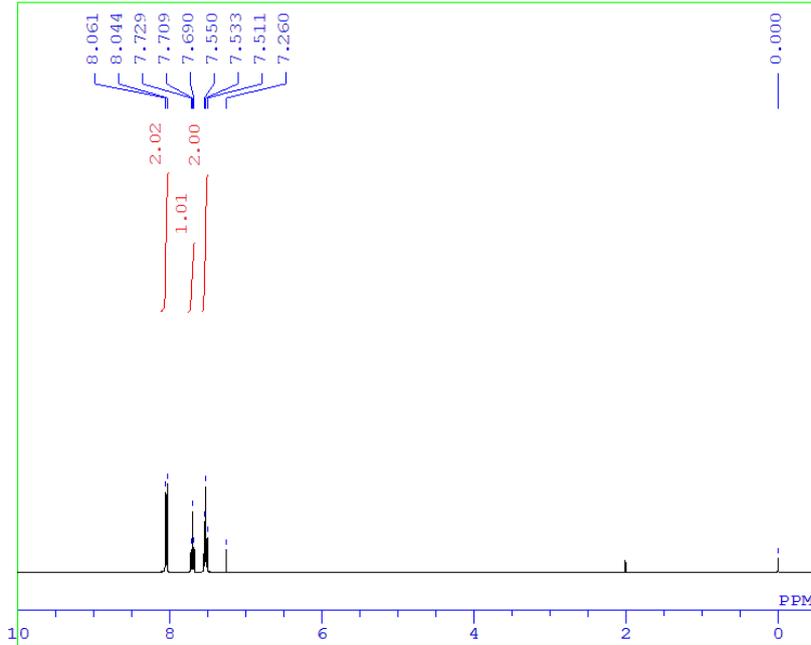
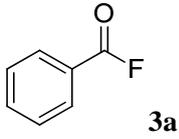


N-Benzylthiazole-5-carboxamide (4za)

4za was obtained from **1** and **2za** by using 1.5 equiv of BnNH₂ as a white crystal (m.p. 146 °C). Yield: 43%

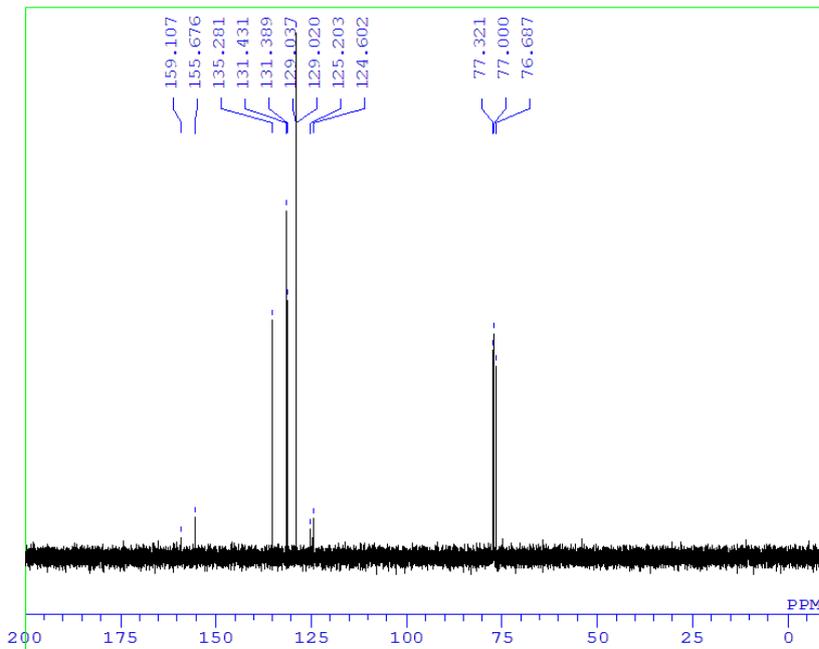
¹H NMR (500 Hz, CDCl₃) δ: 8.71 (d, 1H, *J* = 1.6 Hz), 8.20 (d, 1H, *J* = 1.6 Hz), 7.75 (brs, 1H), 7.37-7.26 (m, 5H), 4.65 (d, 2H, *J* = 4.8 Hz); ¹³C NMR (125 Hz, CDCl₃) δ: 160.8, 152.7, 150.9, 138.0, 128.7, 127.8, 127.5, 123.3, and 43.3; IR (ATR) 3293, 3077, 1648, 1544, 1476, 1268, 924, 879, 725, 693, and 623 cm⁻¹; HRMS (TOF MS ES⁺) [M+H]⁺ calcd for C₁₁H₁₁N₂OS: 219.0592; found 219.0591.

7. NMR spectra of products



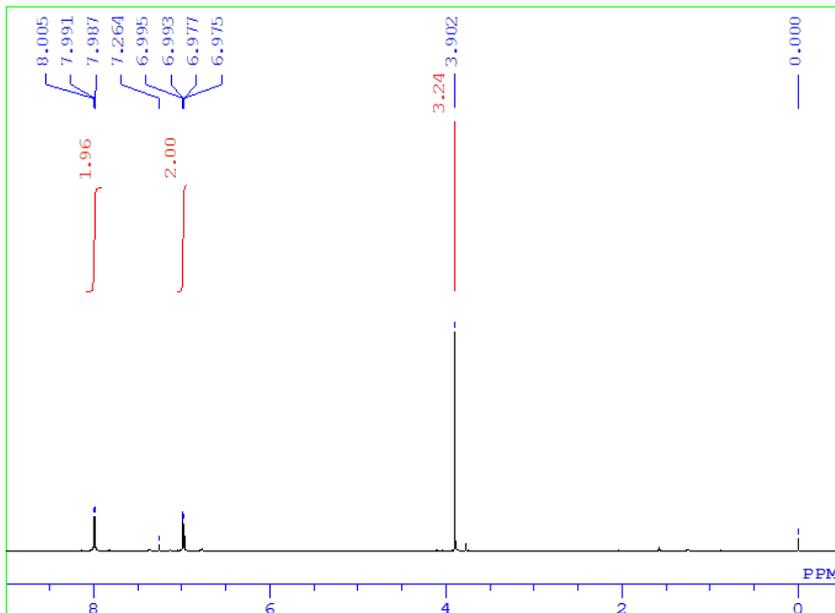
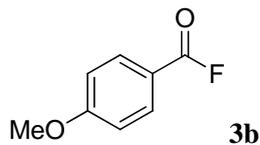
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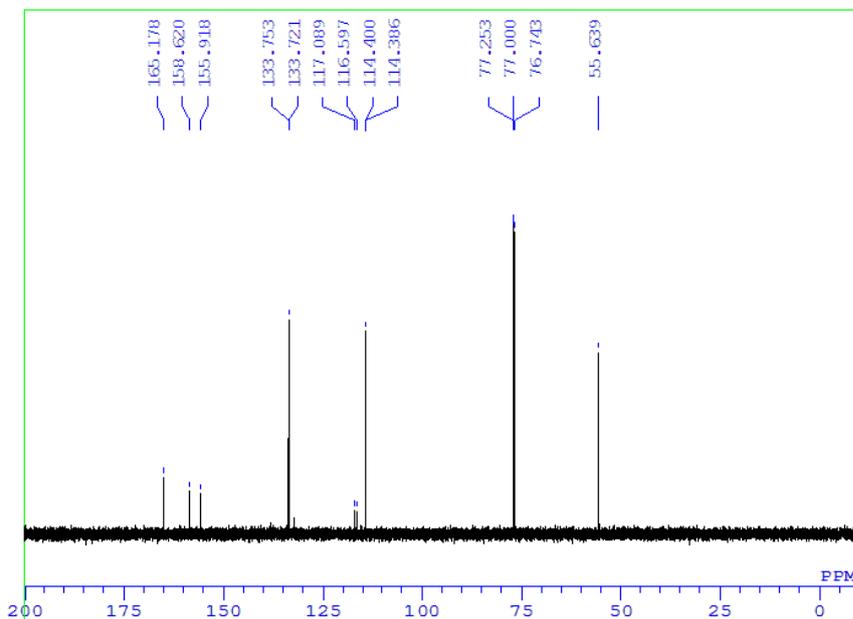
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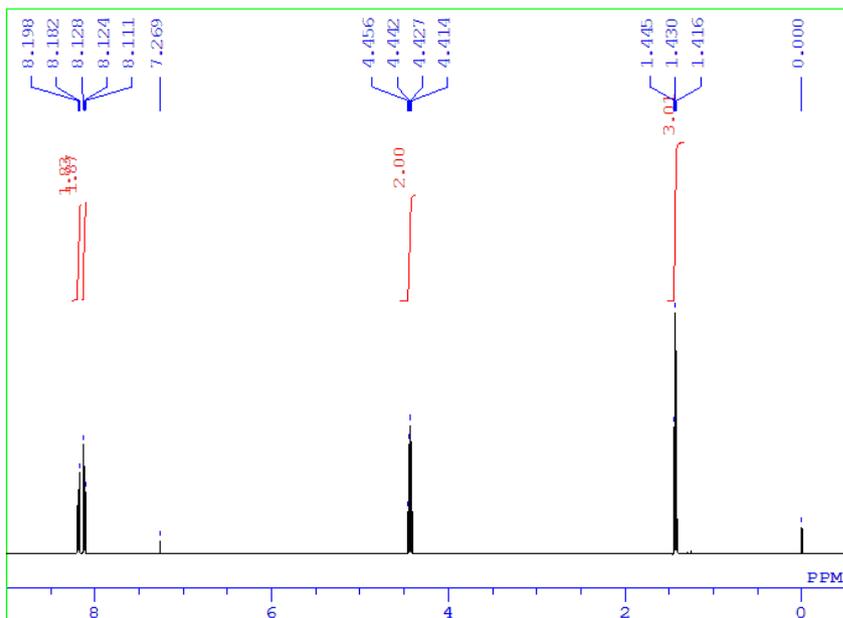
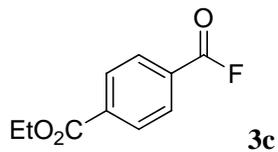
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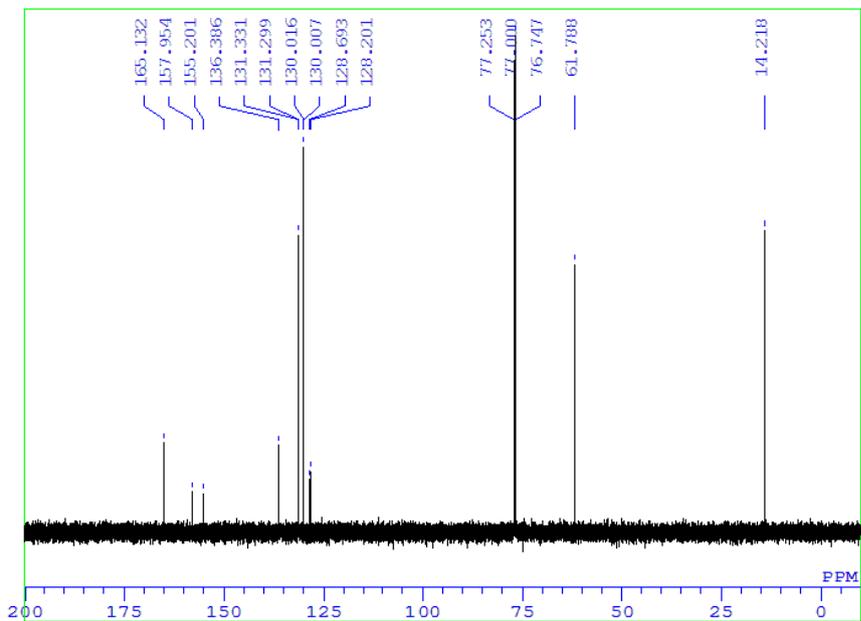


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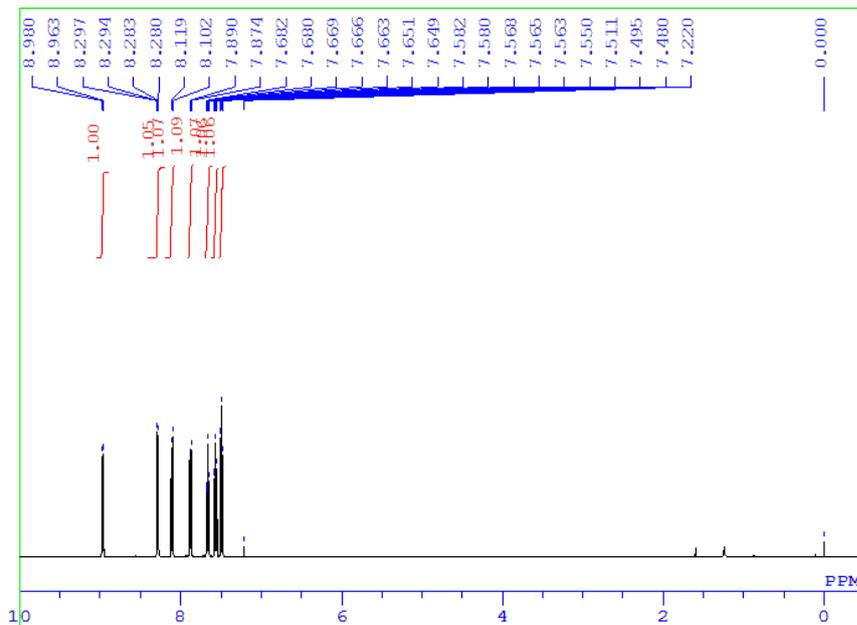
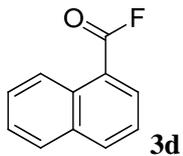
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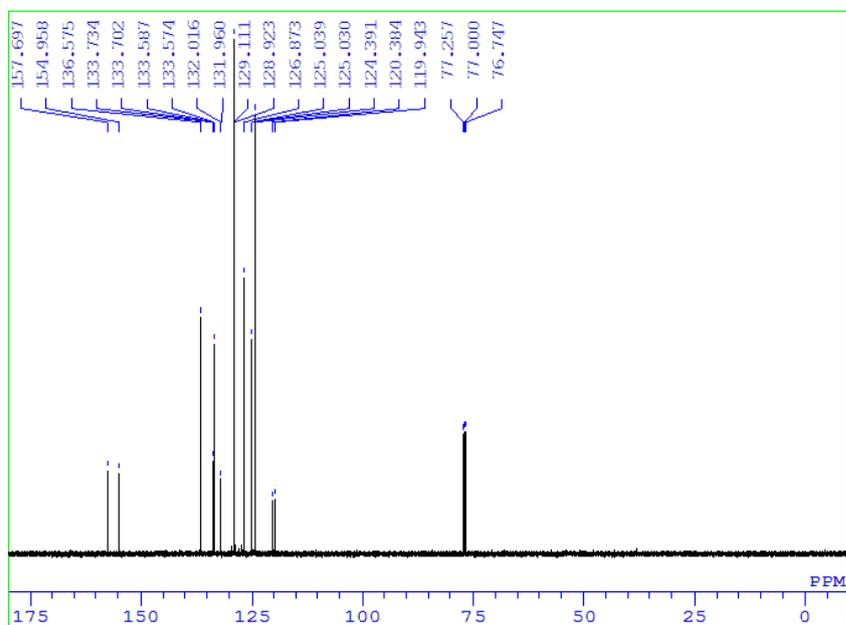
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 ACQTM 1.7302 sec
 PD 0.8000 sec
 PW1 3.28 usec
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 EXREF 77.00 ppm
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 RGAIN 56



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RGAIN 32

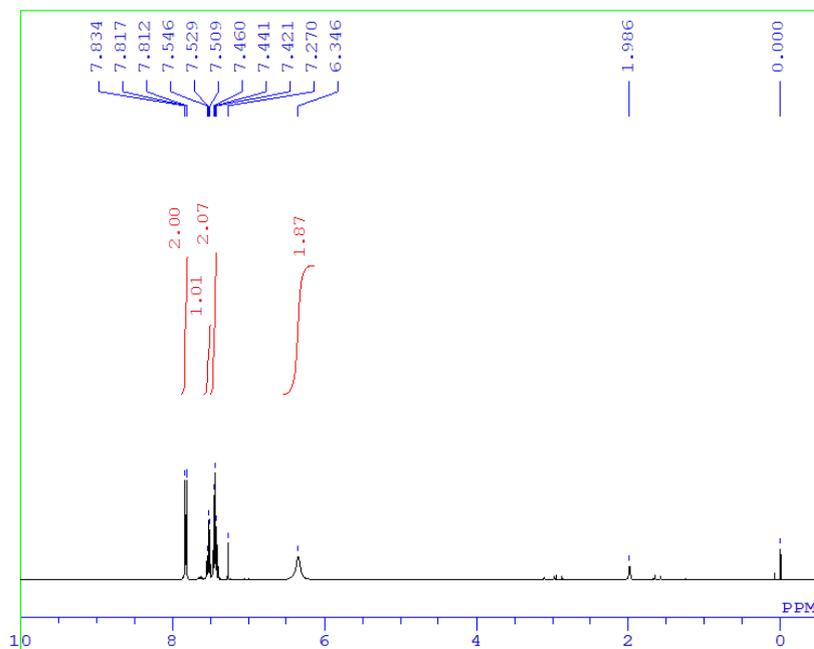
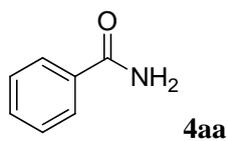
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RGAIN 58

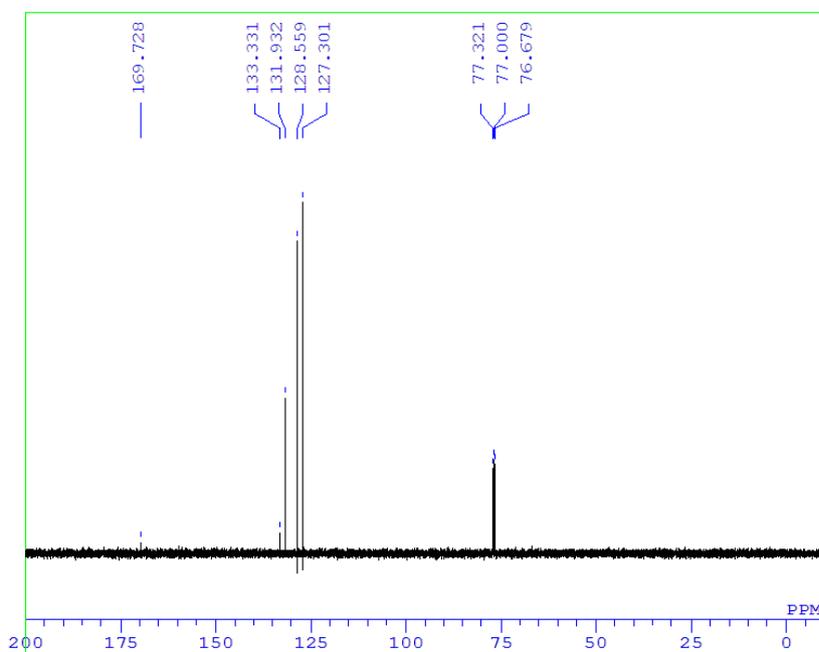
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FREQU 8000.00 Hz
SCANS 8
ACQTM 1.0240 sec
PD 5.9760 sec
PW1 6.10 usec
IRNUC 1H
CTEMP 21.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 17

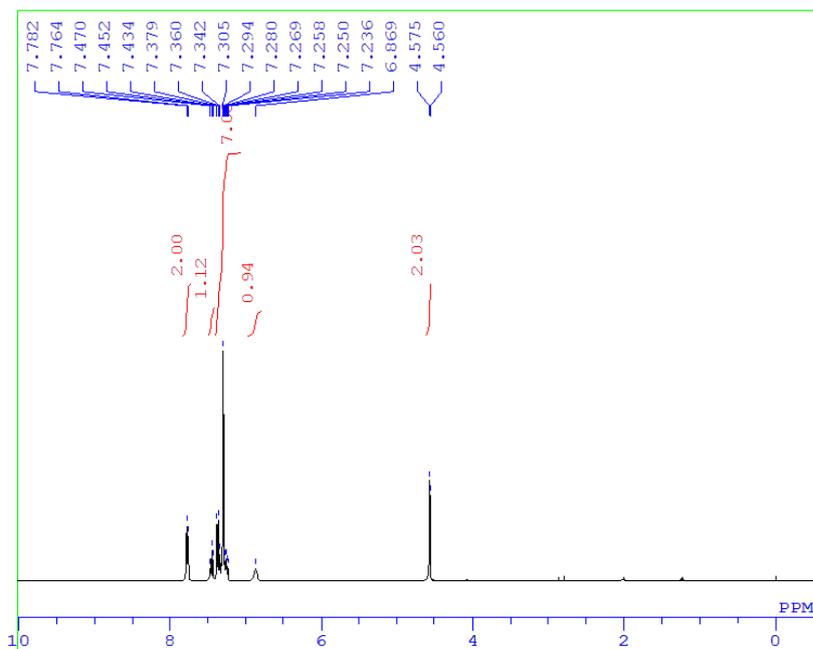
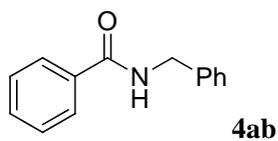
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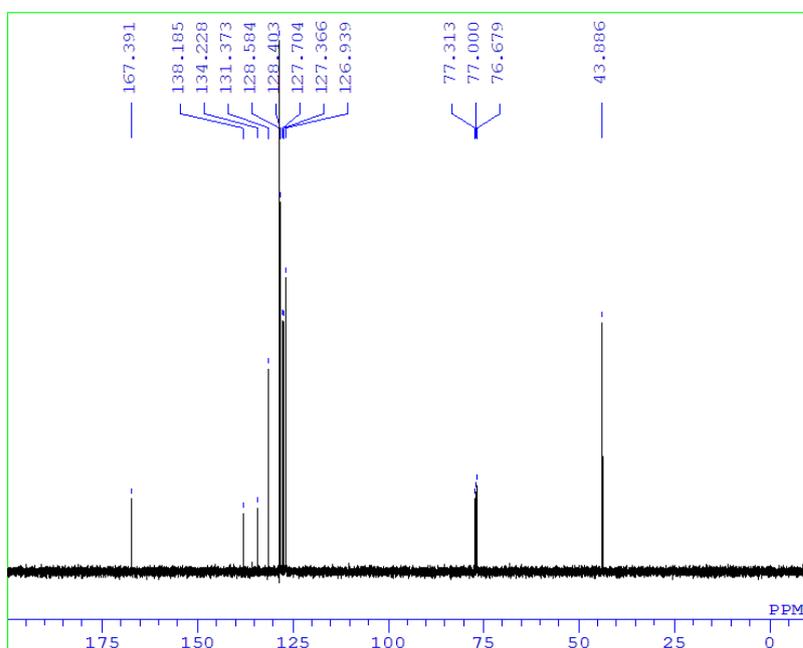
DFILE TU-398-14-13C.als
COMNT TU-398-14
DATIM Tue Jun 25 09:37:
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 64
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 21.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 28

```



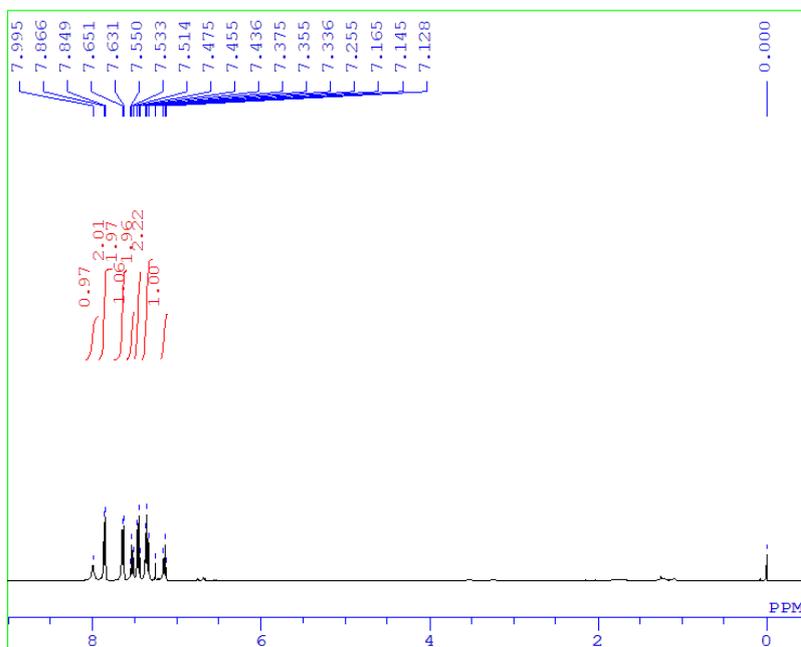
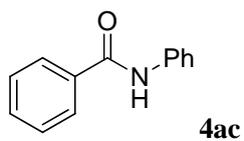
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DFILE  TU-401-1-1H.als
COMNT  TU-401-1
DATIM  Tue Jul 02 16:51:
OBNUC  1H
EXMOD  non
OBFRQ   399.65 MHz
OBSET   0.00 KHz
OBFIN   134300.00 Hz
POINT   8192
FREQU   12004.80 Hz
SCANS   8
ACQTM   0.6824 sec
PD      5.9760 sec
PW1     6.10 usec
IRNUC   1H
CTEMP   23.4 c
SLVNT   CDCL3
EXREF   0.00 ppm
BF      0.12 Hz
RGAIN   12
  
```



```

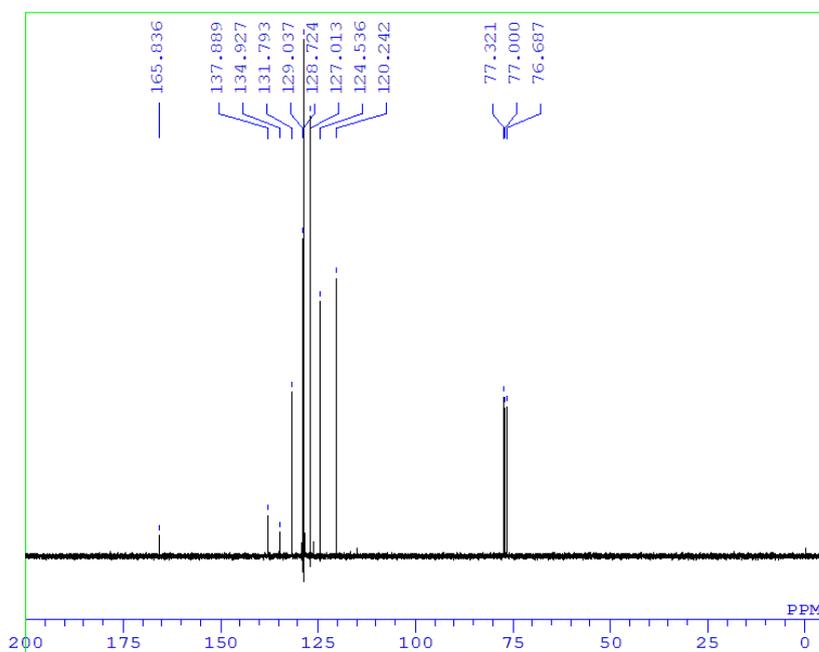
DFILE  TU-401-1-13C.als
COMNT  TU-401-1
DATIM  Tue Jul 02 16:56:
OBNUC  13C
EXMOD  bcm
OBFRQ   100.40 MHz
OBSET   0.00 KHz
OBFIN   135500.00 Hz
POINT   32768
FREQU   27100.27 Hz
SCANS   32
ACQTM   1.2091 sec
PD      1.7909 sec
PW1     5.15 usec
IRNUC   1H
CTEMP   22.7 c
SLVNT   CDCL3
EXREF   77.00 ppm
BF      0.12 Hz
RGAIN   28
  
```



```

DFILE TU-398-5-1H.als
COMNT TU-398-5
DATIM Wed Jun 19 14:12:
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 8192
FREQU 8000.00 Hz
SCANS 8
ACQTM 1.0240 sec
PD 5.9760 sec
PW1 6.10 usec
IRNUC 1H
CTEMP 21.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 16

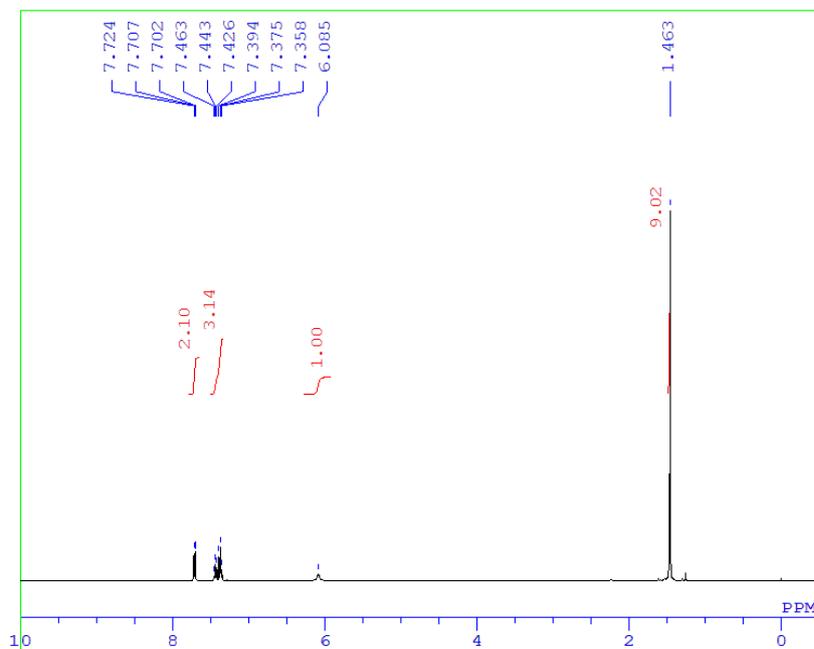
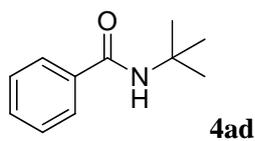
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```

DFILE TU-398-5-13C.als
COMNT TU-398-5-13C
DATIM Wed Jun 19 15:20:
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 512
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 22.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 28

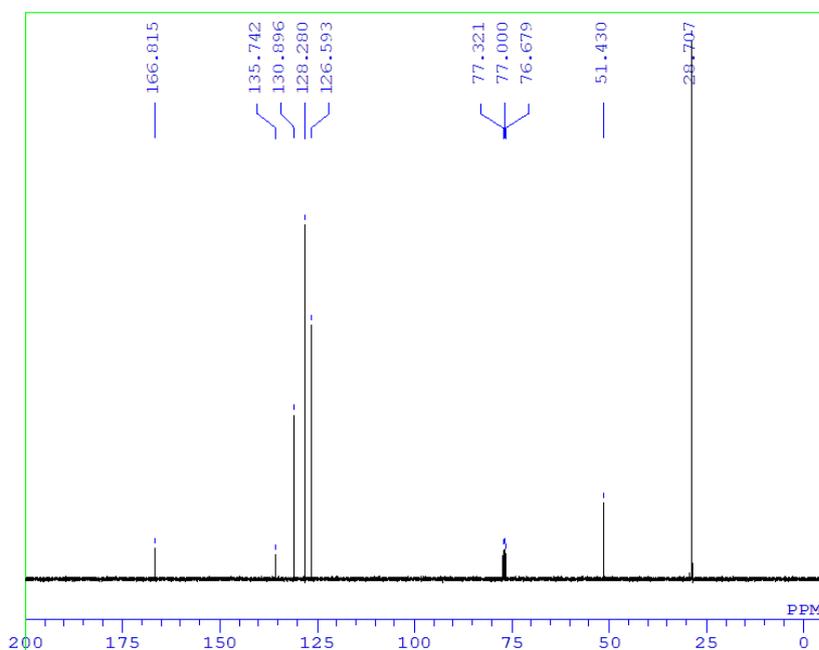
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```

DFILE TU-398-2-1H.als
COMNT TU-398-2
DATIM Tue Jun 18 18:04:
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 8192
FREQU 8000.00 Hz
SCANS 8
ACQTM 1.0240 sec
PD 5.9760 sec
PW1 6.10 usec
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 9

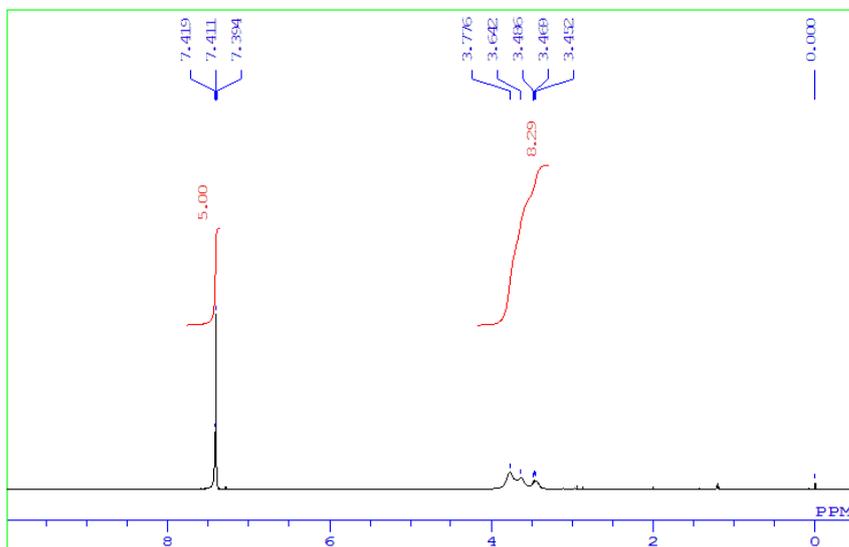
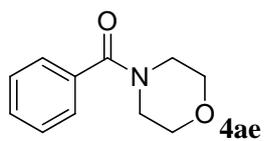
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```

DFILE TU-398-2-13C.als
COMNT TU-398-2
DATIM Tue Jun 18 18:08:
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 32
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 27

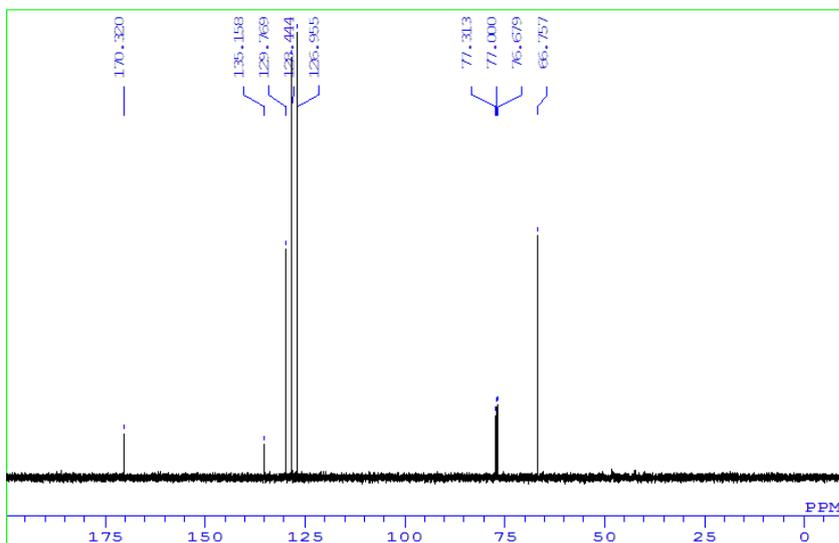
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```

DFILE TU-398-1H.als
COMNT TU-398-1
DATIM Tue Jun 18 13:39:
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 8192
FREQU 8000.00 Hz
SCANS 8
ACQTM 1.0240 sec
PD 5.9760 sec
PW1 6.10 usec
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 12

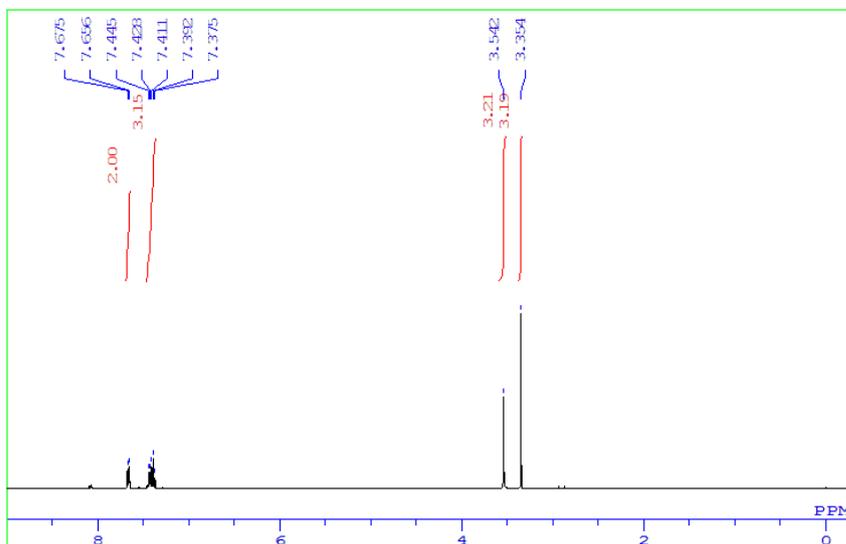
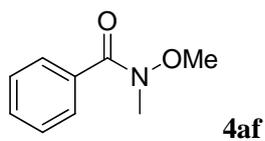
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```

DFILE TU-398-13C.als
COMNT TU-398-1
DATIM Tue Jun 18 13:45:
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 64
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 22.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 28

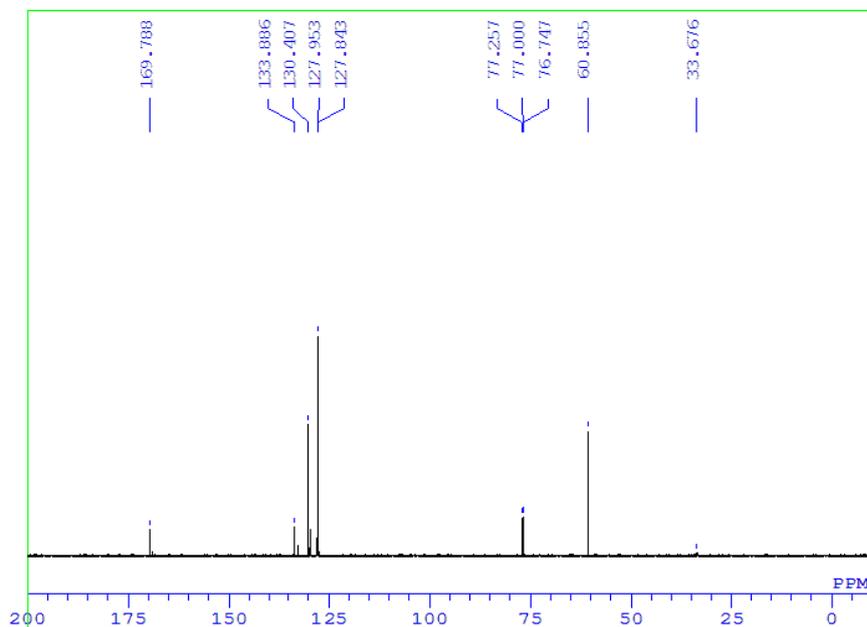
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```

DFILE TU-398-4-1H.als
COMNT TU-398-4
DATIM Tue Jun 18 19:21:
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 8192
FREQU 8000.00 Hz
SCANS 8
ACQTM 1.0240 sec
PD 5.9760 sec
PW1 6.10 usec
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 10

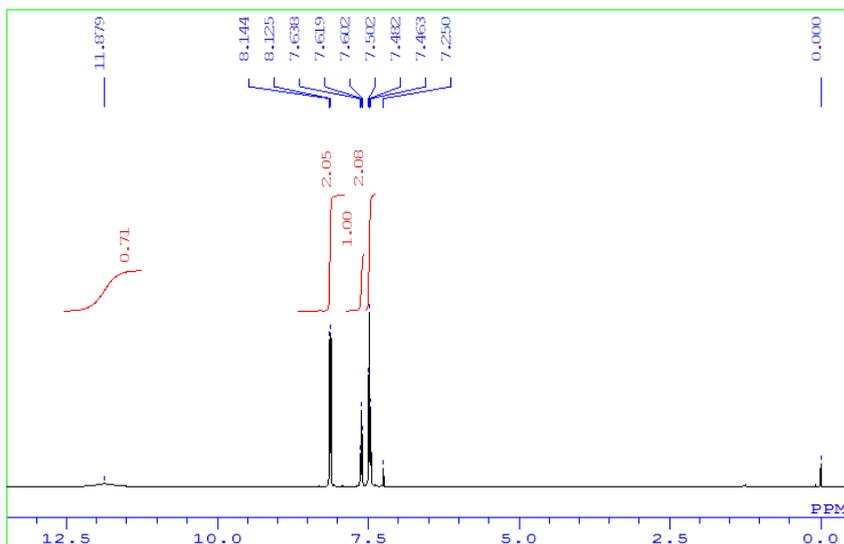
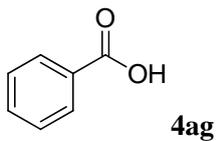
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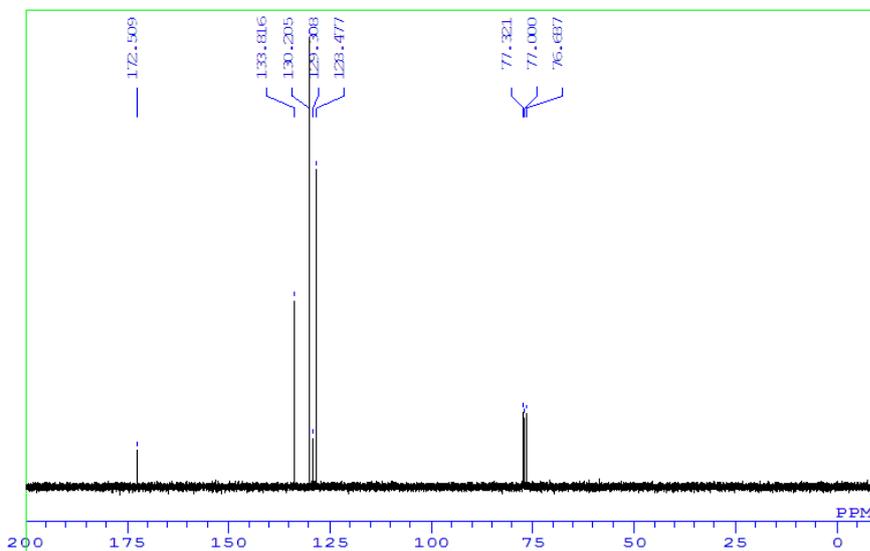
DFILE ZB00976-TU-398-4-
COMNT TU-398-4-2-13C
DATIM 2013-08-05 17:47:
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 9.13 KHz
OBFIN 1.87 Hz
POINT 65535
FREQU 37878.79 Hz
SCANS 128
ACQTM 1.7302 sec
PD 0.8000 sec
PW1 3.28 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50

```



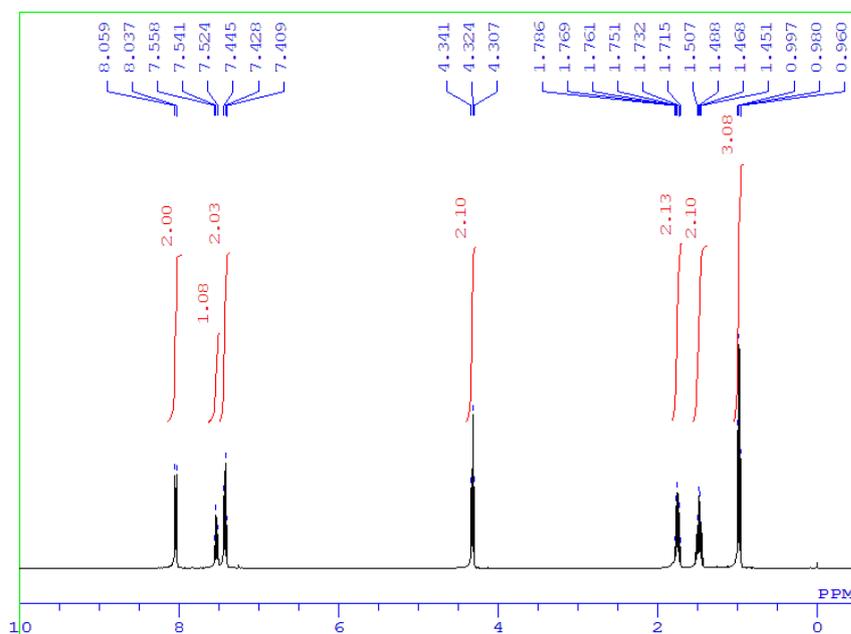
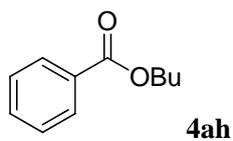
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DFILE TU-398-13-1H.als
COMNT TU-398-13
DATIM Mon Jun 24 09:18:
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 8192
FREQU 8000.00 Hz
SCANS 8
ACQTM 1.0240 sec
PD 5.9760 sec
PW1 6.10 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 16
  
```



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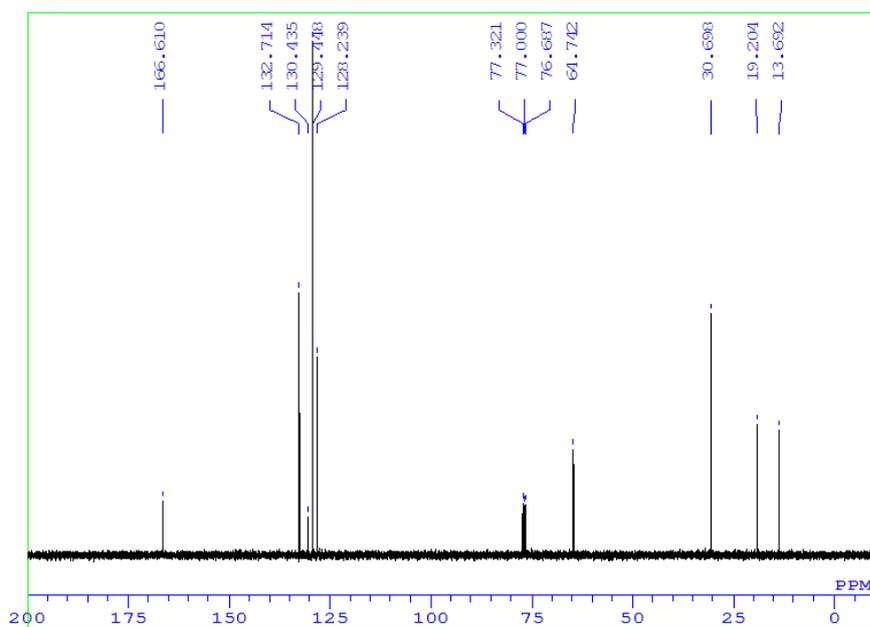
DFILE TU-398-13-13C.als
COMNT TU-398-13
DATIM Mon Jun 24 09:25:
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 64
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 22.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 28
  
```



```

DFILE TU-398-6-1H.als
COMNT TU-398-6
DATIM Wed Jun 19 14:27:
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 8192
FREQU 8000.00 Hz
SCANS 8
ACQTM 1.0240 sec
PD 5.9760 sec
PW1 6.10 usec
IRNUC 1H
CTEMP 21.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 13

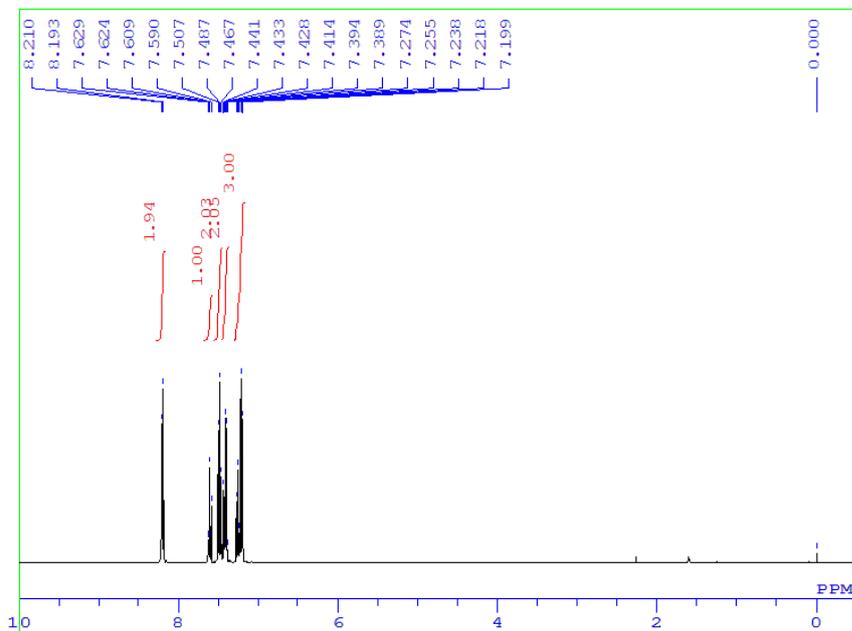
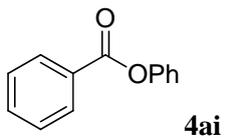
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DFILE TU-398-6-13C.als
COMNT TU-398-6
DATIM Wed Jun 19 14:31:
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 32
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 28

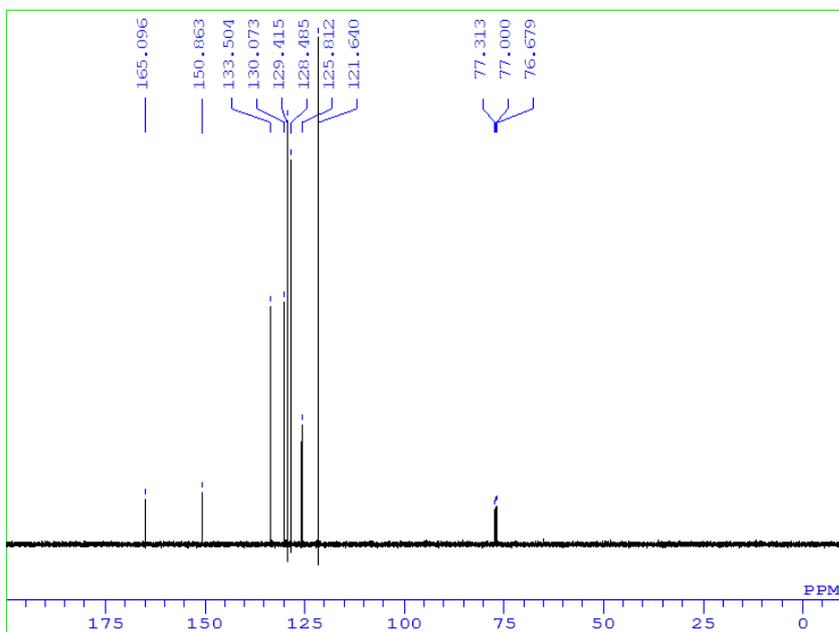
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```

DFILE TU-398-7-1H.als
COMNT TU-398-7
DATIM Wed Jun 19 16:09:
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 8192
FREQU 8000.00 Hz
SCANS 8
ACQTM 1.0240 sec
PD 5.9760 sec
PW1 6.10 usec
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 11

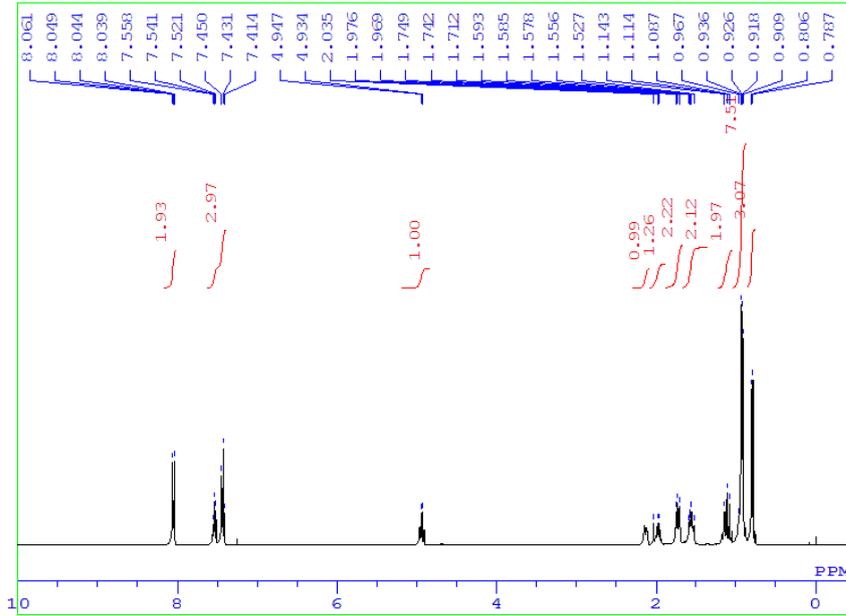
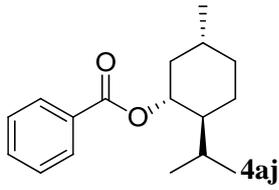
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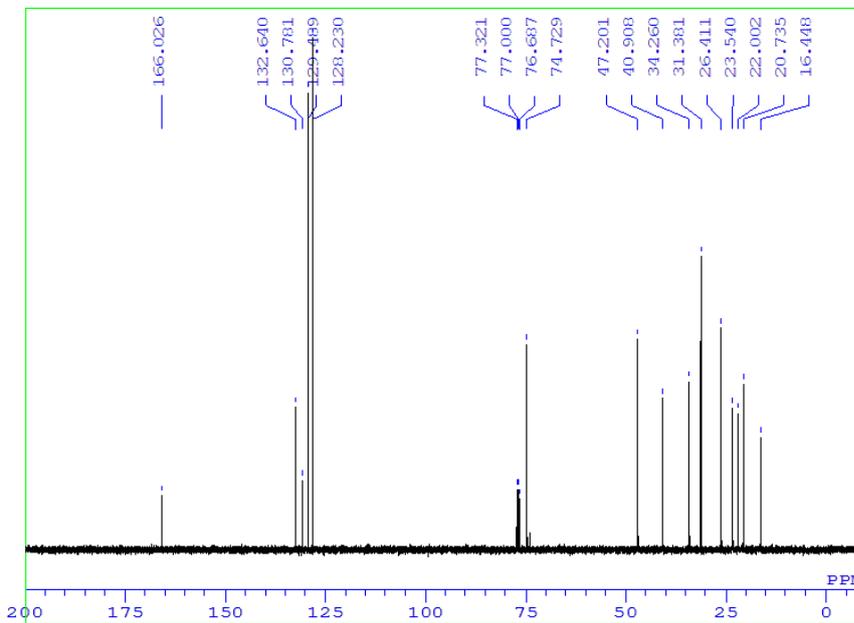
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DFILE TU-398-7-13C.als
COMNT TU-398-7
DATIM Wed Jun 19 16:13:
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 32
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 27

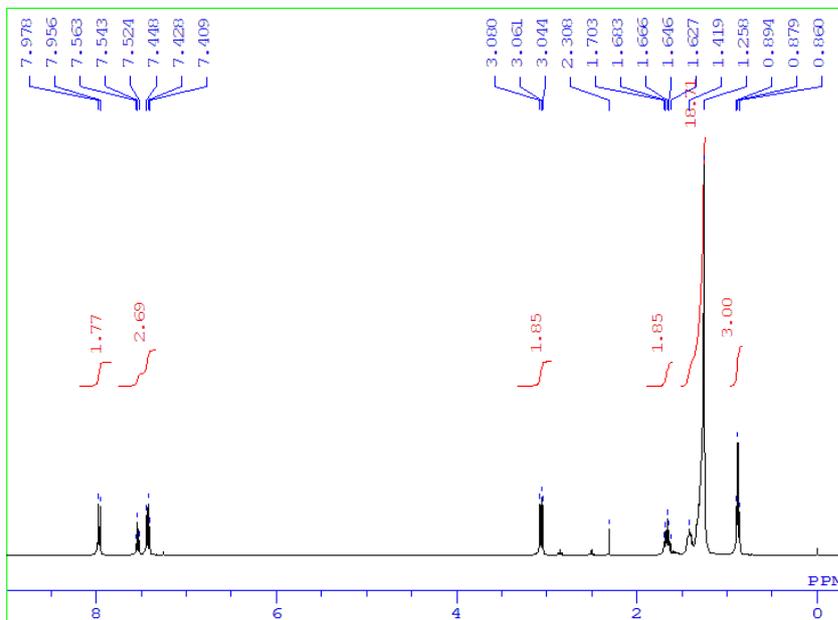
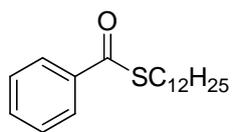
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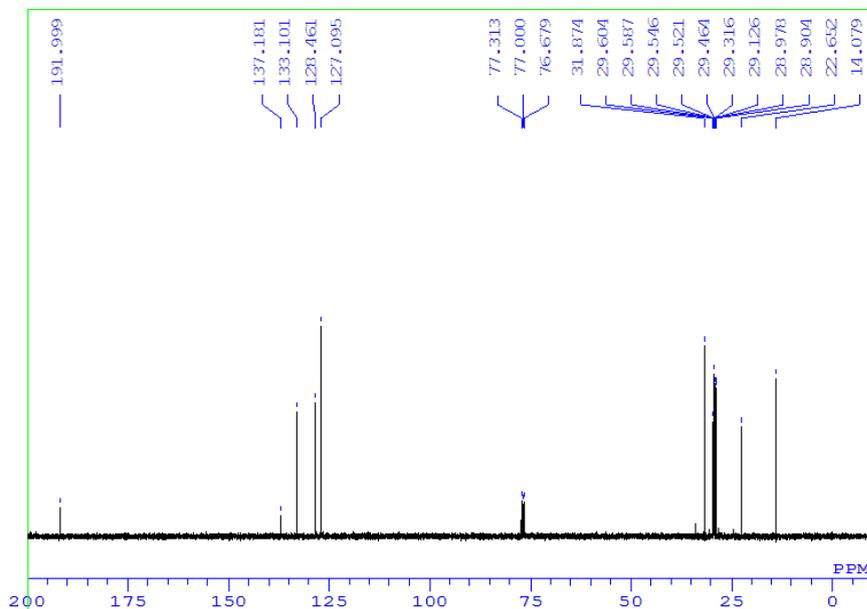
DFILE TU-398-11-1H.als
 COMNT TU-398-11
 DATIM Fri Jun 21 13:38:
 OBNUC 1H
 EXMOD non
 OBFRQ 399.65 MHz
 OBSET 0.00 KHz
 OBFIN 134300.00 Hz
 POINT 8192
 FREQU 8000.00 Hz
 SCANS 8
 ACQTM 1.0240 sec
 PD 5.9760 sec
 PW1 6.10 usec
 IRNUC 1H
 CTEMP 20.4 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 10



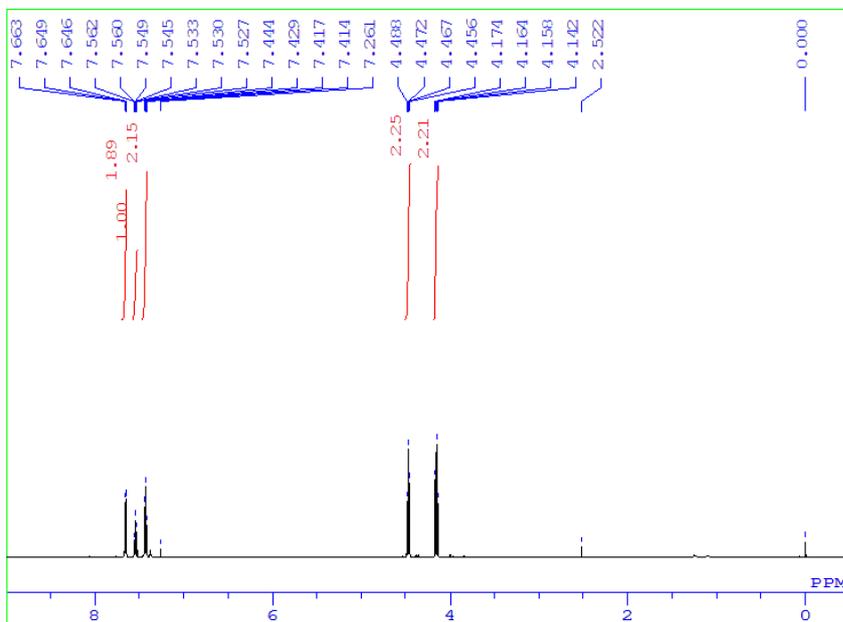
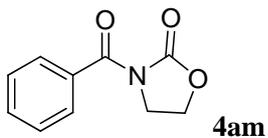
DFILE TU-398-11-13C.als
 COMNT TU-398-11
 DATIM Fri Jun 21 13:45:
 OBNUC 13C
 EXMOD bcm
 OBFRQ 100.40 MHz
 OBSET 0.00 KHz
 OBFIN 135500.00 Hz
 POINT 32768
 FREQU 27100.27 Hz
 SCANS 64
 ACQTM 1.2091 sec
 PD 1.7909 sec
 PW1 5.15 usec
 IRNUC 1H
 CTEMP 21.5 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.00 Hz
 RGAIN 28



DFILE TU-398-9-1H.als
 COMNT TU-398-9
 DATIM Thu Jun 20 18:48:
 OBNUC 1H
 EXMOD non
 OBFRQ 399.65 MHz
 OBSET 0.00 KHz
 OBFIN 134300.00 Hz
 POINT 8192
 FREQU 8000.00 Hz
 SCANS 8
 ACQTM 1.0240 sec
 PD 5.9760 sec
 PW1 6.10 usec
 IRNUC 1H
 CTEMP 20.6 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 8

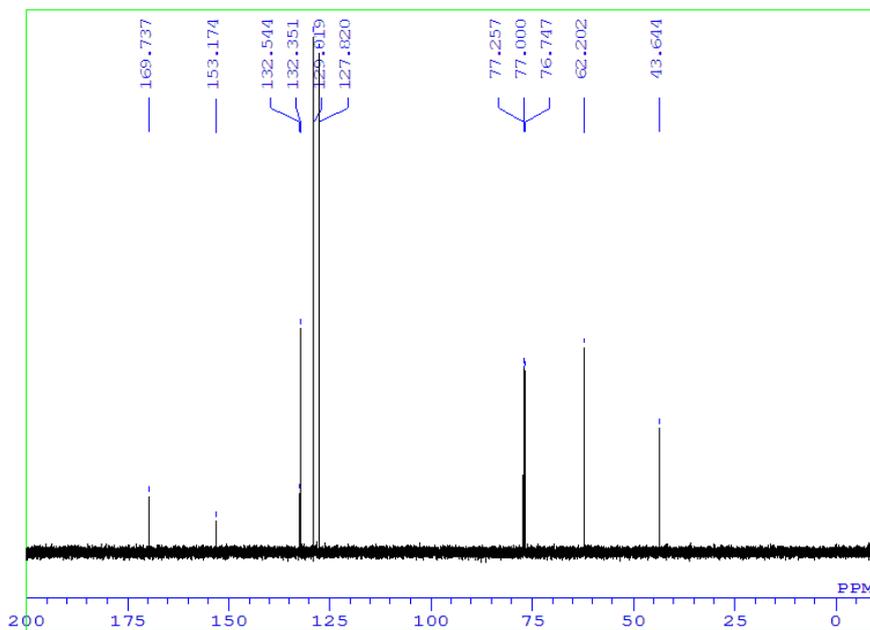


DFILE TU-398-9-13C.als
 COMNT TU-398-9
 DATIM Thu Jun 20 18:56:
 OBNUC 13C
 EXMOD bcm
 OBFRQ 100.40 MHz
 OBSET 0.00 KHz
 OBFIN 135500.00 Hz
 POINT 32768
 FREQU 27100.27 Hz
 SCANS 32
 ACQTM 1.2091 sec
 PD 1.7909 sec
 PW1 5.15 usec
 IRNUC 1H
 CTEMP 21.3 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.00 Hz
 RGAIN 27



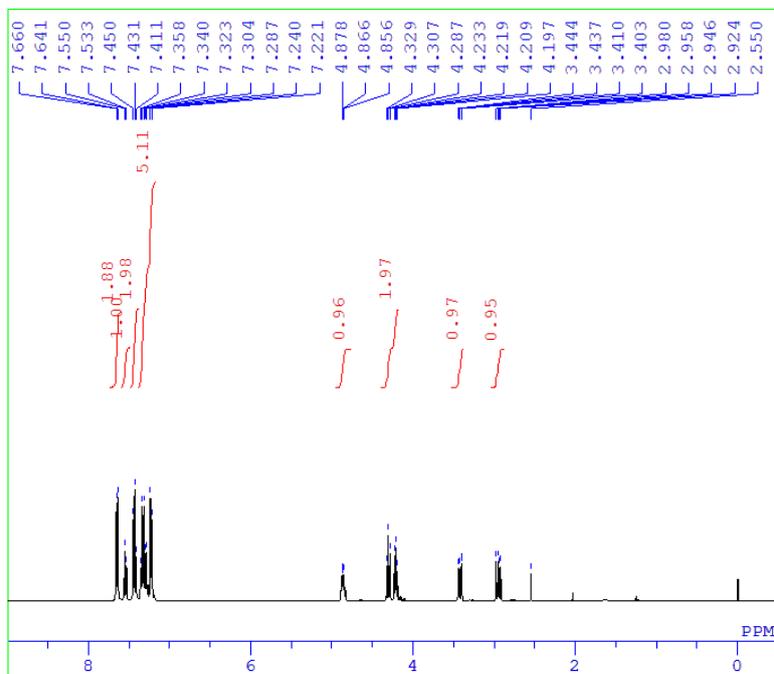
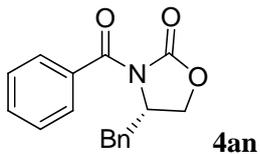
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DFILE ZB00976-TU-403-1_
COMNT TU-403-1
DATIM 2013-07-16 11:19:
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 8127.44 Hz
SCANS 16
ACQTM 2.0159 sec
PD 0.9000 sec
PW1 6.25 usec
IRNUC 1H
CTEMP 21.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 38
  
```

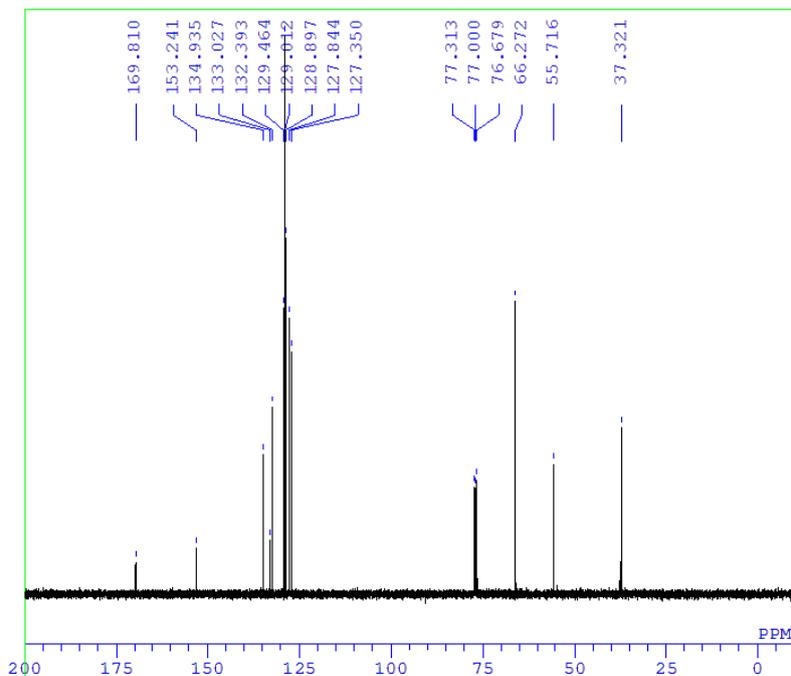


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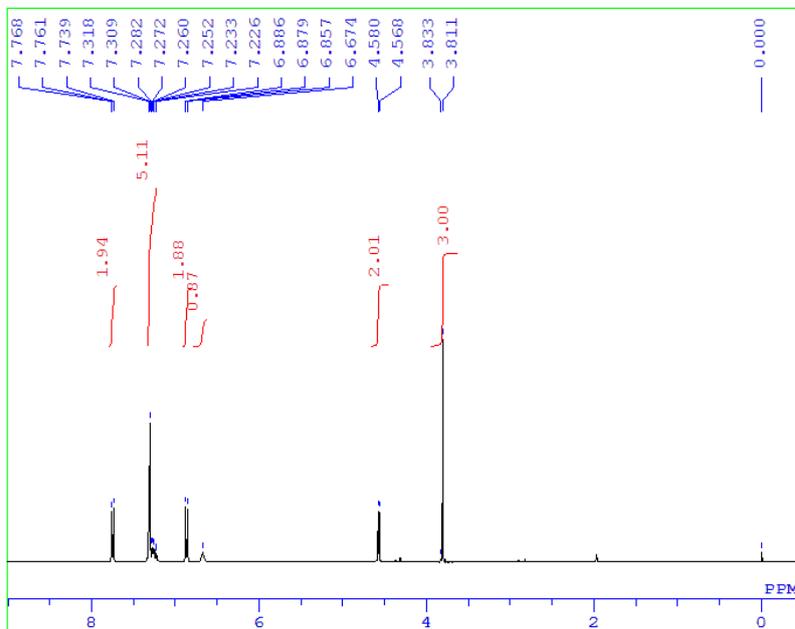
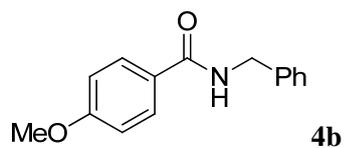
DFILE ZB00976-TU-403-1_
COMNT TU-403-1
DATIM 2013-07-16 11:22:
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 9.13 KHz
OBFIN 1.87 Hz
POINT 65535
FREQU 37878.79 Hz
SCANS 128
ACQTM 1.7302 sec
PD 0.8000 sec
PW1 3.28 usec
IRNUC 1H
CTEMP 21.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 50
  
```



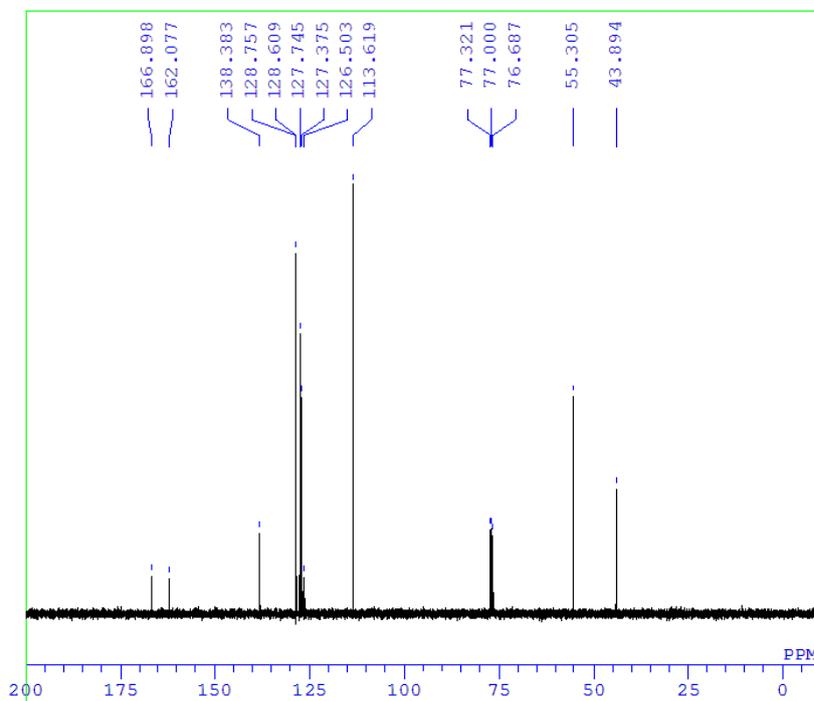
DFILE TU-398-12-1H.als
 COMNT TU-398-12
 DATIM Fri Jun 21 14:35:
 OBNUC 1H
 EXMOD non
 OBFRQ 399.65 MHz
 OBSET 0.00 KHz
 OBFIN 134300.00 Hz
 POINT 8192
 FREQU 8000.00 Hz
 SCANS 8
 ACQTM 1.0240 sec
 PD 5.9760 sec
 PW1 6.10 usec
 IRNUC 1H
 CTEMP 20.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 15



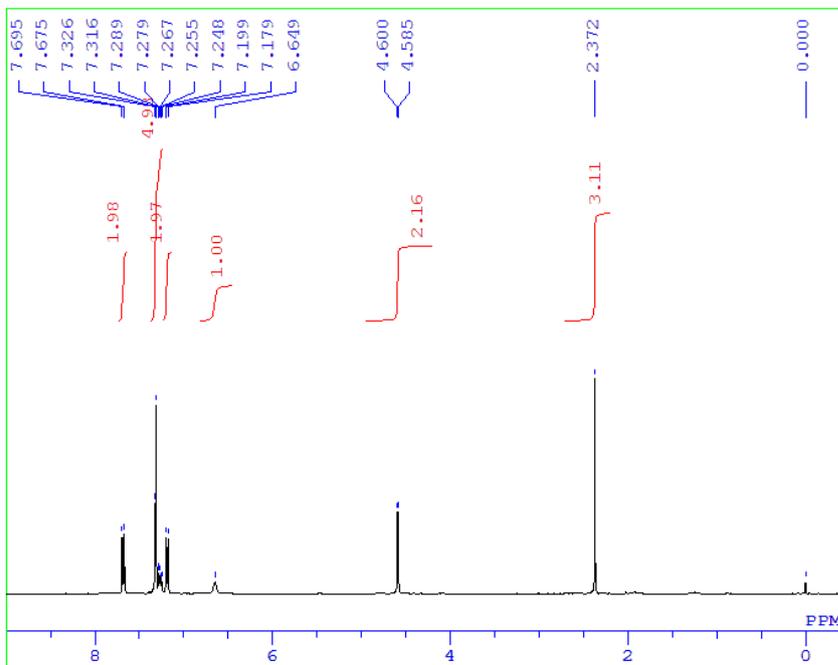
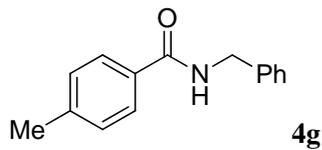
DFILE TU-398-12-13C.als
 COMNT TU-398-12
 DATIM Fri Jun 21 14:44:
 OBNUC 13C
 EXMOD bcm
 OBFRQ 100.40 MHz
 OBSET 0.00 KHz
 OBFIN 135500.00 Hz
 POINT 32768
 FREQU 27100.27 Hz
 SCANS 128
 ACQTM 1.2091 sec
 PD 1.7909 sec
 PW1 5.15 usec
 IRNUC 1H
 CTEMP 21.9 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.00 Hz
 RGAIN 28



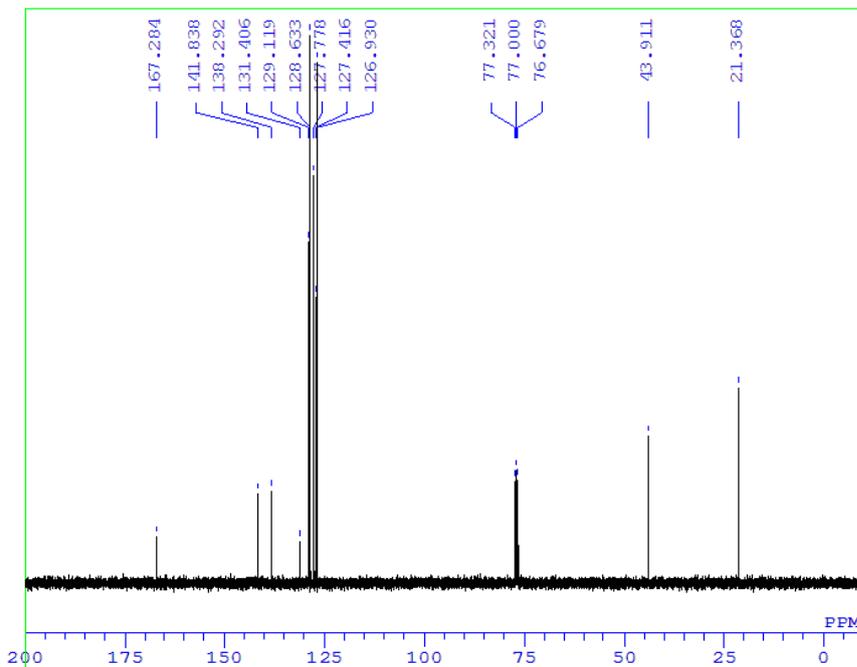
DFILE TU-399-2-1H.als
 COMNT TU-399-2
 DATIM Wed Jun 26 09:00:
 OBNUC 1H
 EXMOD non
 OBFRQ 399.65 MHz
 OBSET 0.00 KHz
 OBFIN 134300.00 Hz
 POINT 8192
 FREQU 8000.00 Hz
 SCANS 8
 ACQTM 1.0240 sec
 PD 5.9760 sec
 PW1 6.10 usec
 IRNUC 1H
 CTEMP 21.0 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 14



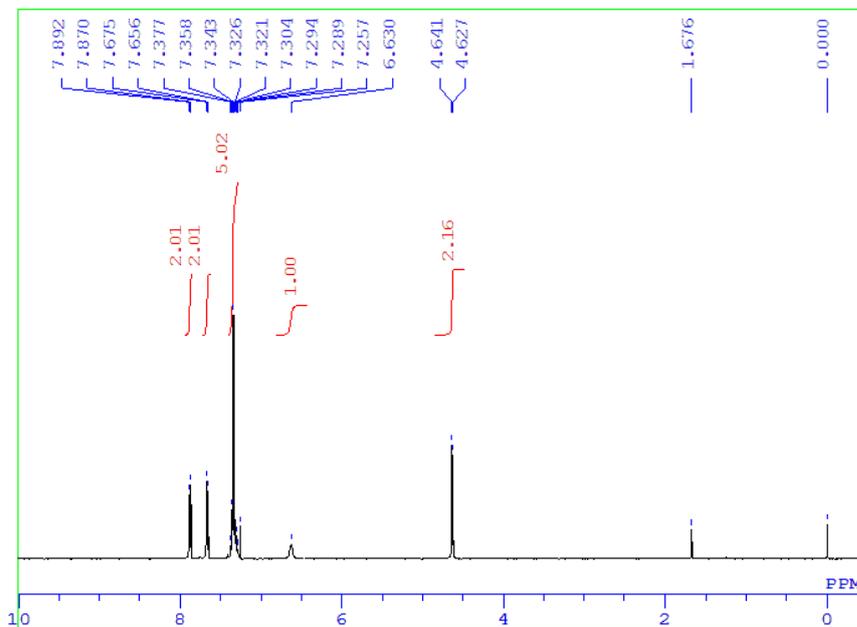
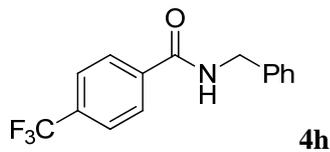
DFILE TU-399-2-13C.als
 COMNT TU-399-2
 DATIM Wed Jun 26 09:06:
 OBNUC 13C
 EXMOD bcm
 OBFRQ 100.40 MHz
 OBSET 0.00 KHz
 OBFIN 135500.00 Hz
 POINT 32768
 FREQU 27100.27 Hz
 SCANS 64
 ACQTM 1.2091 sec
 PD 1.7909 sec
 PW1 5.15 usec
 IRNUC 1H
 CTEMP 21.8 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.00 Hz
 RGAIN 28



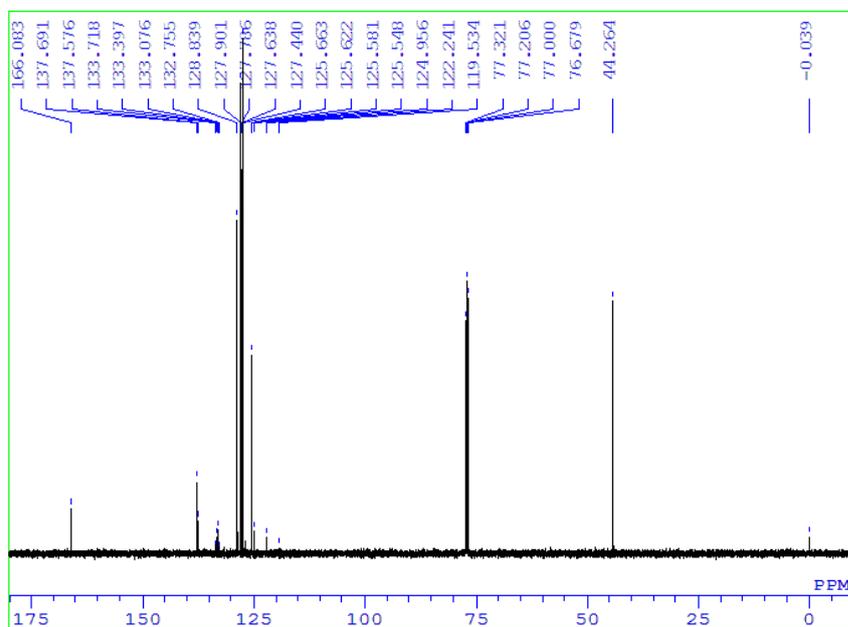
DFILE TU-399-1-1H.als
 COMNT TU-399-1
 DATIM Tue Jun 25 18:27:
 OBNUC 1H
 EXMOD non
 OBFRQ 399.65 MHz
 OBSET 0.00 KHz
 OBFIN 134300.00 Hz
 POINT 8192
 FREQU 8000.00 Hz
 SCANS 8
 ACQTM 1.0240 sec
 PD 5.9760 sec
 PW1 6.10 usec
 IRNUC 1H
 CTEMP 20.9 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 13



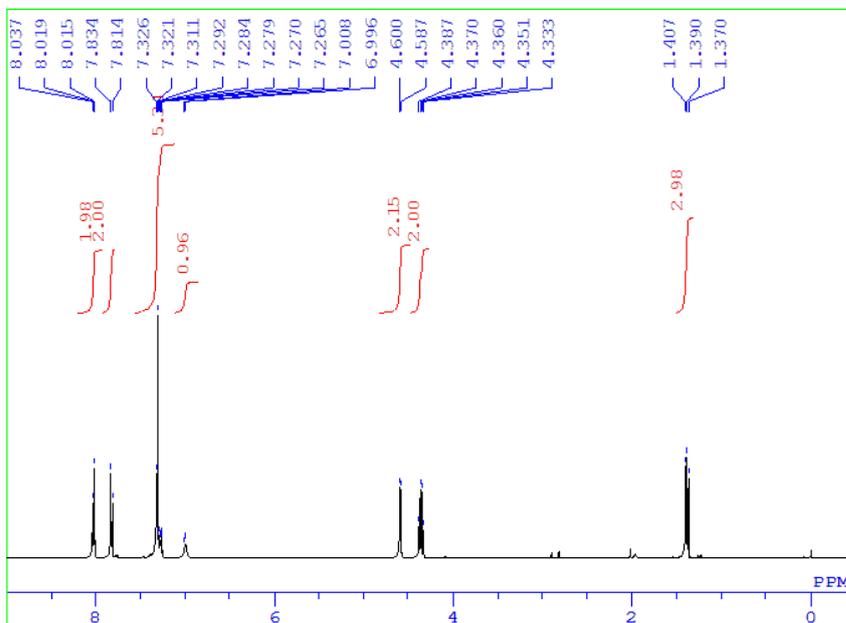
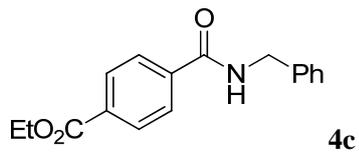
DFILE TU-399-1-13C.als
 COMNT TU-399-1
 DATIM Tue Jun 25 18:33:
 OBNUC 13C
 EXMOD bcm
 OBFRQ 100.40 MHz
 OBSET 0.00 KHz
 OBFIN 135500.00 Hz
 POINT 32768
 FREQU 27100.27 Hz
 SCANS 64
 ACQTM 1.2091 sec
 PD 1.7909 sec
 PW1 5.15 usec
 IRNUC 1H
 CTEMP 22.0 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.00 Hz
 RGAIN 28



DFILE TU-399-5-1H.als
 COMNT TU-399-5
 DATIM Tue Jun 25 18:59:
 OBNUC 1H
 EXMOD non
 OBFRQ 399.65 MHz
 OBSET 0.00 KHz
 OBFIN 134300.00 Hz
 POINT 8192
 FREQU 8000.00 Hz
 SCANS 8
 ACQTM 1.0240 sec
 PD 5.9760 sec
 PW1 6.10 usec
 IRNUC 1H
 CTEMP 21.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.00 Hz
 RGAIN 17

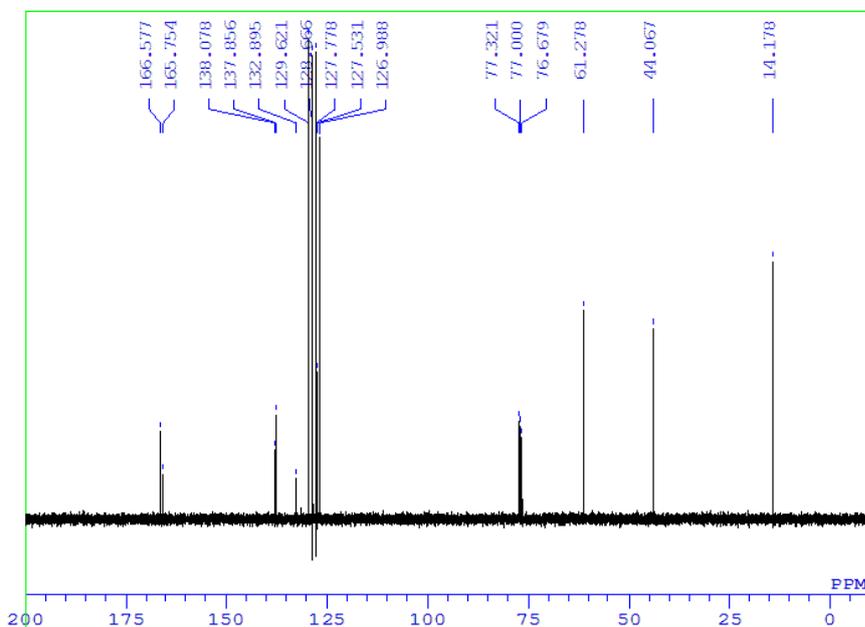


DFILE TU-399-5-13C.als
 COMNT TU-399-5
 DATIM Tue Jun 25 19:58:
 OBNUC 13C
 EXMOD bcm
 OBFRQ 100.40 MHz
 OBSET 0.00 KHz
 OBFIN 135500.00 Hz
 POINT 32768
 FREQU 27100.27 Hz
 SCANS 1028
 ACQTM 1.2091 sec
 PD 1.7909 sec
 PW1 5.15 usec
 IRNUC 1H
 CTEMP 22.7 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.00 Hz
 RGAIN 28



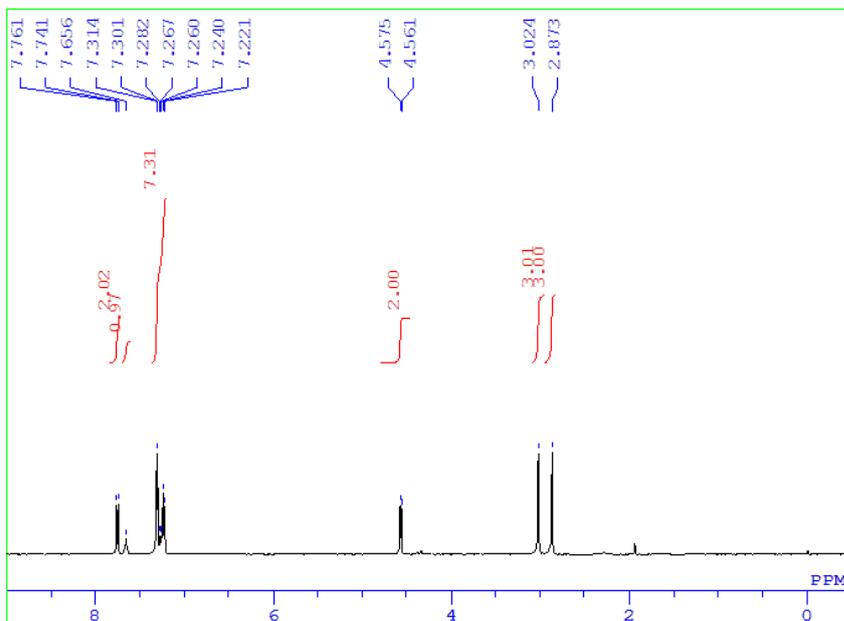
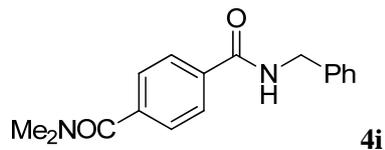
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DFILE TU-399-3-1H.als
COMNT TU-399-3
DATIM Wed Jun 26 09:14:
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 8192
FREQU 8000.00 Hz
SCANS 8
ACQTM 1.0240 sec
PD 5.9760 sec
PW1 6.10 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 14
  
```



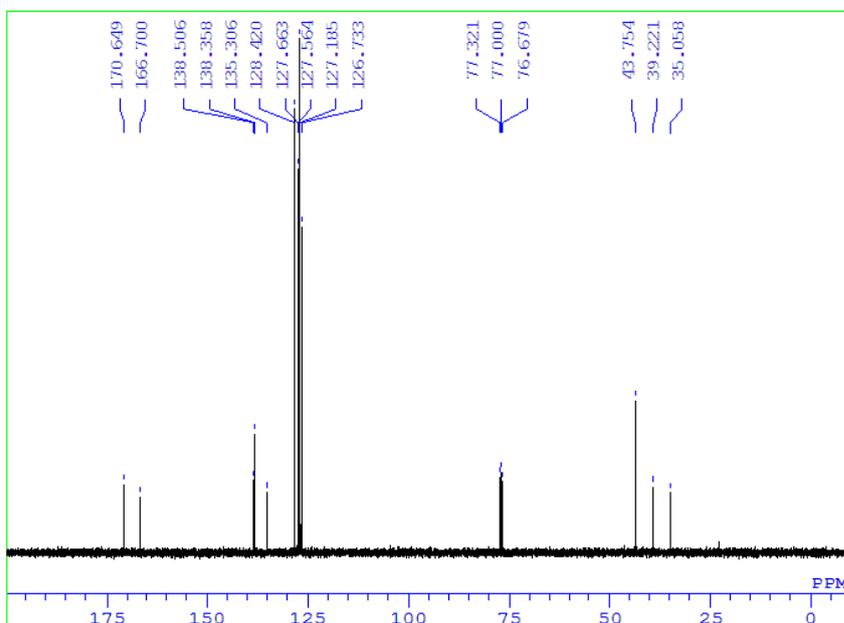
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DFILE TU-399-3-13C.als
COMNT TU-399-3
DATIM Wed Jun 26 09:19:
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 64
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 22.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 28
  
```



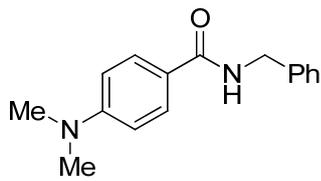
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DFILE TU-399-11-1H.als
COMNT TU-399-11
DATIM Thu Jun 27 14:33:
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 8192
FREQU 8000.00 Hz
SCANS 8
ACQTM 1.0240 sec
PD 5.9760 sec
PW1 6.10 usec
IRNUC 1H
CTEMP 20.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 12
  
```

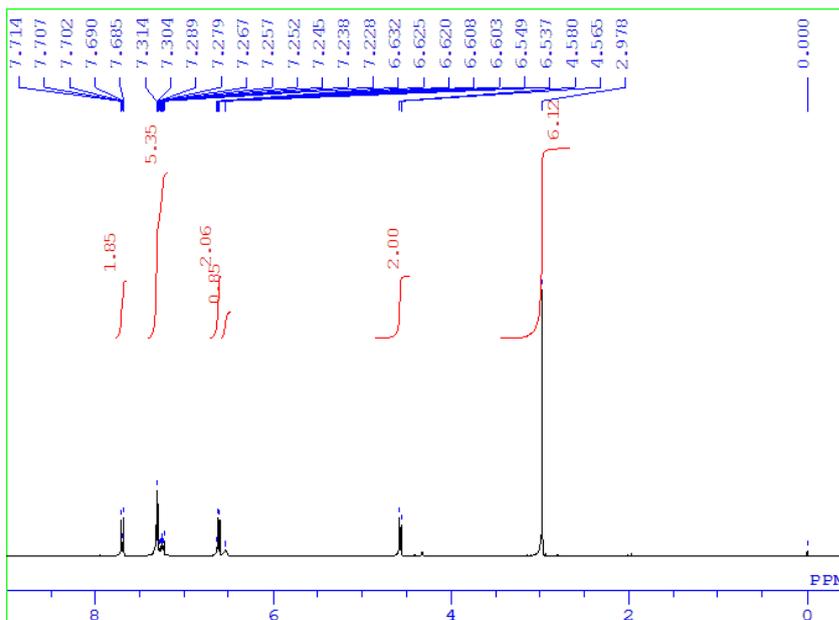


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DFILE TU-399-11-13C.als
COMNT TU-399-11
DATIM Thu Jun 27 14:41:
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 64
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 21.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 28
  
```

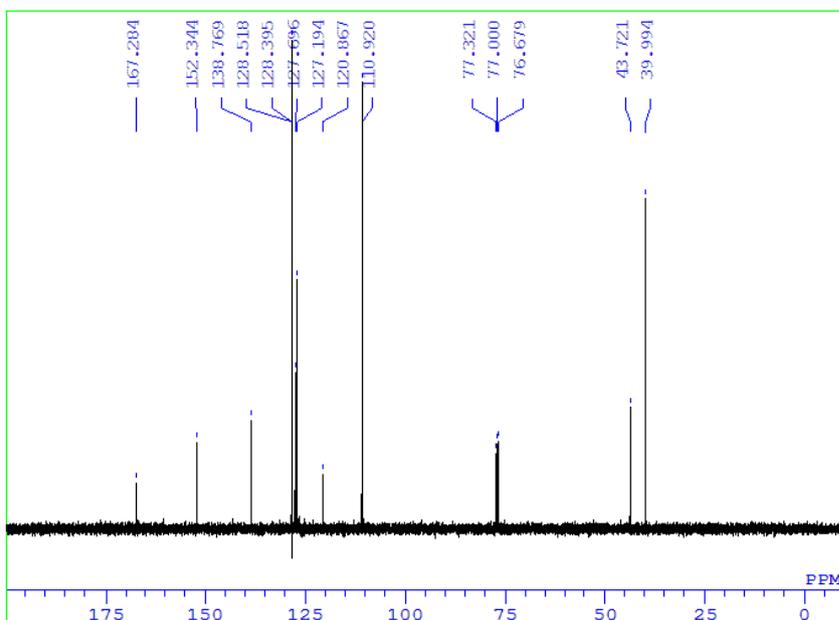


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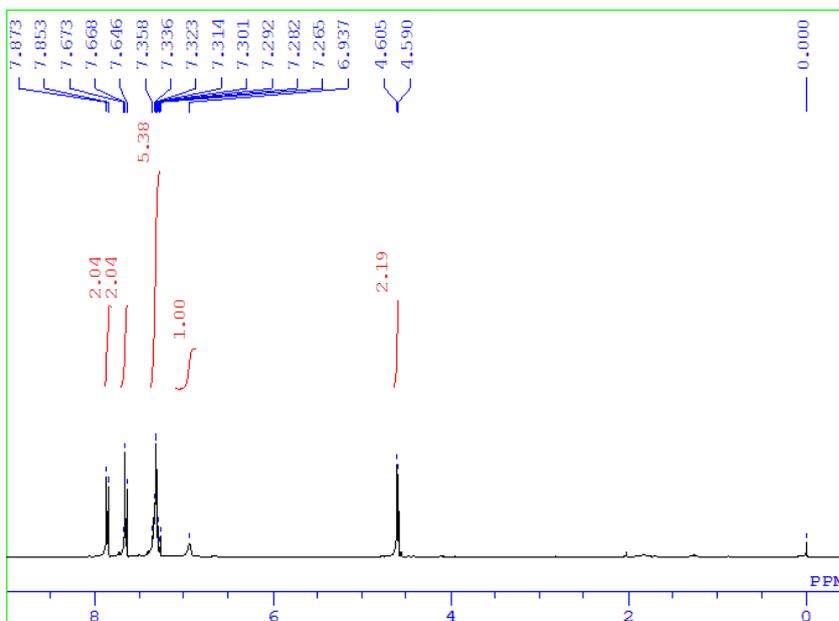
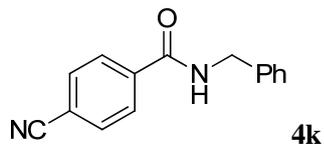
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DFILE TU-399-10-1H.als
COMNT TU-399-10
DATIM Wed Jun 26 17:59:
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 8192
FREQU 8000.00 Hz
SCANS 8
ACQTM 1.0240 sec
PD 5.9760 sec
PW1 6.10 usec
IRNUC 1H
CTEMP 21.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 13
  
```



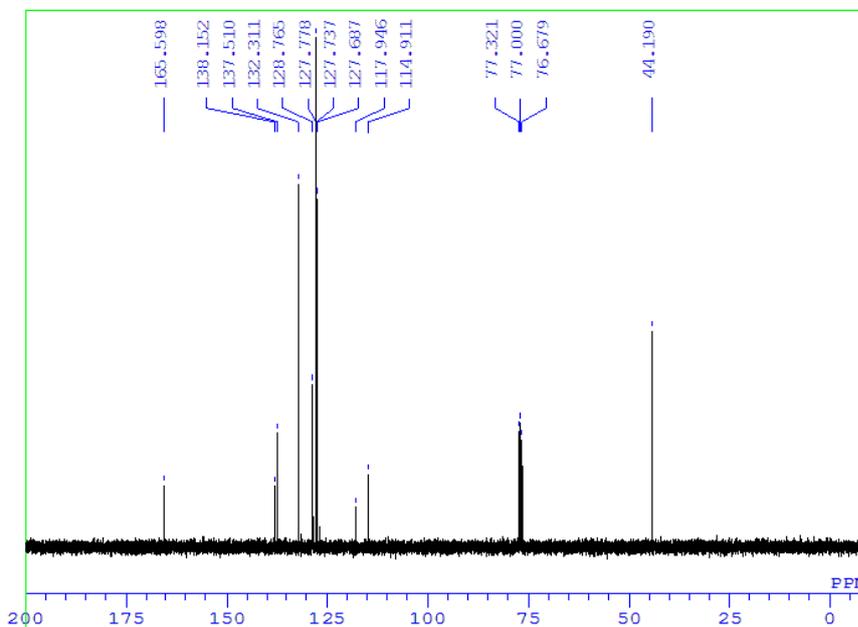
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DFILE TU-399-10-13C.als
COMNT TU-399-10
DATIM Wed Jun 26 18:06:
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 64
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 28
  
```



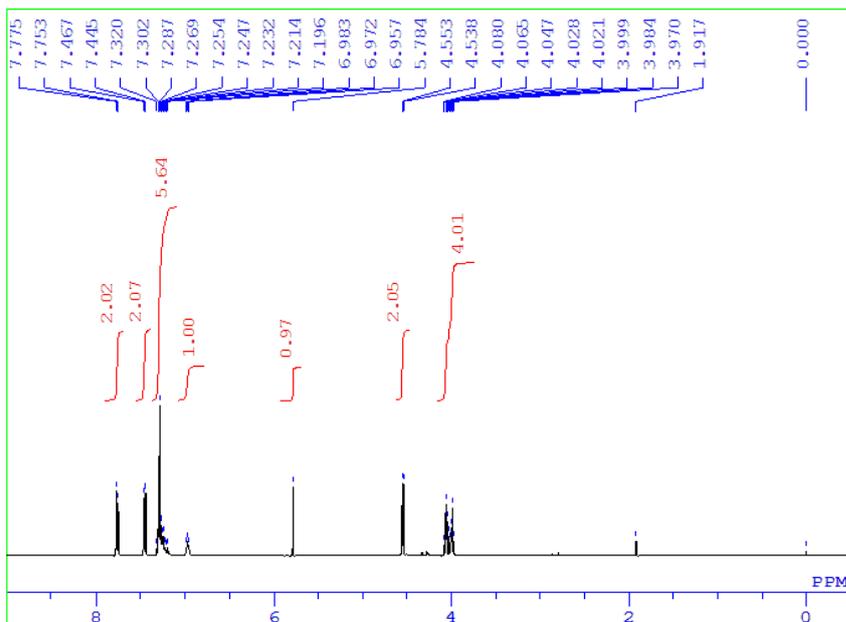
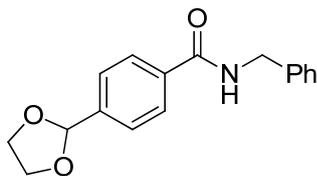
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DFILE TU-399-4-1H.als
COMNT TU-399-4
DATIM Wed Jun 26 18:21:
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 8192
FREQU 8000.00 Hz
SCANS 8
ACQTM 1.0240 sec
PD 5.9760 sec
PW1 6.10 usec
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 15
  
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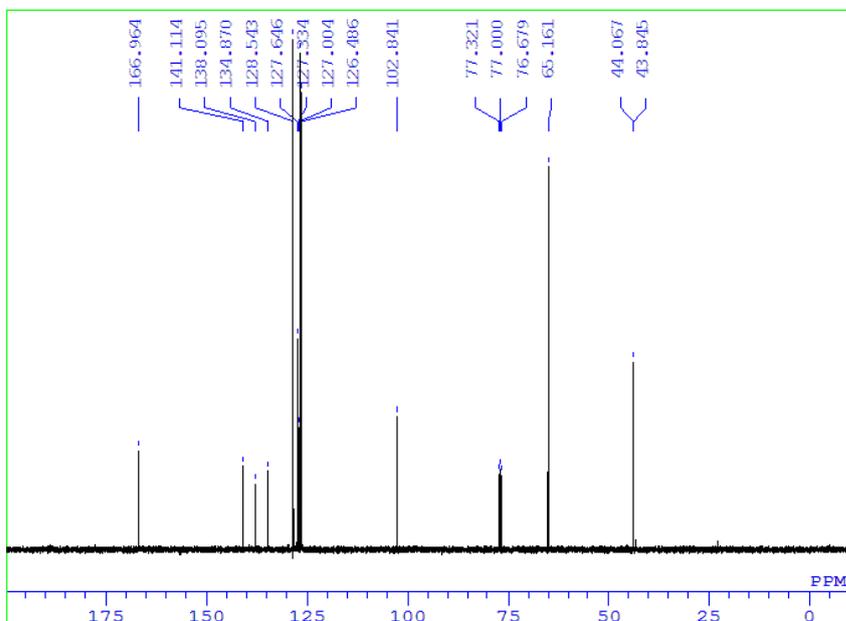
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DFILE TU-399-4-13C.als
COMNT TU-399-4
DATIM Wed Jun 26 18:28:
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 64
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 28
  
```



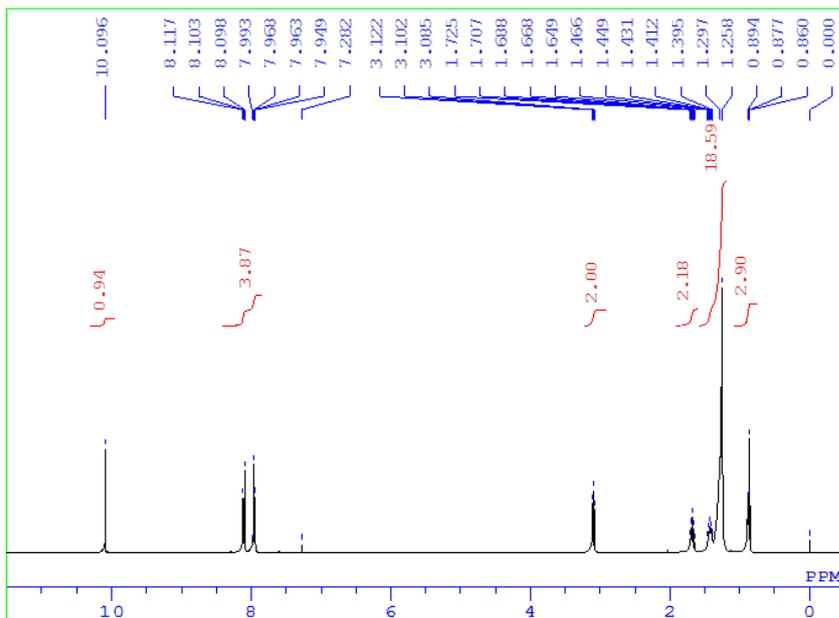
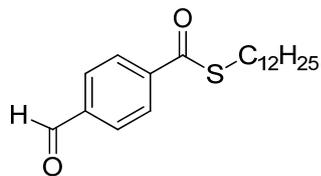
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DFILE TU-400-5-1H.als
COMNT TU-400-5
DATIM Mon Jul 01 09:21:
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 8192
FREQU 12004.80 Hz
SCANS 8
ACQTM 0.6824 sec
PD 5.9760 sec
PW1 6.10 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 11
  
```

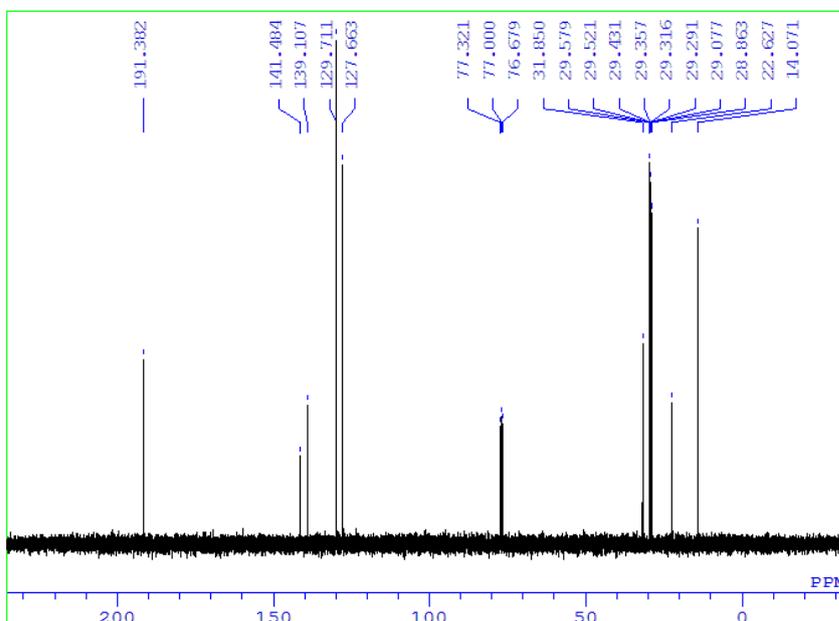


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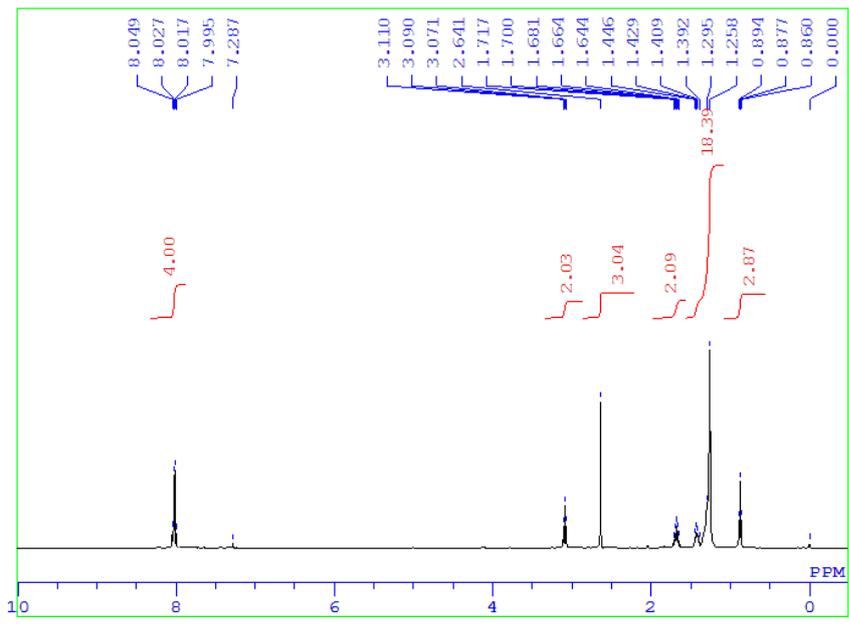
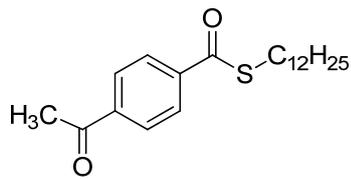
DFILE TU-400-5-13C.als
COMNT TU-400-5
DATIM Mon Jul 01 09:28:
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 64
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 22.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 28
  
```



DFILE TU-400-4-1H.als
 COMNT TU-400-4
 DATIM Mon Jul 01 14:55:
 OBNUC 1H
 EXMOD non
 OBFRQ 399.65 MHz
 OBSET 0.00 KHz
 OBFIN 134300.00 Hz
 POINT 8192
 FREQU 8000.00 Hz
 SCANS 8
 ACQTM 1.0240 sec
 PD 5.9760 sec
 PW1 6.10 usec
 IRNUC 1H
 CTEMP 21.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 10



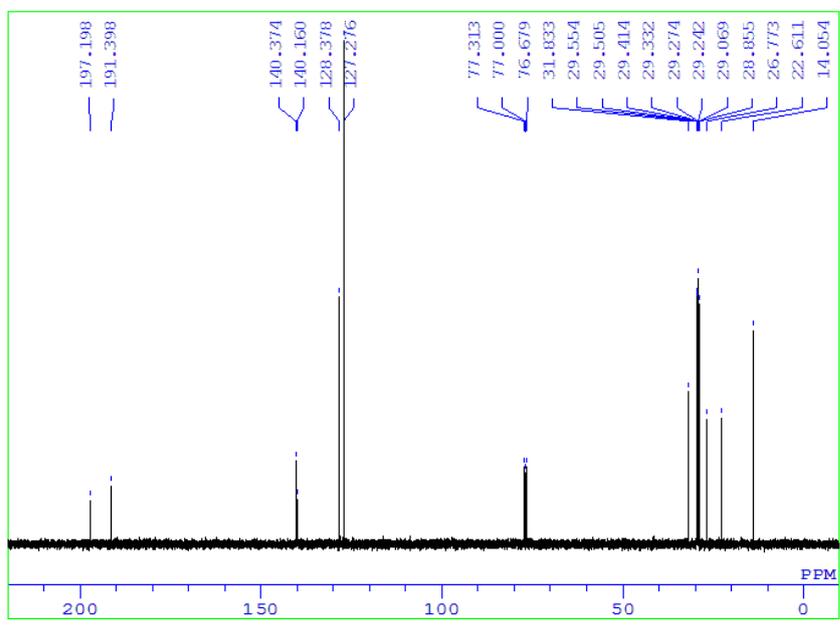
DFILE TU-400-4-13C.als
 COMNT TU-400-4
 DATIM Mon Jul 01 15:00:
 OBNUC 13C
 EXMOD bcm
 OBFRQ 100.40 MHz
 OBSET 0.00 KHz
 OBFIN 135500.00 Hz
 POINT 32768
 FREQU 27100.27 Hz
 SCANS 32
 ACQTM 1.2091 sec
 PD 1.7909 sec
 PW1 5.15 usec
 IRNUC 1H
 CTEMP 21.9 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 27



```

DFILE TU-400-3-1H.als
COMNT TU-400-3
DATIM Mon Jul 01 14:34:
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 8192
FREQU 8000.00 Hz
SCANS 8
ACQTM 1.0240 sec
PD 5.9760 sec
PW1 6.10 usec
IRNUC 1H
CTEMP 21.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 9

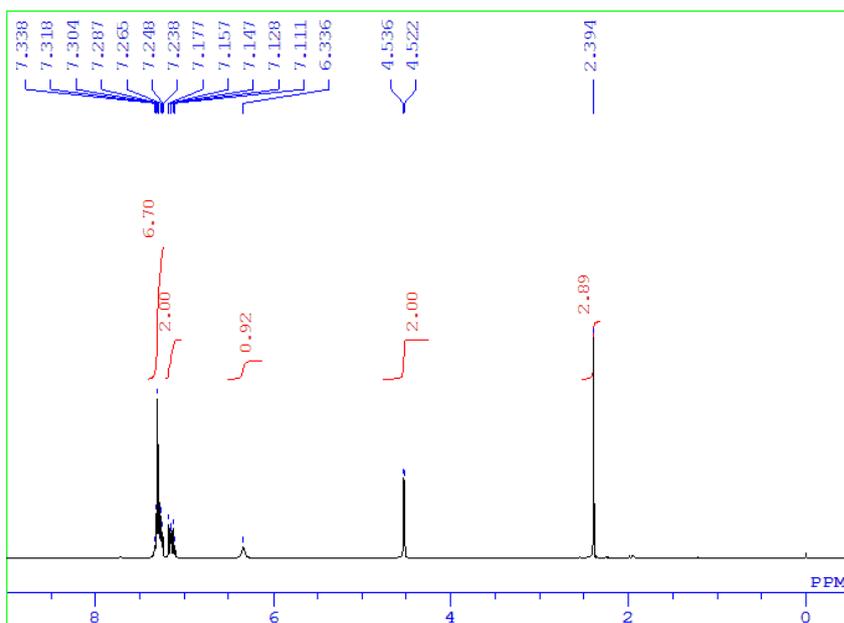
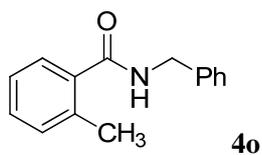
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DFILE TU-400-3-13C.als
COMNT TU-400-3
DATIM Mon Jul 01 14:38:
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 32
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 27

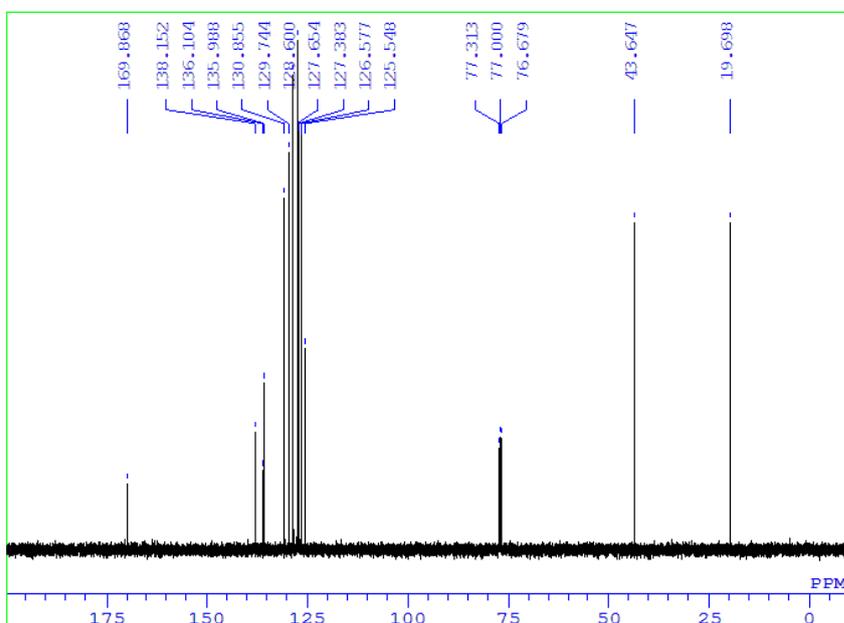
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```

DFILE TU-400-1-1H.als
COMNT TU-400-1
DATIM Mon Jul 01 10:02:
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 8192
FREQU 8000.00 Hz
SCANS 8
ACQTM 1.0240 sec
PD 5.9760 sec
PW1 6.10 usec
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 11

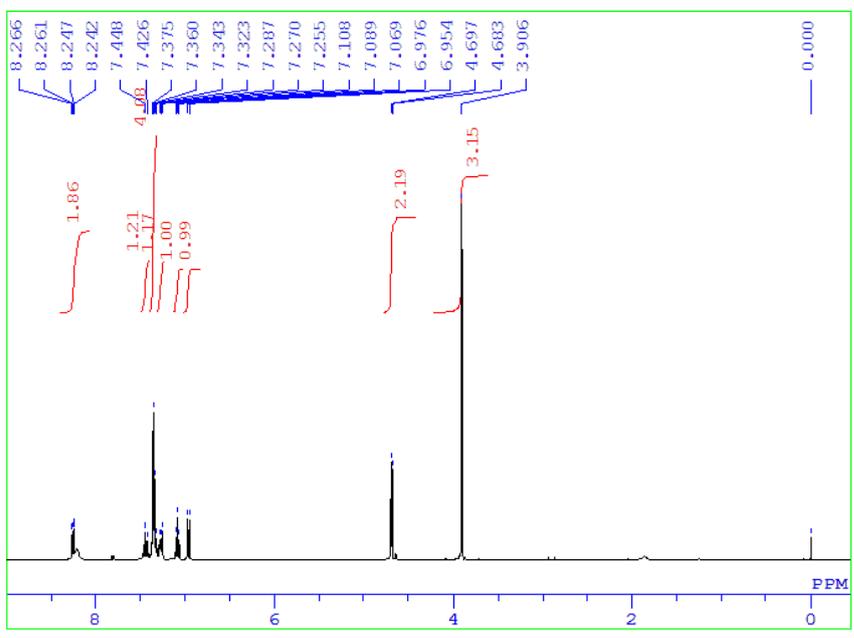
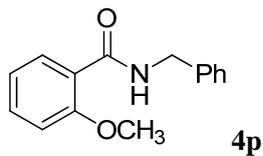
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```

DFILE TU-400-1-13C.als
COMNT TU-400-1
DATIM Mon Jul 01 10:06:
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 32
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 28

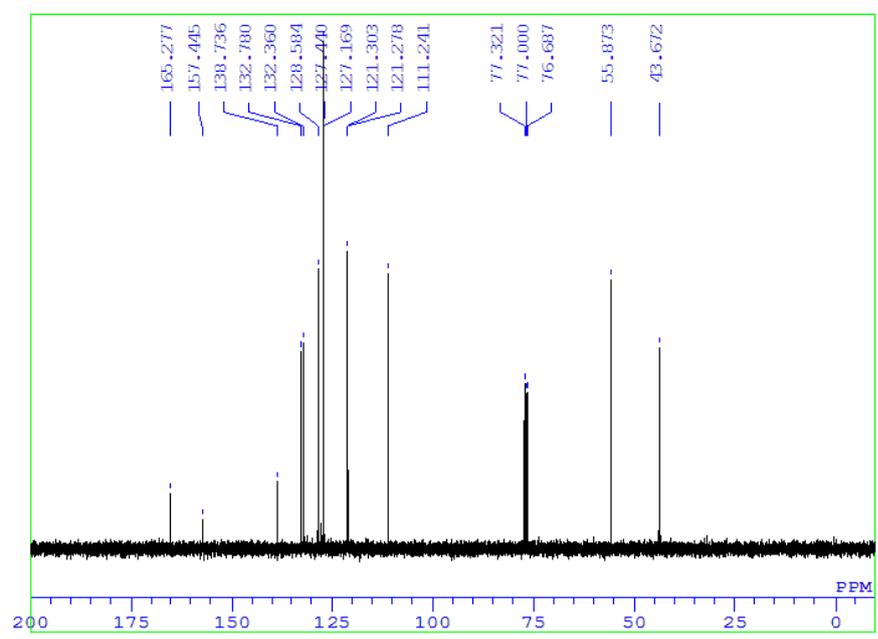
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```

DFILE TU-400-2-1H.als
COMNT TU-400-2
DATIM Mon Jul 01 13:41:
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 8192
FREQU 8000.00 Hz
SCANS 8
ACQTM 1.0240 sec
PD 5.9760 sec
PW1 6.10 usec
IRNUC 1H
CTEMP 21.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 15

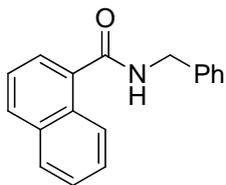
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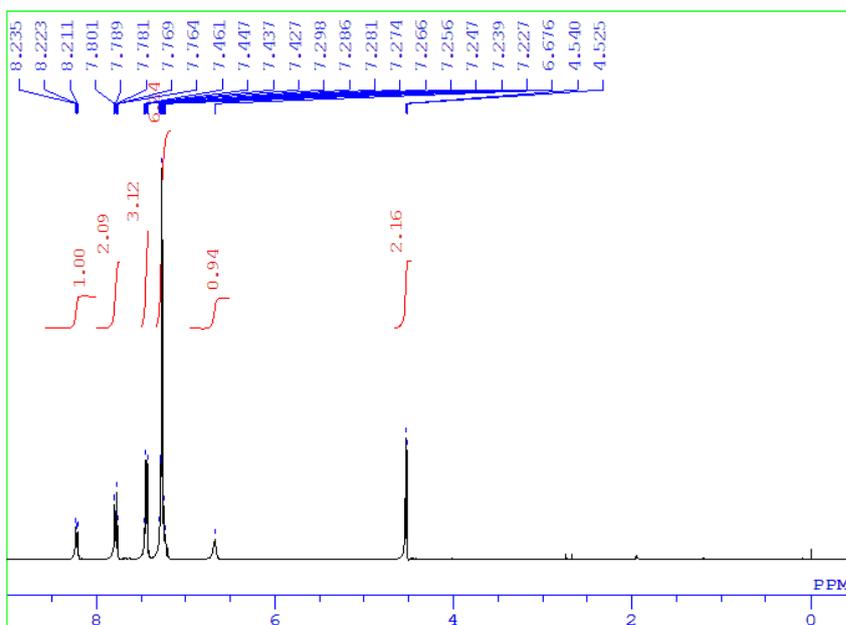
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DFILE TU-400-2-13C.als
COMNT TU-400-2
DATIM Mon Jul 01 13:48:
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 64
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 22.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 28

```

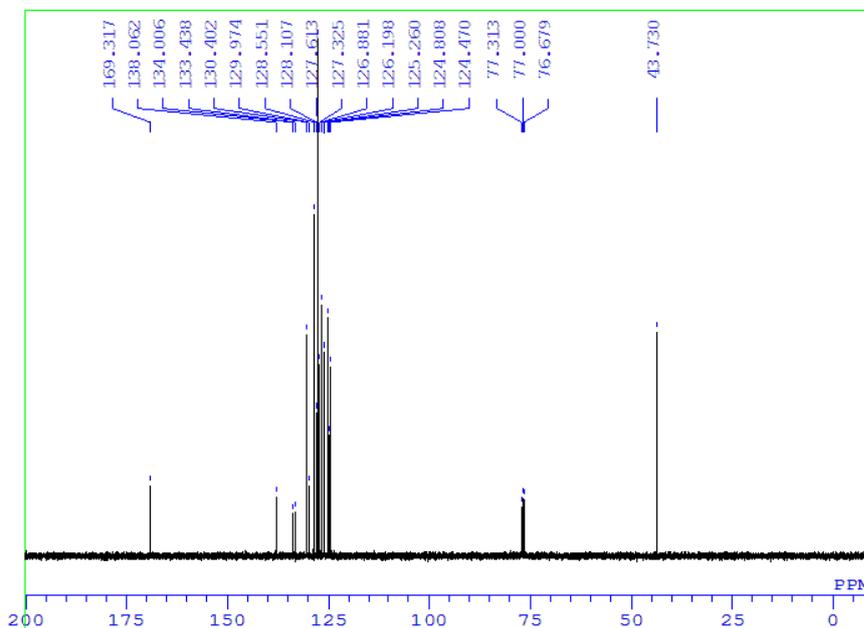


4d



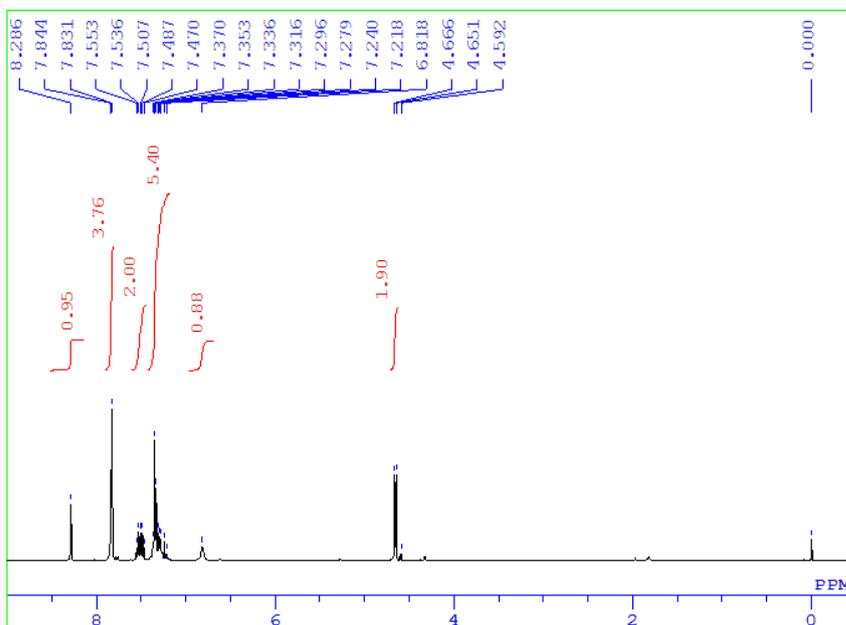
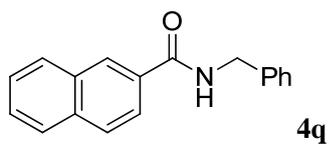
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DFILE TU-399-9-1H.als
COMNT TU-399-9
DATIM Wed Jun 26 14:43:
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 8192
FREQU 7993.60 Hz
SCANS 8
ACQTM 1.0248 sec
PD 5.9752 sec
PW1 5.90 usec
IRNUC 1H
CTEMP 20.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 11
  
```



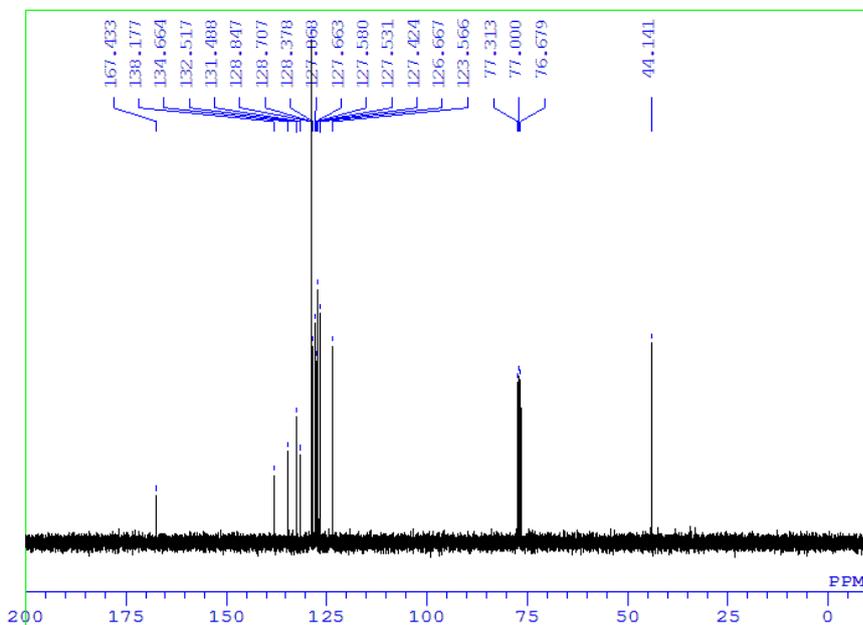
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DFILE TU-399-9-13C.als
COMNT TU-399-9
DATIM Wed Jun 26 14:51:
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 64
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 28
  
```



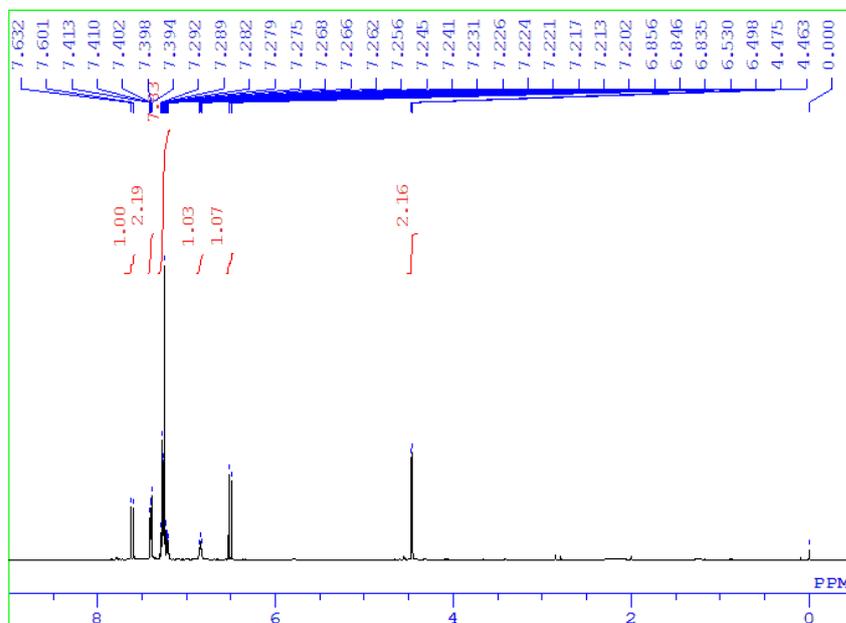
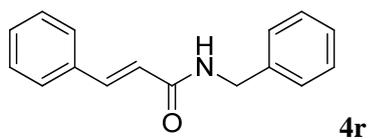
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DFILE TU-399-8-1H.als
COMNT TU-399-8
DATIM Tue Jun 25 20:06:
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 8192
FREQU 8000.00 Hz
SCANS 8
ACQTM 1.0240 sec
PD 5.9760 sec
PW1 6.10 usec
IRNUC 1H
CTEMP 22.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 15
  
```



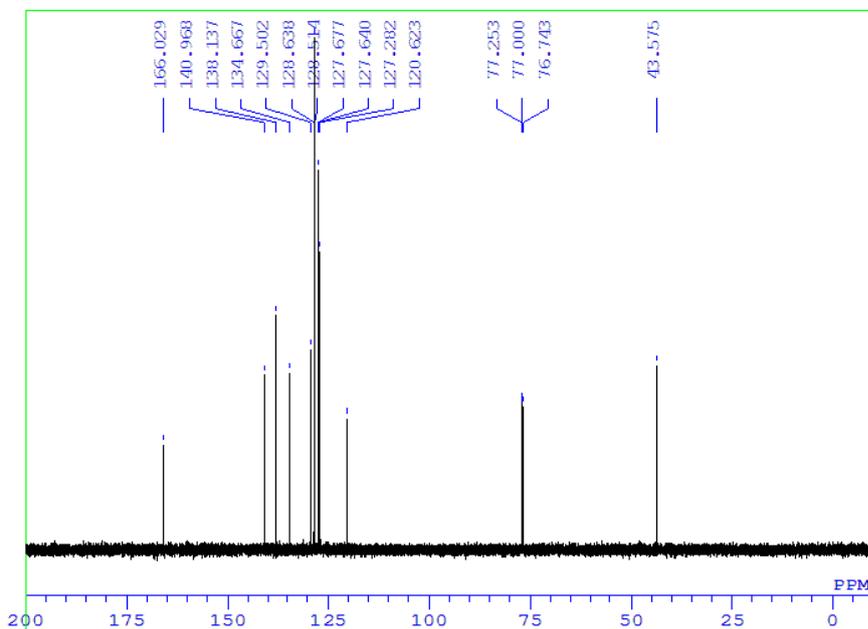
```

DFILE TU-399-8-13C.als
COMNT TU-399-8
DATIM Tue Jun 25 20:12:
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 64
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 22.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 28
  
```



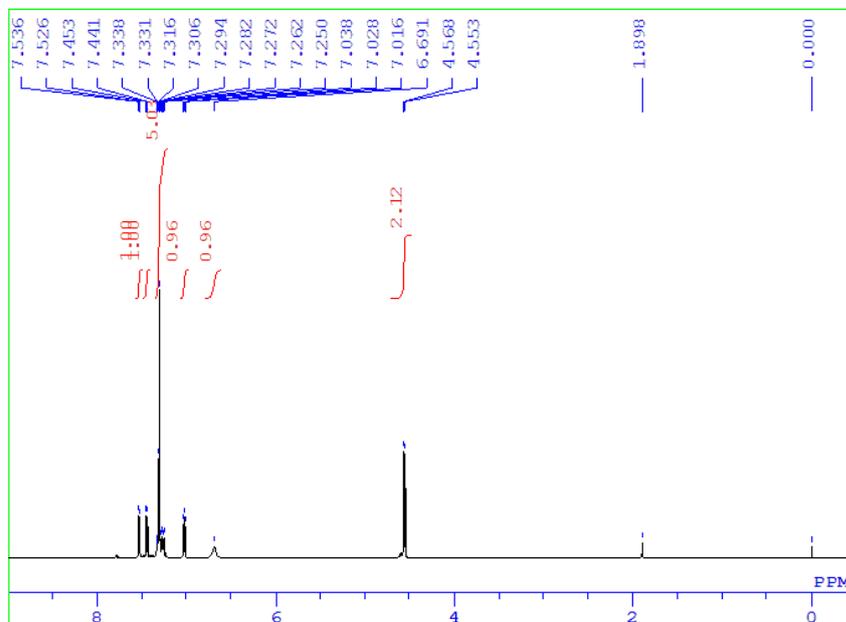
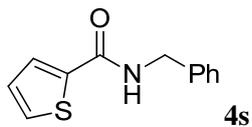
```

DFILE ZB00976-TU-402-2
COMNT TU-402-2-H
DATIM 2013-07-09 16:07:
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 8127.44 Hz
SCANS 16
ACQTM 2.0159 sec
PD 0.9000 sec
PW1 6.25 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 30
  
```



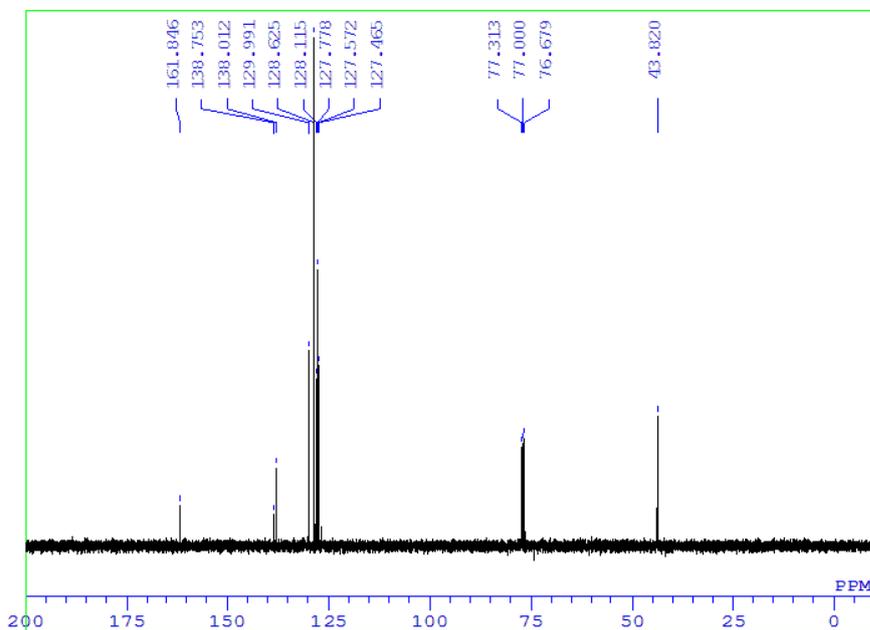
```

DFILE ZB00976-TU-402-2
COMNT TU-402-2-13C
DATIM 2013-07-09 16:10:
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 9.13 KHz
OBFIN 1.87 Hz
POINT 65535
FREQU 37878.79 Hz
SCANS 64
ACQTM 1.7302 sec
PD 0.8000 sec
PW1 3.28 usec
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 50
  
```



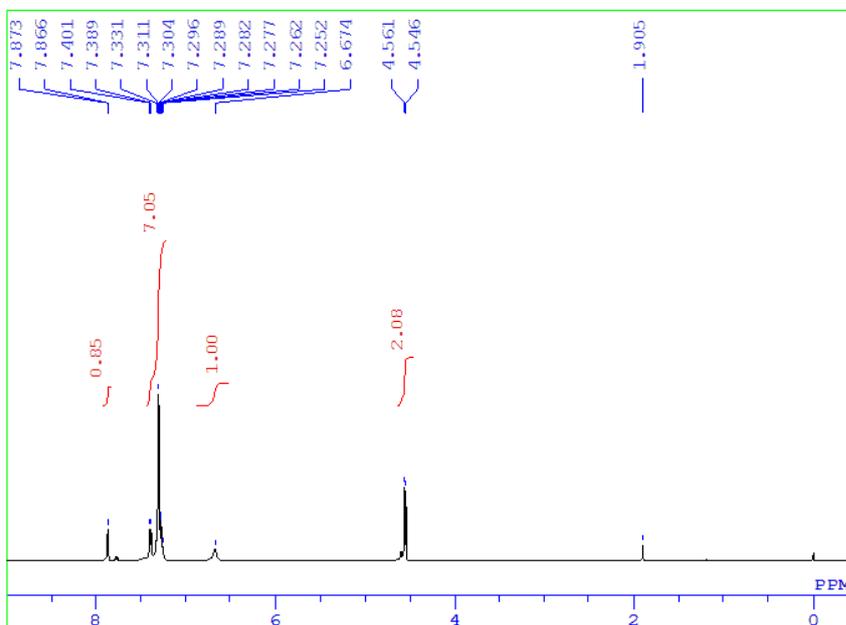
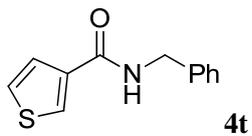
```

DFILE TU-401-4-1H.als
COMNT TU-401-4
DATIM Wed Jul 03 10:02:
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 8192
FREQU 8000.00 Hz
SCANS 8
ACQTM 1.0240 sec
PD 5.9760 sec
PWL 6.10 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 13
  
```



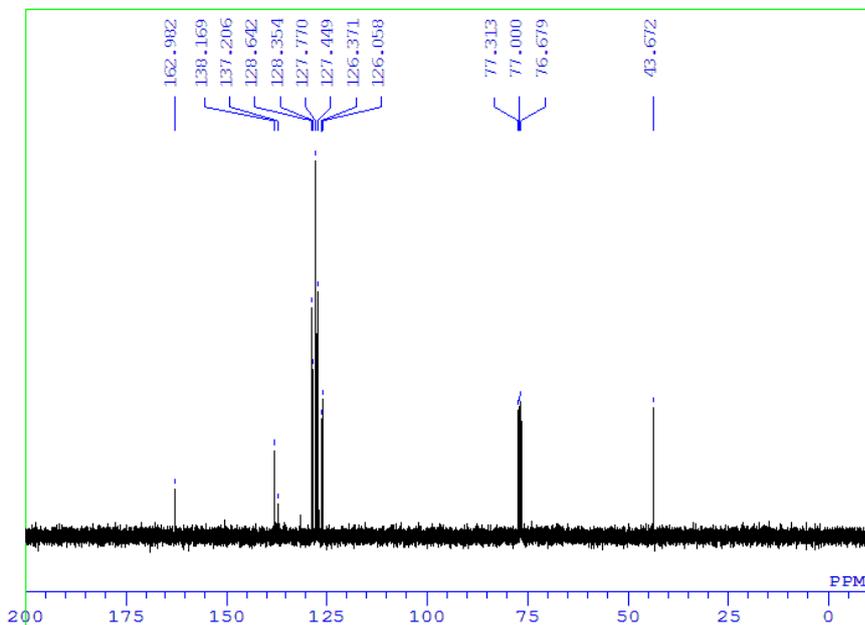
```

DFILE TU-401-4-13C.als
COMNT TU-401-4
DATIM Wed Jul 03 10:07:
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 32
ACQTM 1.2091 sec
PD 1.7909 sec
PWL 5.15 usec
IRNUC 1H
CTEMP 22.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 28
  
```



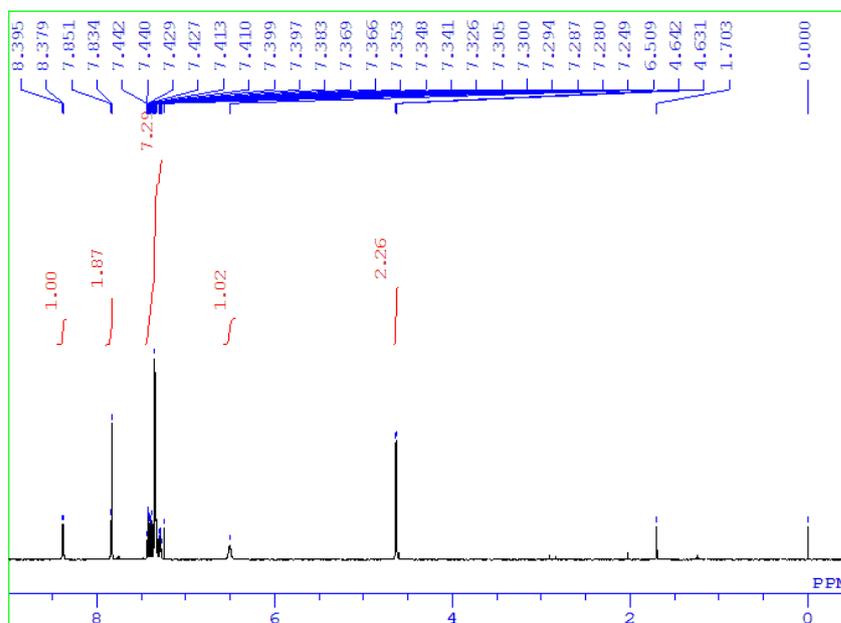
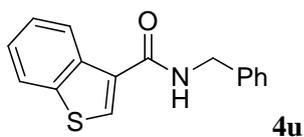
```

DFILE TU-401-3-1H-2.als
COMNT TU-401-3
DATIM Wed Jul 03 10:13:
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 8192
FREQU 8000.00 Hz
SCANS 8
ACQTM 1.0240 sec
PD 5.9760 sec
PW1 6.10 usec
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 15
  
```



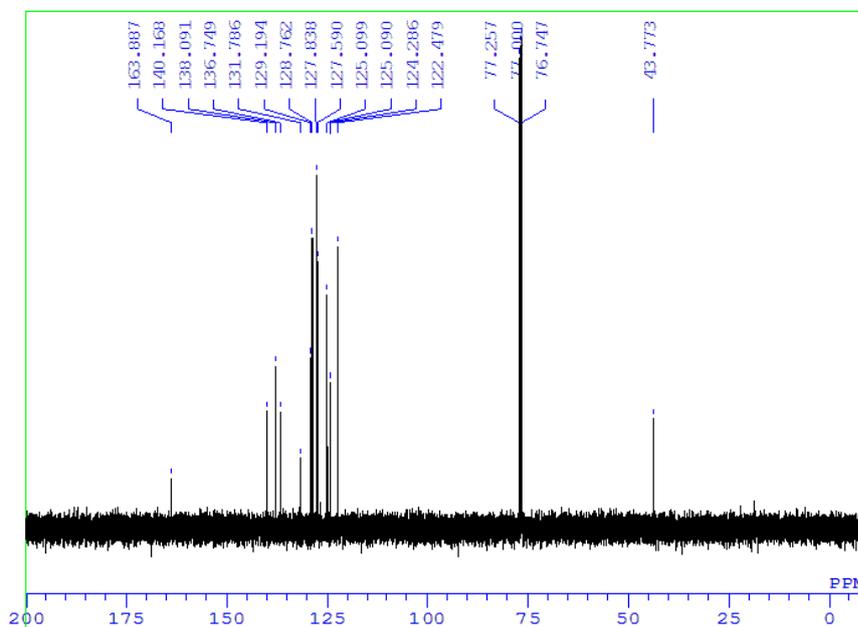
```

DFILE TU-401-3-13C.als
COMNT TU-401-3
DATIM Wed Jul 03 10:19:
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 32
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 22.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 28
  
```



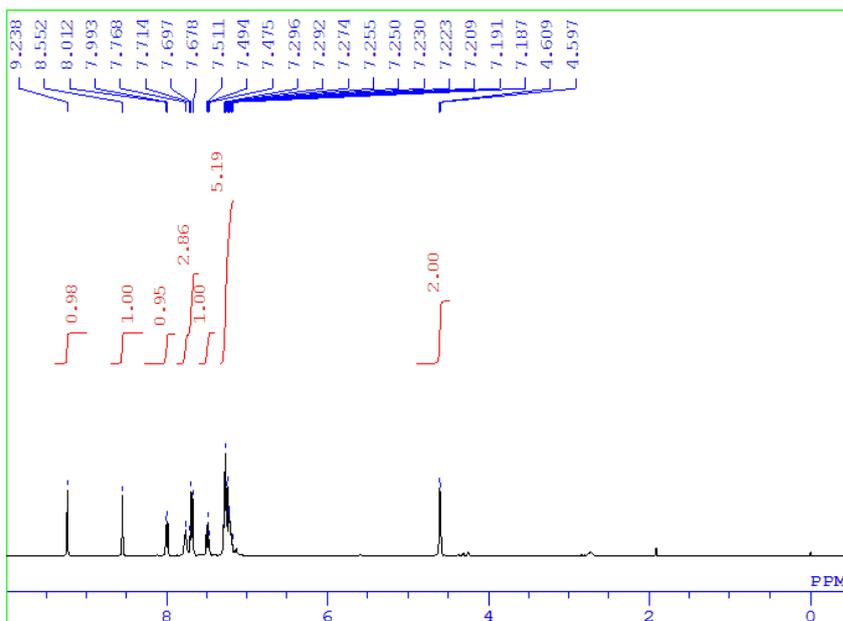
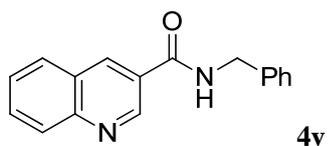
```

DFILE ZB00976-TU-402-6_
COMNT TU-402-6-H
DATIM 2013-07-09 14:34:
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 8127.44 Hz
SCANS 16
ACQTM 2.0159 sec
PD 0.9000 sec
PW1 6.25 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 40
  
```



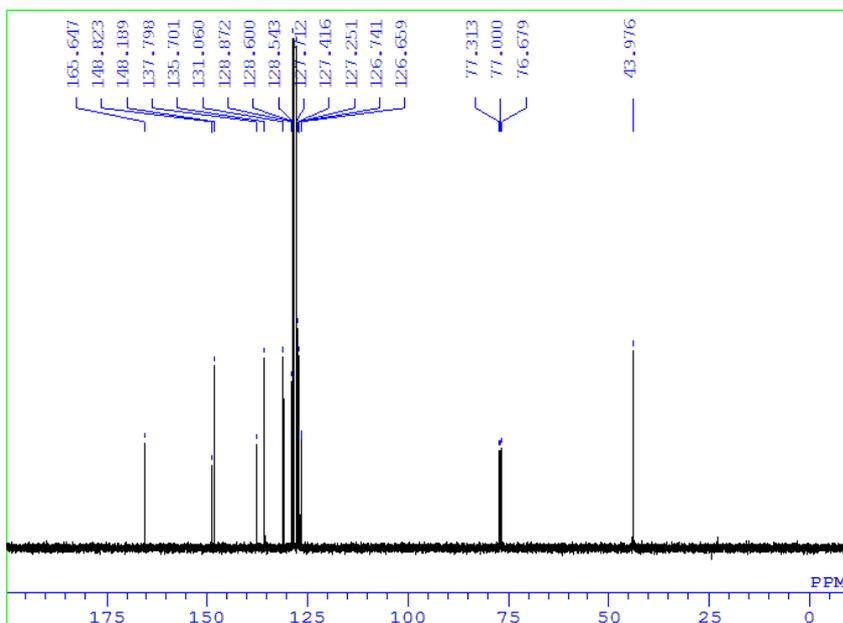
```

DFILE ZB00976-TU-402-6_
COMNT TU-402-6-13C
DATIM 2013-07-09 14:38:
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 9.13 KHz
OBFIN 1.87 Hz
POINT 65535
FREQU 37878.79 Hz
SCANS 64
ACQTM 1.7302 sec
PD 0.8000 sec
PW1 3.28 usec
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50
  
```



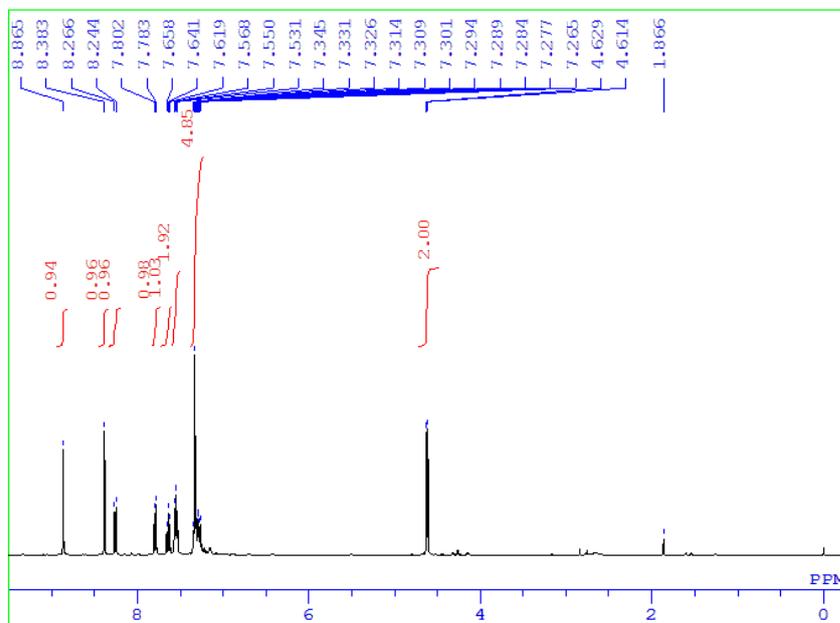
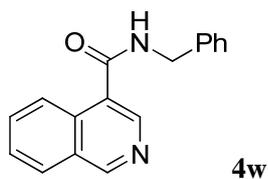
```

DFILE TU-401-7-1H.als
COMNT TU-401-7
DATIM Thu Jul 04 13:18:
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 8192
FREQU 8000.00 Hz
SCANS 8
ACQTM 1.0240 sec
PD 5.9760 sec
PW1 6.10 usec
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 12
  
```



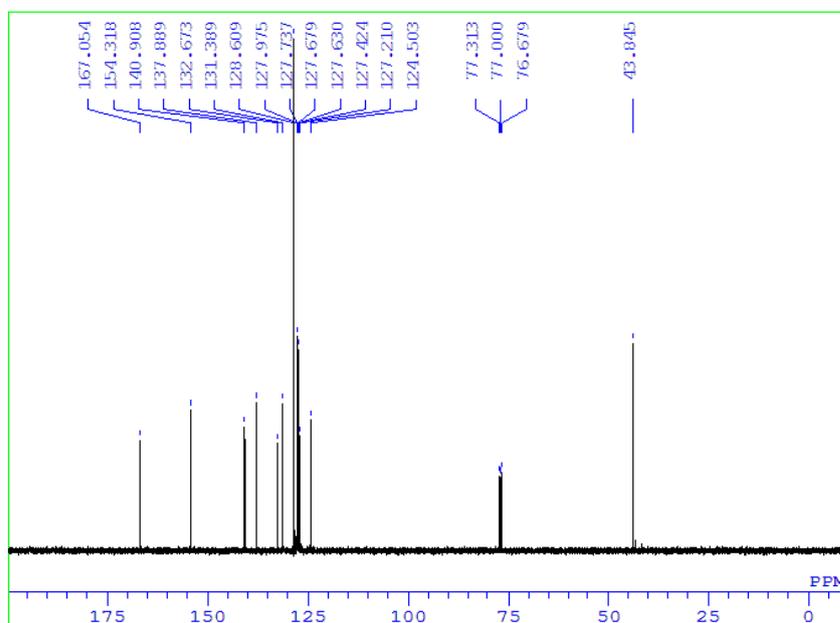
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DFILE TU-401-7-13C.als
COMNT TU-401-7
DATIM Thu Jul 04 13:27:
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 64
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 21.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 27
  
```



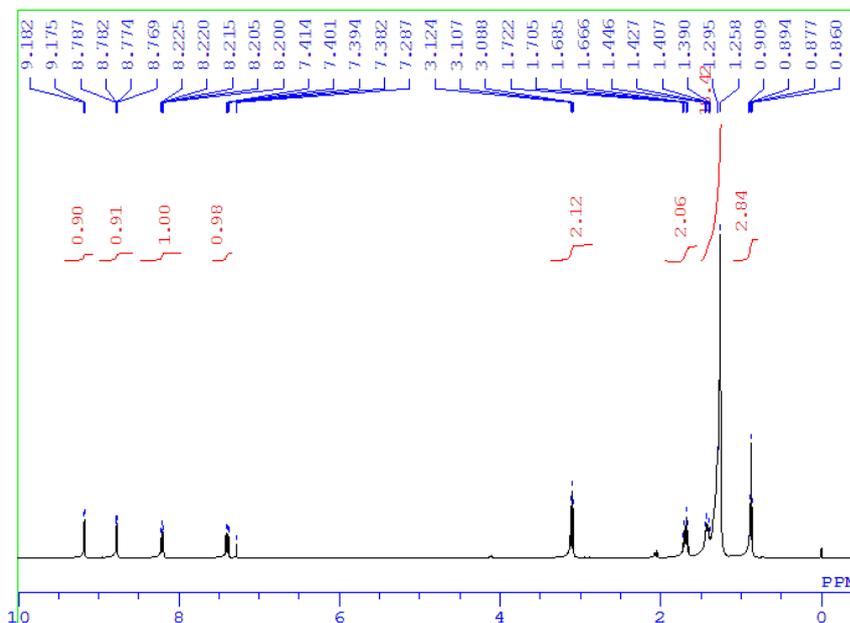
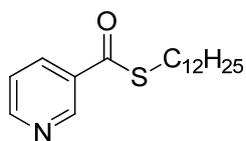
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DFILE TU-401-8-1H.als
COMNT TU-401-8
DATIM Thu Jul 04 13:41:
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 8192
FREQU 8000.00 Hz
SCANS 8
ACQTM 1.0240 sec
PD 5.9760 sec
PW1 6.10 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 13
  
```



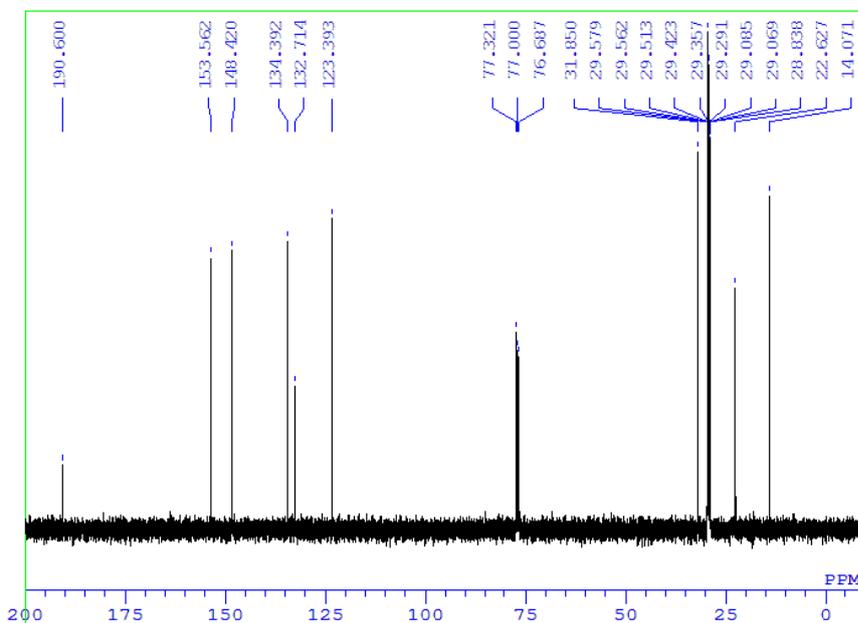
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DFILE TU-401-8-13C.als
COMNT TU-401-8
DATIM Thu Jul 04 13:48:
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 64
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 28
  
```



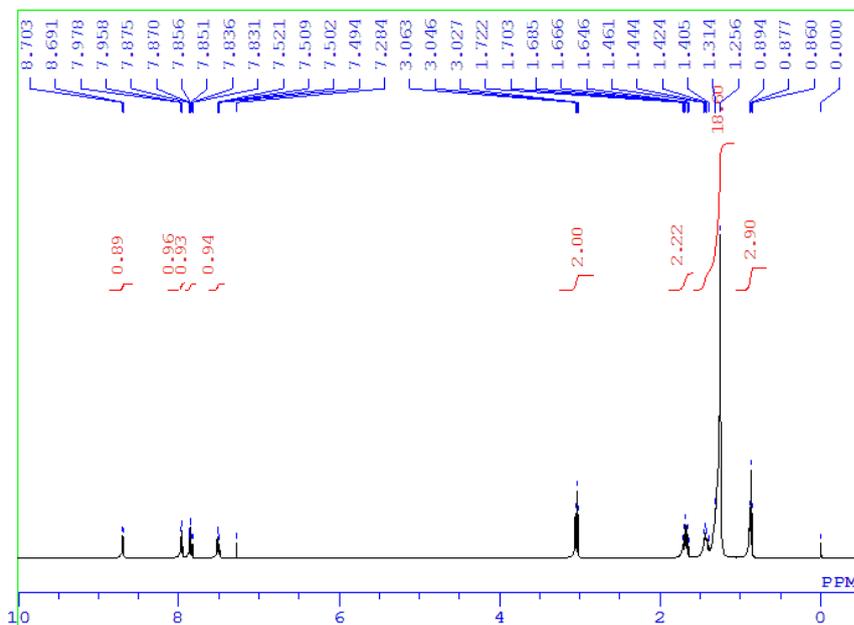
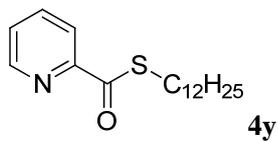
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DFILE TU-401-5-1H.als
COMNT TU-401-5
DATIM Wed Jul 03 14:24:
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 8192
FREQU 8000.00 Hz
SCANS 8
ACQTM 1.0240 sec
PD 5.9760 sec
PW1 6.10 usec
IRNUC 1H
CTEMP 20.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 10
  
```



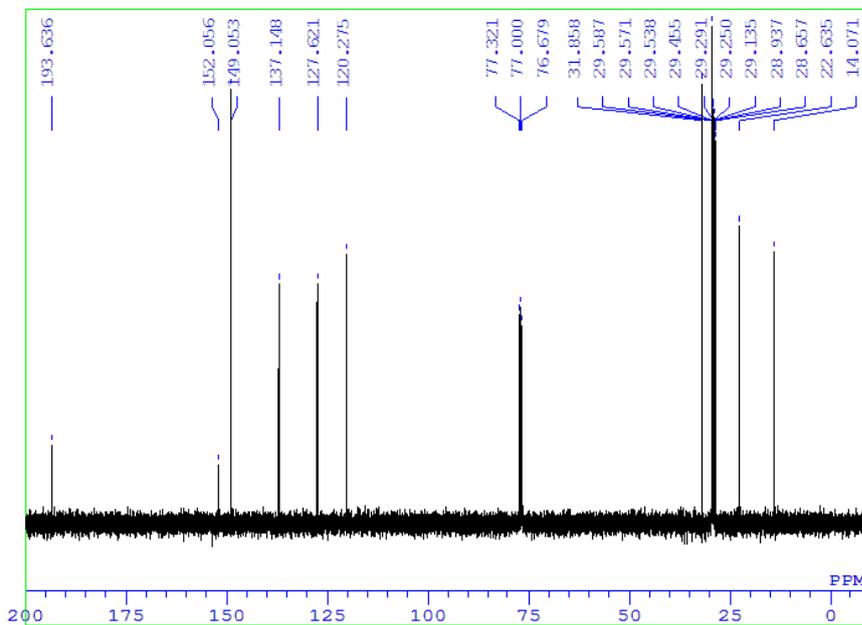
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DFILE TU-401-5-13C.als
COMNT TU-401-5
DATIM Wed Jul 03 14:32:
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 64
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 21.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 28
  
```



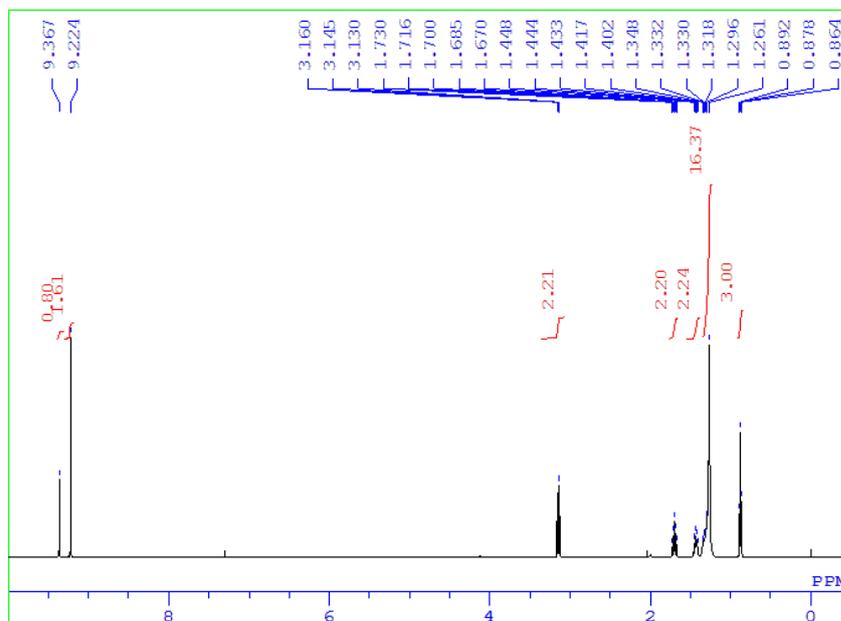
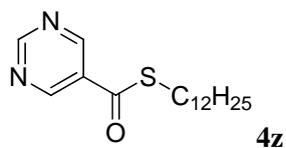
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DFILE TU-401-6-1H.als
COMNT TU-401-6
DATIM Wed Jul 03 17:15:
OBNUC 1H
EXMOD non
OBFRQ 399.65 MHz
OBSET 0.00 KHz
OBFIN 134300.00 Hz
POINT 8192
FREQU 8000.00 Hz
SCANS 8
ACQTM 1.0240 sec
PD 5.9760 sec
PW1 6.10 usec
IRNUC 1H
CTEMP 20.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 11
  
```



```

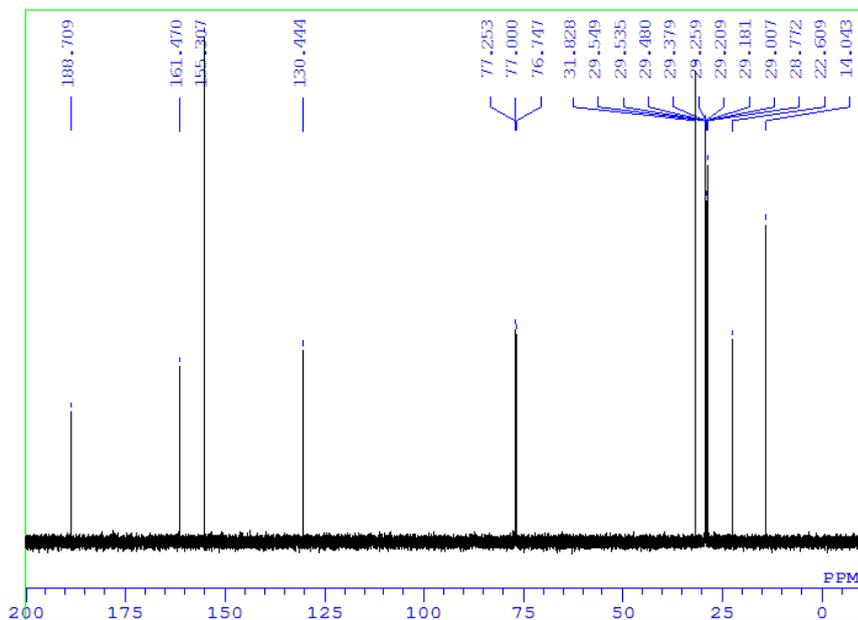
DFILE TU-401-6-13C.als
COMNT TU-401-6
DATIM Wed Jul 03 17:20:
OBNUC 13C
EXMOD bcm
OBFRQ 100.40 MHz
OBSET 0.00 KHz
OBFIN 135500.00 Hz
POINT 32768
FREQU 27100.27 Hz
SCANS 64
ACQTM 1.2091 sec
PD 1.7909 sec
PW1 5.15 usec
IRNUC 1H
CTEMP 22.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 28
  
```



```

DFILE ZB00976-TU-402-1
COMNT TU-402-1-H
DATIM 2013-07-09 18:05:
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 8127.44 Hz
SCANS 16
ACQTM 2.0159 sec
PD 0.9000 sec
PW1 6.25 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 26

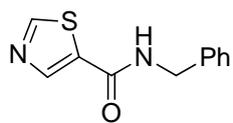
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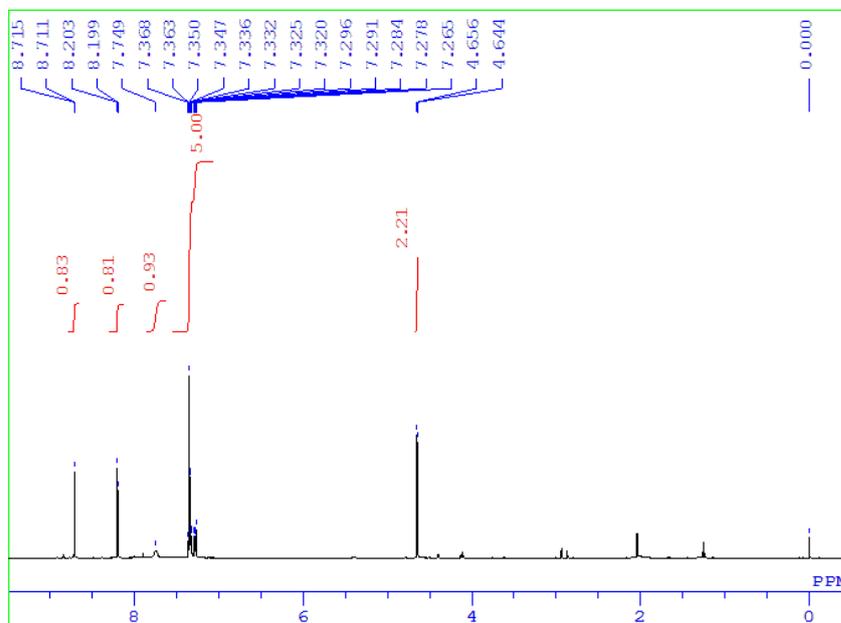
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DFILE ZB00976-TU-402-1
COMNT TU-402-1-13C
DATIM 2013-07-09 18:08:
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 9.13 KHz
OBFIN 1.87 Hz
POINT 65535
FREQU 37878.79 Hz
SCANS 64
ACQTM 1.7302 sec
PD 0.8000 sec
PW1 3.28 usec
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 50

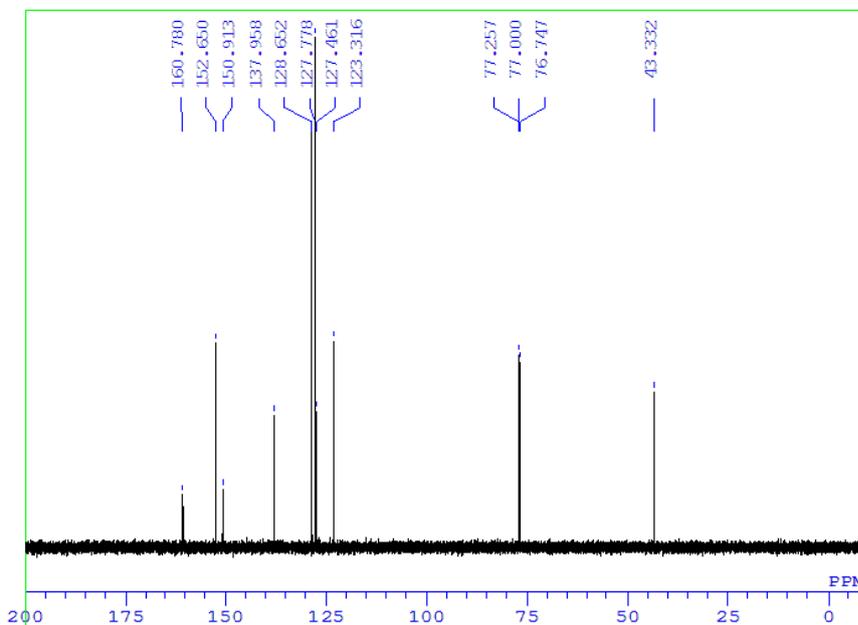
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4za



DFILE ZB00976-TU-402-4_
 COMNT TU-402-4-H
 DATIM 2013-07-09 17:52:
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 16384
 FREQU 8127.44 Hz
 SCANS 16
 ACQTM 2.0159 sec
 PD 0.9000 sec
 PW1 6.25 usec
 IRNUC 1H
 CTEMP 21.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 38



DFILE ZB00976-TU-402-4_
 COMNT TU-402-4-13C
 DATIM 2013-07-09 17:55:
 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 125.77 MHz
 OBSET 9.13 KHz
 OBFIN 1.87 Hz
 POINT 65535
 FREQU 37878.79 Hz
 SCANS 64
 ACQTM 1.7302 sec
 PD 0.8000 sec
 PW1 3.28 usec
 IRNUC 1H
 CTEMP 22.0 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 50