Efficient and convenient preparation of 3-aryl-2,2-dimethylpropanoates via Negishi coupling

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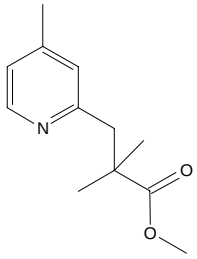
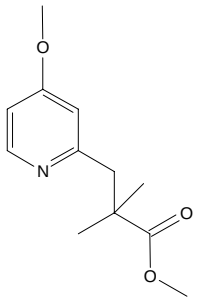
Supplementary Material;

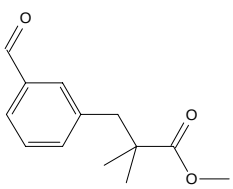
1. General procedure for pyridine and pyrimidine substrates.
2. Spectral data of all the new compounds.
3. Experimental procedure for compound **5**
4. Copies of ^{1}H, ^{13}C, HRMS, FT-IR for all new compounds (Note: All the compounds below were > 95% by HPLC unless described otherwise.)



General procedure for the Negishi coupling; Preparation of **3**: A suspension of Zn-Cu (3.45 g) couple in toluene / DMA (13:1, 30 mL) was degassed by bubbling N_2 into the system for 15 min. Iodide **1** (2.21 g, 9.14 mmol) was added to the suspension, and the resulting mixture heated to 110 ^{o}C for 6 hr. The rxn was allowed to cool to 70 ^{o}C and 2-bromo-4-methyl pyridine (751 uL, 6.77 mmol) and $Pd(PPh_3)_4$ (235 mg, 0.203 mmol) were added. The reaction was maintained at 70 ^{o}C for 22 hr. Upon cooling, the mixture was filtered, and the filter cake rinsed with Et_2O. The filtrate was extracted with 1N HCl (2x75 mL). The acidic extracts were basified by addition of $NaHCO_3$, and the resulting solution extracted with Et_2O (2x75 mL). The combined organics were dried over Na_2SO_4 and the solvent evaporated to yield **3** (1.31 g, 6.32 mmol, 93%) as an oil which was pure by NMR and HPLC.^{1}H NMR (400 MHz, DICHLOROMETHANE-*d*2) δ ppm 1.19 (s, 6 H) 2.32 (s, 3 H) 3.00 (s, 2 H) 3.63 (s, 3 H) 6.94 (s, 1 H) 6.99 (d, *J*=4.93 Hz, 1 H) 8.32 (d, *J*=5.05 Hz, 1 H); ^{13}C NMR (101 MHz, DICHLOROMETHANE-*d*2) δ ppm 21.23 (s) 25.51 (s) 43.46 (s) 48.54 (s) 51.99 (s) 122.84 (s) 125.46 (s) 147.51 (s) 149.13 (s) 158.86 (s) 178.17 (s); HRMS-ESI (m/z): [M + H]+ calculated for C12H18NO2, 208.1338; found, 208.1336.

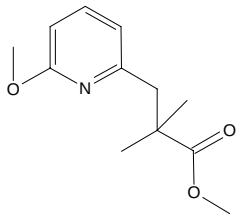


Compound 4: ^{1}H NMR (400 MHz, DICHLOROMETHANE-*d*2) δ ppm 1.19 (s, 6 H) 2.96 (s, 2 H) 3.64 (s, 3 H) 3.81 (s, 3 H) 6.61 (d, *J*=2.53 Hz, 1 H) 6.67 (dd, *J*=5.75, 2.46 Hz, 1 H) 8.28 (d, *J*=5.81 Hz, 1 H); ^{13}C NMR (101 MHz, DICHLOROMETHANE-*d*2) δ ppm 150.56 (s) 160.72 (s) 166.24 (s) 178.14 (s); HRMS-ESI (m/z): [M + H]+ calculated for C12H18NO3, 224.1287; found, 224.1282.

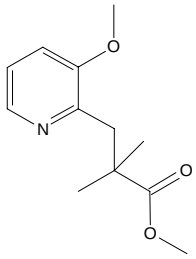


Compound 5: To a stirred solution of methyl 3-iodo-2,2-

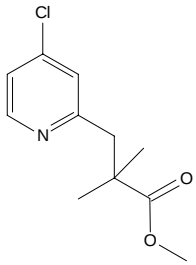
dimethylpropanoate (145mg, 0.599 mmol) in Toluene (3 ml) and DMA (0.5 ml) were added Zinc-Copper couple (157 mg, 2.396 mmol). The reaction was stirred for 2 hrs at 100 ºC and cooled to 70 ºC. 3-bromobenzaldehyde (166 mg, 0.899 mmol) and PalladiumTetrakis (20.77 mg, 0.018 mmol) were added and the reaction was stirred for overnight. The reaction was cooled to rt and diluted with EtOAc (20 ml). It was filtered to remove the insolubles and the filtrate was washed with 1N-HCl (15 ml) and brine. The separated organic layer was dried with $MgSO_4$ and the volatiles were removed in vacuo. The resulting crude oil was purified by column chromatography (heptane and ethylacetate gradient) to provide 103 mg (78%, 0.468 mmol) of the product. ^{1}H NMR (400 MHz, DICHLOROMETHANE-*d*2) δ ppm 1.15 (s, 6 H) 2.90 (br. s., 2 H) 3.61 (s, 3 H) 7.36 (br. s., 1 H) 7.42 (br. s., 1 H) 7.58 (s, 1H) 7.70 (br. s., 1 H) 9.95 (s, 1 H); ^{13}C NMR (101 MHz, DICHLOROMETHANE-*d*2) δ ppm 25.04 (s) 43.89 (s) 46.20 (s) 51.90 (s) 128.30 (s) 129.01 (s) 131.24 (s) 136.45 (s) 136.72 (s) 139.85 (s) 177.51 (s) 192.59 (s); HRMS-ESI (m/z): [M+H]+ calculated for C13H16O3, 221.1178; found, 221.1169.



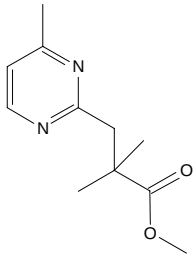
Entry 1: ^{1}H NMR (400 MHz, DICHLOROMETHANE-*d*2) δ ppm 1.21 (s, 6 H) 3.06 (s, 2 H) 3.65 (s, 3 H) 3.87 (s, 3 H) 6.60 (d, *J*=8.21 Hz, 1 H) 6.72 (d, *J*=7.33 Hz, 1 H) 7.55 (t, *J*=7.83 Hz, 1 H); ^{13}C NMR (101 MHz, DICHLOROMETHANE-*d*2) δ ppm 25.74 (s) 42.96 (s) 47.98 (s) 51.96 (s) 108.47 (s) 117.01 (s) 139.02 (s) 157.03 (s) 163.87 (s) 178.28 (s); HRMS-ESI (m/z): [M+H]+ calculated for C12H18NO3, 224.1287; found, 224.1279.



Entry 2: ^{1}H NMR (400 MHz, DICHLOROMETHANE-*d*2) δ ppm 1.19 (s, 6 H) 3.06 (s, 2 H) 3.61 (s, 3 H) 3.79 (s, 3 H) 7.03 - 7.19 (m, 2 H) 8.04 (dd, *J*=3.79, 2.40 Hz, 1 H); ^{13}C NMR (101 MHz, DICHLOROMETHANE-*d*2) δ ppm 25.66 (s) 41.85 (s) 42.62 (s) 51.91 (s) 55.64 (s) 117.01 (s) 122.41 (s) 140.48 (s) 149.13 (s) 154.59 (s) 178.55 (s); HRMS-ESI (m/z): [M+H]+ calculated for C12H18NO3, 224.1287; found, 224.1282.

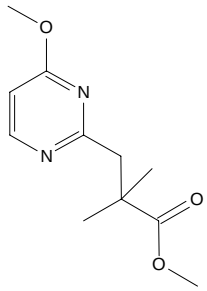


Entry 3: ^{1}H NMR (400 MHz, DICHLOROMETHANE-*d*2) δ ppm 1.19 (s, 6 H) 3.00 (s, 2 H) 3.64 (s, 3 H) 7.09 - 7.19 (m, 2 H) 8.38 (d, *J*=5.31 Hz, 1 H); ^{13}C NMR (101 MHz, DICHLOROMETHANE-*d*2) δ ppm 25.53 (s) 43.46 (s) 48.22 (s) 52.12 (s) 122.28 (s) 124.88 (s) 150.28 (s) 160.93 (s) 177.84 (s); HRMS-ESI (m/z): [M+H]+ calculated for C11H15ClNO2, 228.0791; found, 228.0785.

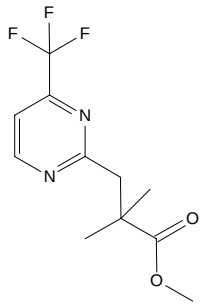


Entry 5: ^{1}H NMR (400 MHz, DICHLOROMETHANE-*d*2) δ ppm 1.21 (s, 6 H) 2.42 (s, 3 H) 3.14 (s, 2 H) 3.63 (s, 3 H) 6.97 (d, *J*=5.05 Hz, 1 H) 8.44 (d, *J*=5.18 Hz, 1 H); ^{13}C NMR (101 MHz, DICHLOROMETHANE-*d*2) δ ppm 24.43 (s) 25.67 (s) 42.61 (s) 49.70 (s) 51.99 (s) 118.54 (s) 156.74 (s) 167.14 (s) 168.37 (s) 178.22

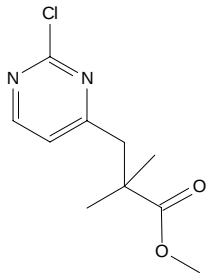
(s); HRMS-ESI (m/z): [M+H]+ calculated for C11H17N2O2, 209.1290; found, 209.1283.



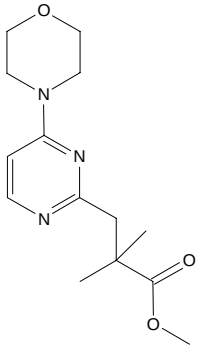
Entry 6: ^{1}H NMR (400 MHz, DICHLOROMETHANE-*d*2) δ ppm 1.24 (s, 6 H) 3.13 (s, 2 H) 3.64 (s, 3 H) 3.88 (s, 3 H) 6.53 (d, *J*=5.81 Hz, 1 H) 8.29 (d, *J*=5.81 Hz, 1 H); ^{13}C NMR (101 MHz, DICHLOROMETHANE-*d*2) δ ppm 25.82 (s) 42.23 (s) 49.31 (s) 52.00 (s) 54.16 (s) 105.98 (s) 157.32 (s) 168.73 (s) 169.62 (s) 178.23 (s); HRMS-ESI (m/z): [M+H]+ calculated for C11H17N2O3, 225.1239; found, 225.1234.



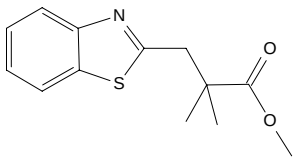
Entry 7: ^{1}H NMR (400 MHz, DICHLOROMETHANE-*d*2) δ ppm 1.26 (s, 6 H) 3.32 (s, 2 H) 3.64 (s, 3 H) 7.48 (d, *J*=5.05 Hz, 1 H) 8.90 (d, *J*=5.05 Hz, 1 H); ^{13}C NMR (101 MHz, DICHLOROMETHANE-*d*2) δ ppm 25.68 (s) 42.66 (s) 49.45 (s) 52.10 (s) 114.82 (q, *J*=2.56 Hz) 121.15 (q, *J*=274.80 Hz) 155.51 (q, *J*=35.86 Hz) 159.85 (s) 170.18 (s) 177.74 (s); HRMS-ESI (m/z): [M+H]+ calculated for C11H14F3N2O2, 263.1007; found, 263.1004.



Entry 8: ^{1}H NMR (400 MHz, DICHLOROMETHANE-*d*2) δ ppm 1.22 (s, 6 H) 2.99 (s, 2 H) 3.67 (s, 3 H) 7.09 (d, *J*=5.05 Hz, 1 H) 8.47 (d, *J*=5.05 Hz, 1 H); ^{13}C NMR (101 MHz, ACETONITRILE-*d*3) δ ppm 25.48 (s) 43.40 (s) 47.65 (s) 52.43 (s) 121.73 (s) 160.60 (s) 161.29 (s) 172.13 (s) 177.67 (s); HRMS-ESI (m/z): [M+H]+ calculated for C10H14ClN2O2, 229.0744; found, 229.0741.

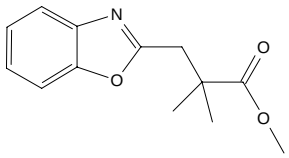


Entry 9: ^{1}H NMR (400 MHz, DICHLOROMETHANE-*d*2) δ ppm 1.22 (s, 6 H) 3.03 (s, 2 H) 3.48 - 3.57 (m, 4 H) 3.60 (s, 3 H) 3.68 - 3.75 (m, 4 H) 6.31 (d, *J*=6.19 Hz, 1 H) 8.11 (d, *J*=6.19 Hz, 1 H); ^{13}C NMR (101 MHz, DICHLOROMETHANE-*d*2) δ ppm 25.88 (s) 41.98 (s) 44.65 (s) 49.43 (s) 51.91 (s) 66.97 (s) 100.62 (s) 155.95 (s) 162.16 (s) 167.73 (s) 178.46 (s); HRMS-ESI (m/z): [M+H]+ calculated for C14H22N3O3, 280.1661; found, 280.1658.



Entry 10: ^{1}H NMR (400 MHz, DICHLOROMETHANE-*d*2) δ ppm 1.21 (br. s., 6 H) 3.28 (s, 2 H) 3.64 (s, 3 H) 7.27 (s, 1 H) 7.37 (s, 1 H) 7.77 (br. s., 1 H) 7.85 (br. s., 1 H); ^{13}C NMR (101 MHz, DICHLOROMETHANE-*d*2) δ ppm 25.47 (s) 43.64 (s)

44.33 (s) 52.26 (s) 121.76 (s) 122.97 (s) 125.13 (s) 126.14 (s) 135.90 (s) 153.56 (s) 167.83 (s) 177.21 (s); HRMS-ESI (m/z): [M+H]+ calculated for C13H15ClNO2S, 250.0902; found, 250.09019.



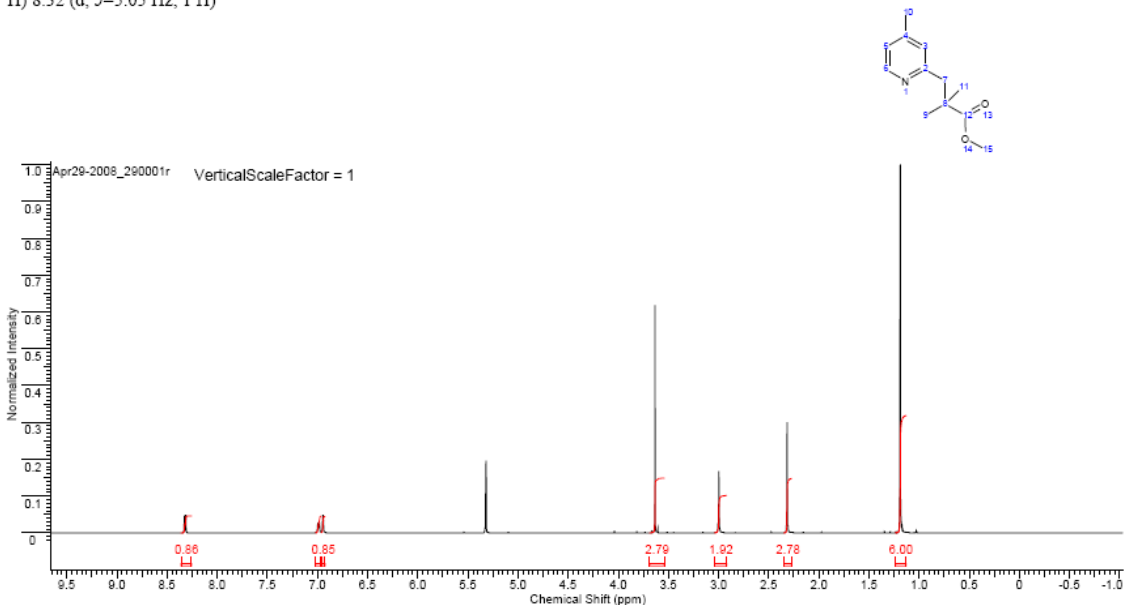
Entry 11: ^{1}H NMR (400 MHz, DICHLOROMETHANE-*d*2) δ ppm 1.21 (s, 6 H) 3.08 (s., 2 H) 3.59 (s, 3 H) 7.19 (m, 2 H) 7.38 (m., 1 H) 7.54(m, 1 H); ^{13}C NMR (101 MHz, DICHLOROMETHANE-*d*2) δ ppm 25.26 (s) 38.86 (s) 42.31 (s) 52.23 (s) 110.51 (s) 119.83 (s) 124.28 (s) 124.80 (s) 141.66 (s) 151.00 (s) 164.53 (s) 176.90 9s); HRMS-ESI (m/z): [M+H]+ calculated for C13H15NO3, 234.1130; found, 234.11234.

Compound 3

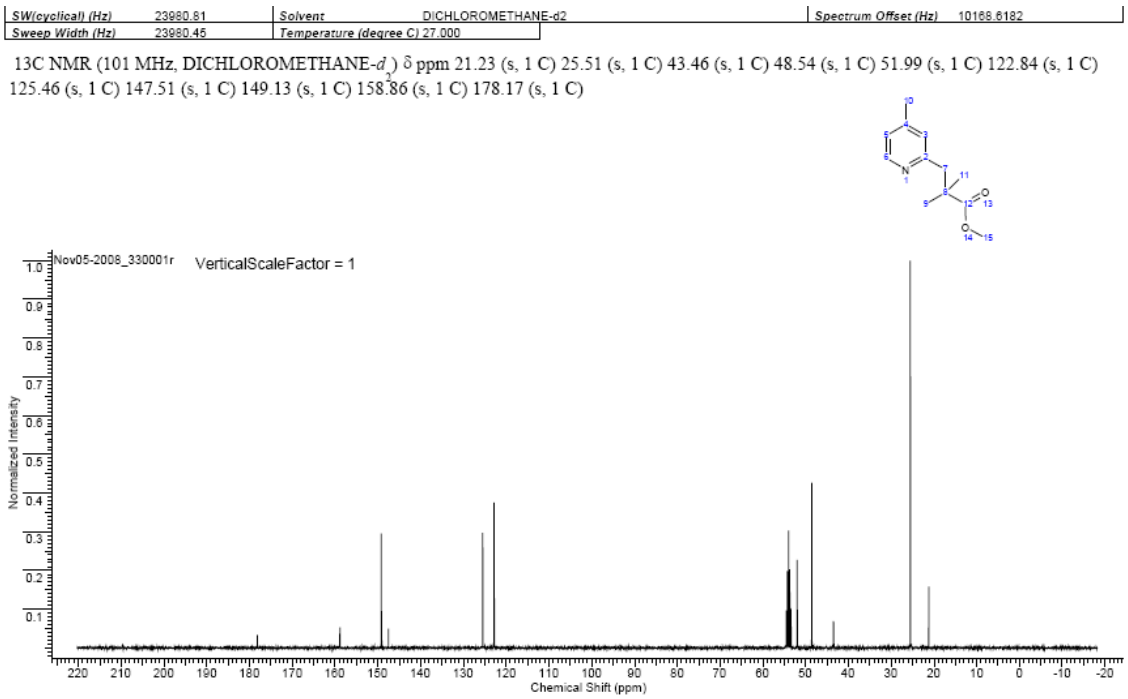
^{1}H NMR

SW(cyclical) (Hz)	8278.15	*Solvent*	DICHLOROMETHANE-d2	*Spectrum Offset (Hz)*	2431.2781
Sweep Width (Hz)	8278.02	*Temperature (degree C)*	27.000		

1H NMR (400 MHz, DICHLOROMETHANE-d_2) δ ppm 1.19 (s, 6 H) 2.32 (s, 3 H) 3.00 (s, 2 H) 3.63 (s, 3 H) 6.94 (s, 1 H) 6.99 (d, *J*=4.93 Hz, 1 H) 8.32 (d, *J*=5.05 Hz, 1 H)

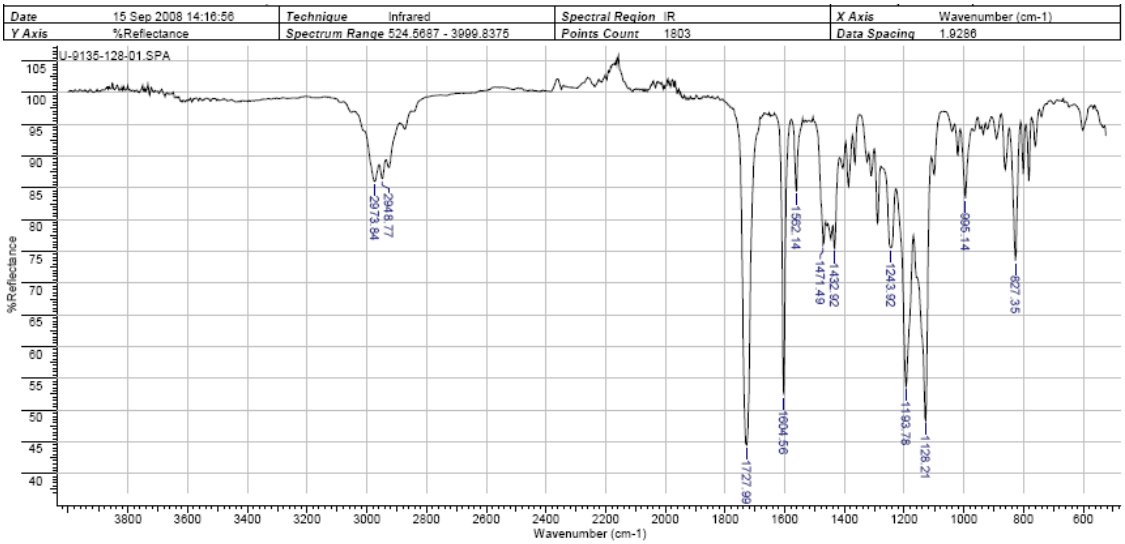


^{13} C NMR



13C NMR (101 MHz, DICHLOROMETHANE-d_2) δ ppm 21.23 (s, 1 C) 25.51 (s, 1 C) 43.46 (s, 1 C) 48.54 (s, 1 C) 51.99 (s, 1 C) 122.84 (s, 1 C) 125.46 (s, 1 C) 147.51 (s, 1 C) 149.13 (s, 1 C) 158.86 (s, 1 C) 178.17 (s, 1 C)

FT-IR

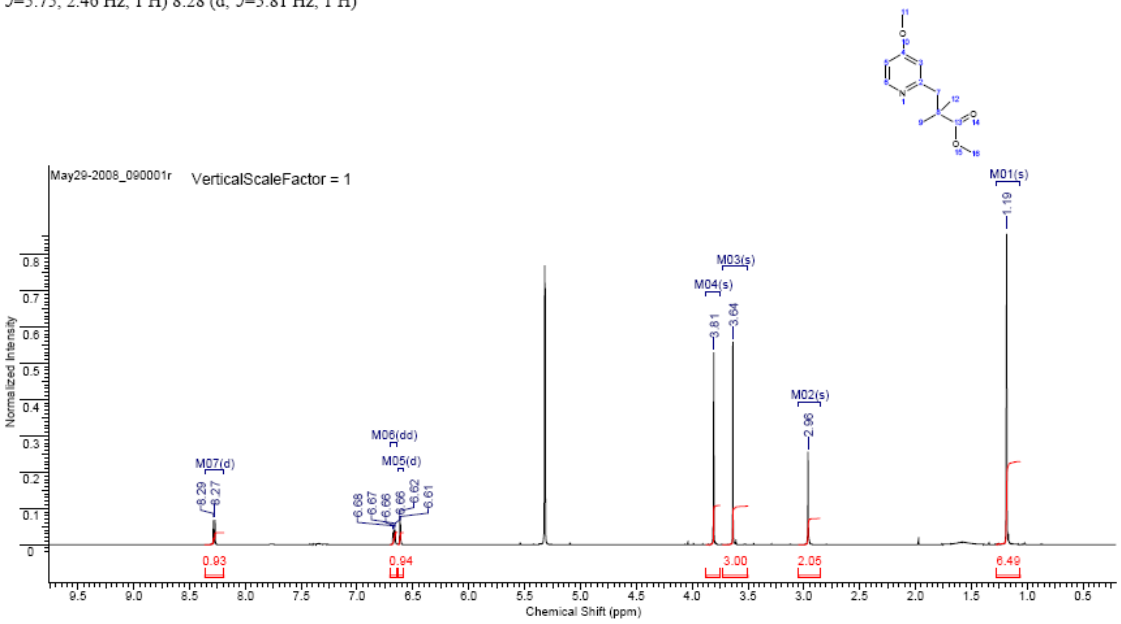


Compound 4

^{1}H NMR

SW(cyclical) (Hz)	8278.15	*Solvent*	DICHLOROMETHANE-d2	*Spectrum Offset (Hz)*	2427.7412
Sweep Width (Hz)	8278.02	*Temperature (degree C)*	27.000		

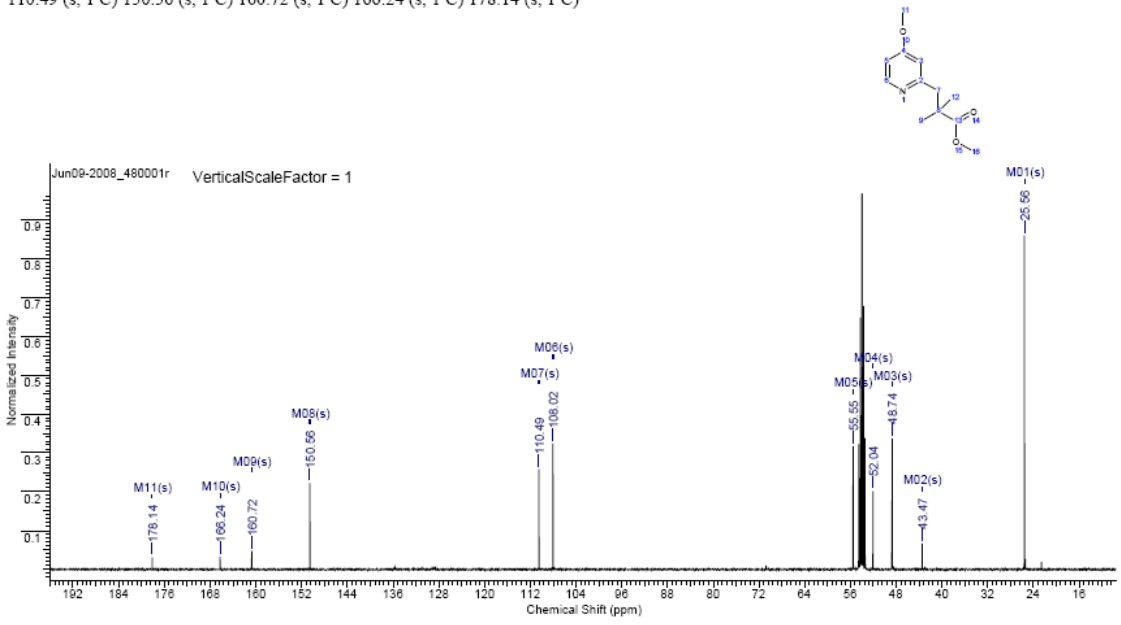
1H NMR (400 MHz, DICHLOROMETHANE-d_2) δ ppm 1.19 (s, 6 H) 2.96 (s, 2 H) 3.64 (s, 3 H) 3.81 (s, 3 H) 6.61 (d, *J*=2.53 Hz, 1 H) 6.67 (dd, *J*=5.75, 2.46 Hz, 1 H) 8.28 (d, *J*=5.81 Hz, 1 H)



^{13}C NMR

SW(cyclical) (Hz)	23980.81	*Solvent*	DICHLOROMETHANE-d2	*Spectrum Offset (Hz)*	10110.8027
Sweep Width (Hz)	23980.45	*Temperature (degree C)*	27.000		

13C NMR (101 MHz, DICHLOROMETHANE-d_2) δ ppm 25.56 (s, 1 C) 43.47 (s, 1 C) 48.74 (s, 1 C) 52.04 (s, 1 C) 55.55 (s, 1 C) 108.02 (s, 1 C) 110.49 (s, 1 C) 150.56 (s, 1 C) 160.72 (s, 1 C) 166.24 (s, 1 C) 178.14 (s, 1 C)



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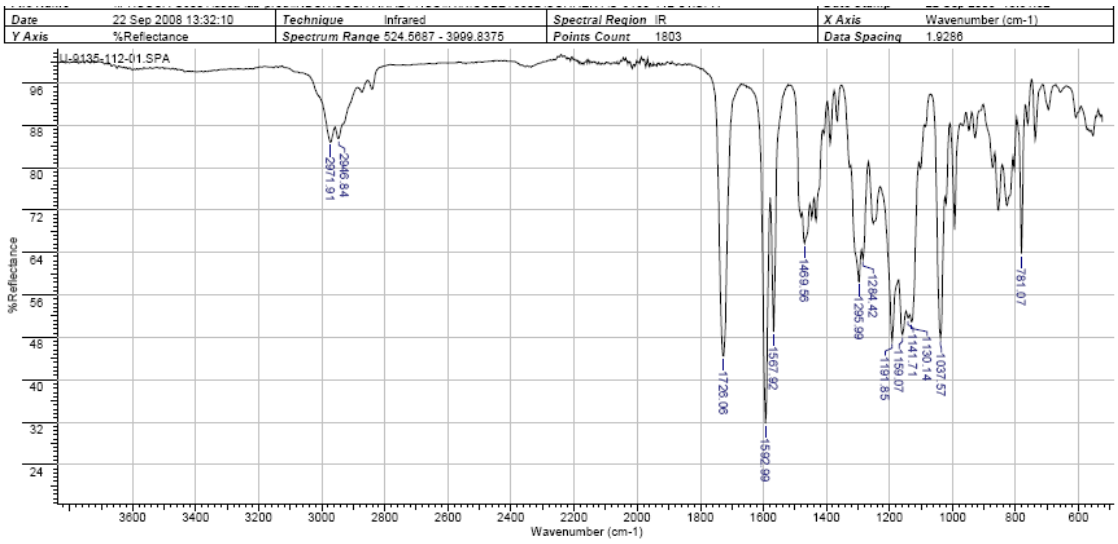
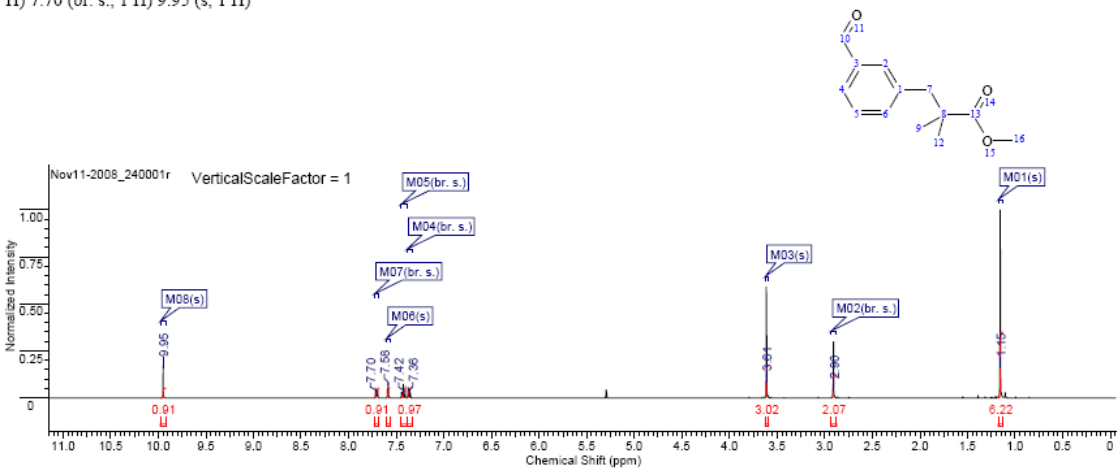


Compound 5

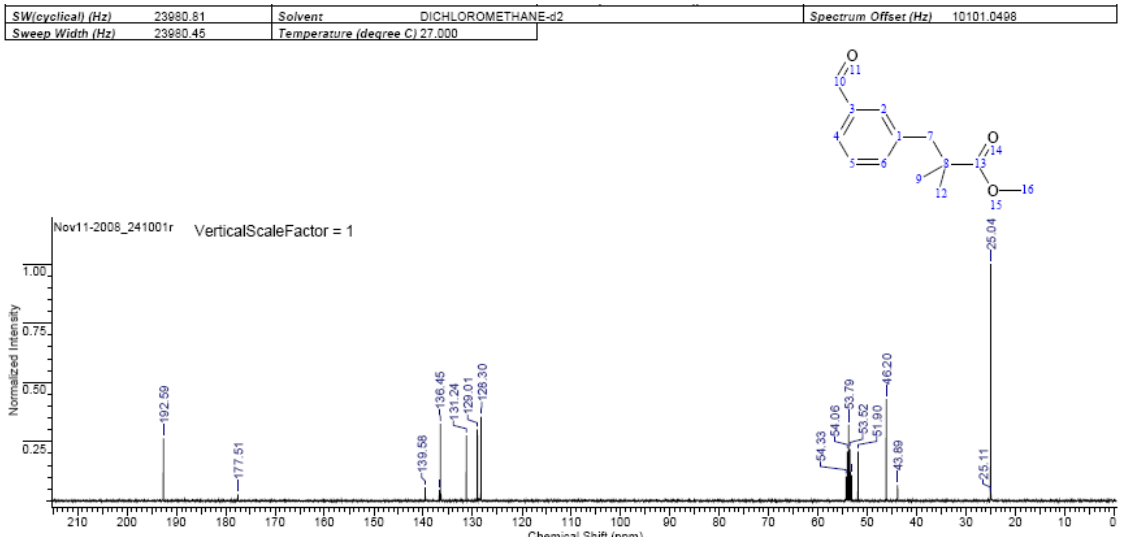
^{1}H NMR

SW(cyclical) (Hz)	8278.15	Solvent	DICHLOROMETHANE-d2	Spectrum Offset (Hz)	2470.9668
Sweep Width (Hz)	8278.02	Temperature (degree C)	27.000		

1H NMR (400 MHz, DICHLOROMETHANE-d_2) δ ppm 1.15 (s, 6 H) 2.90 (br. s., 2 H) 3.61 (s, 3 H) 7.36 (br. s., 1 H) 7.42 (br. s., 1 H) 7.58 (s, 1 H) 7.70 (br. s., 1 H) 9.95 (s, 1 H)



^{13}C NMR



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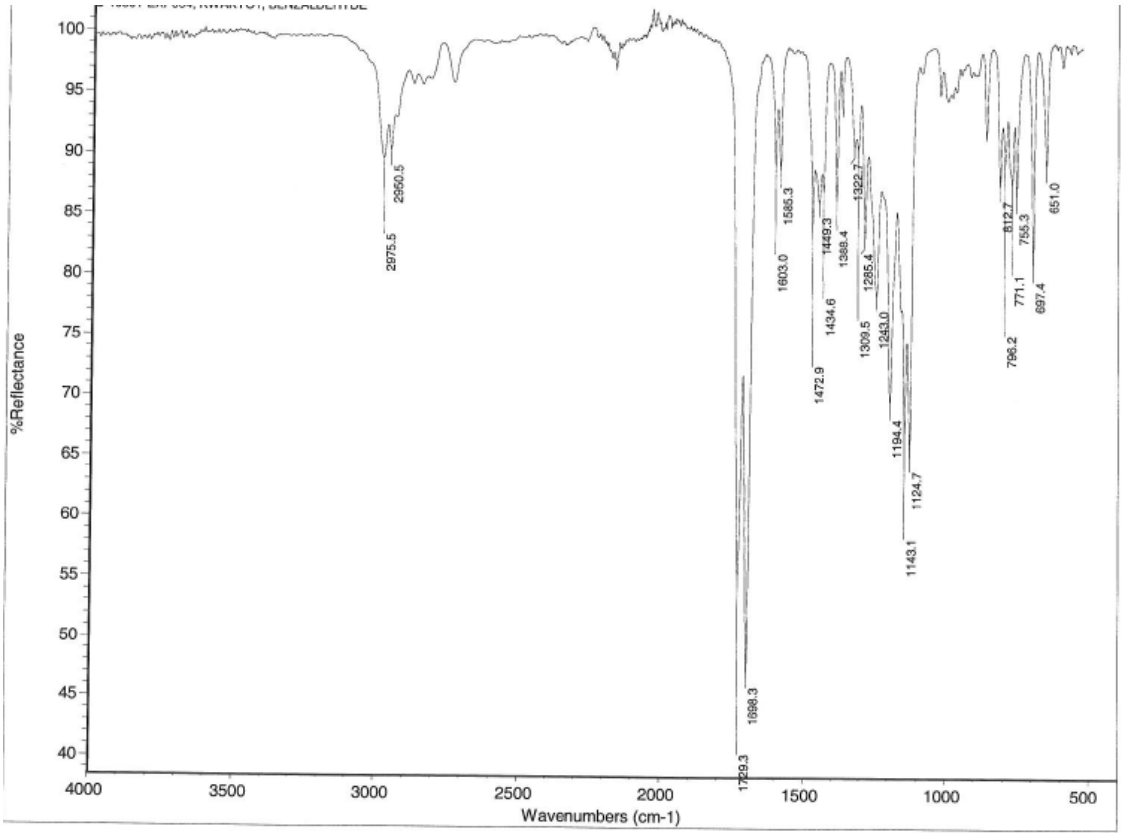
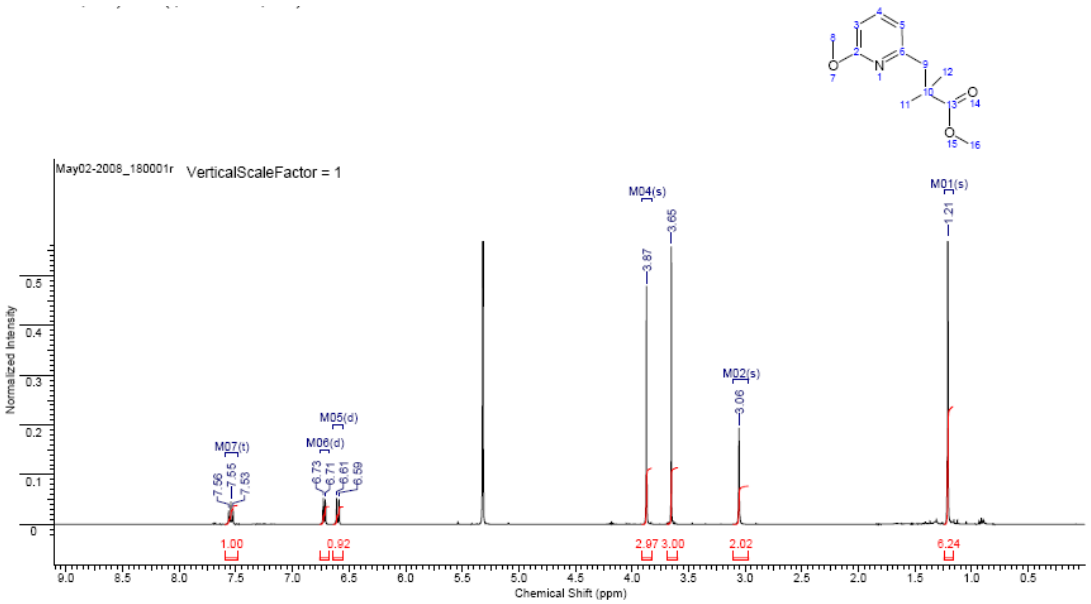


Entry 1

^{1}H NMR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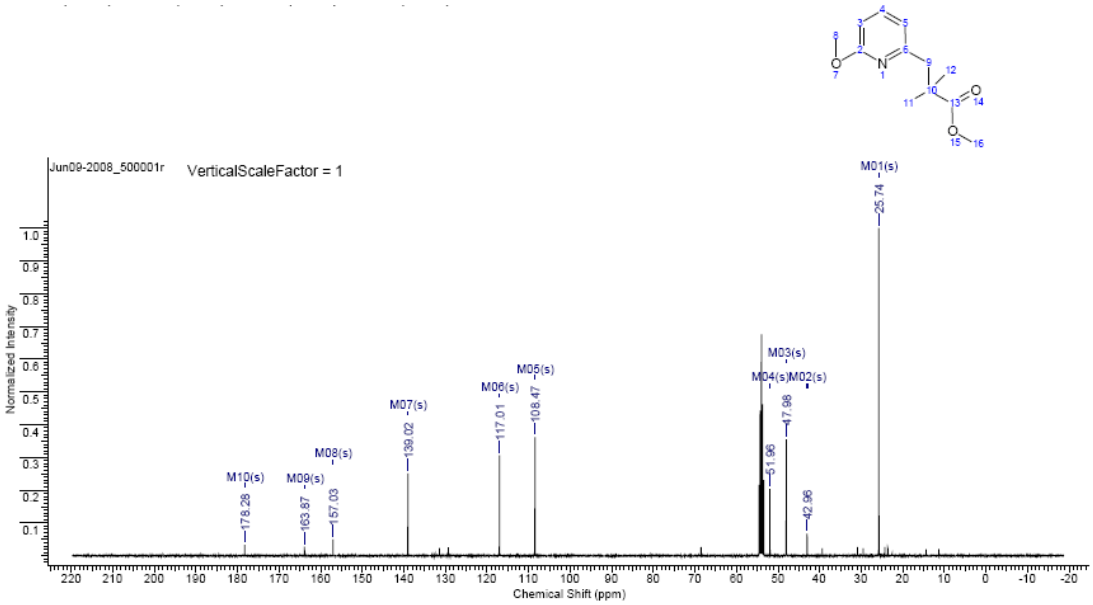
Normalized Intensity
Chemical Shift (ppm)

^{13}C NMR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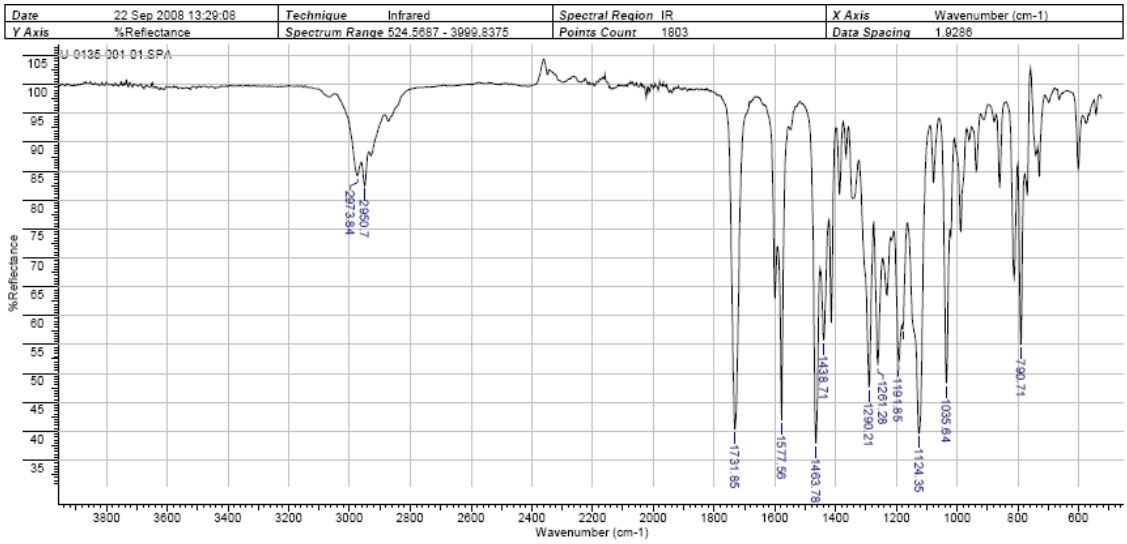
Normalized Intensity
Chemical Shift (ppm)

FT-IR

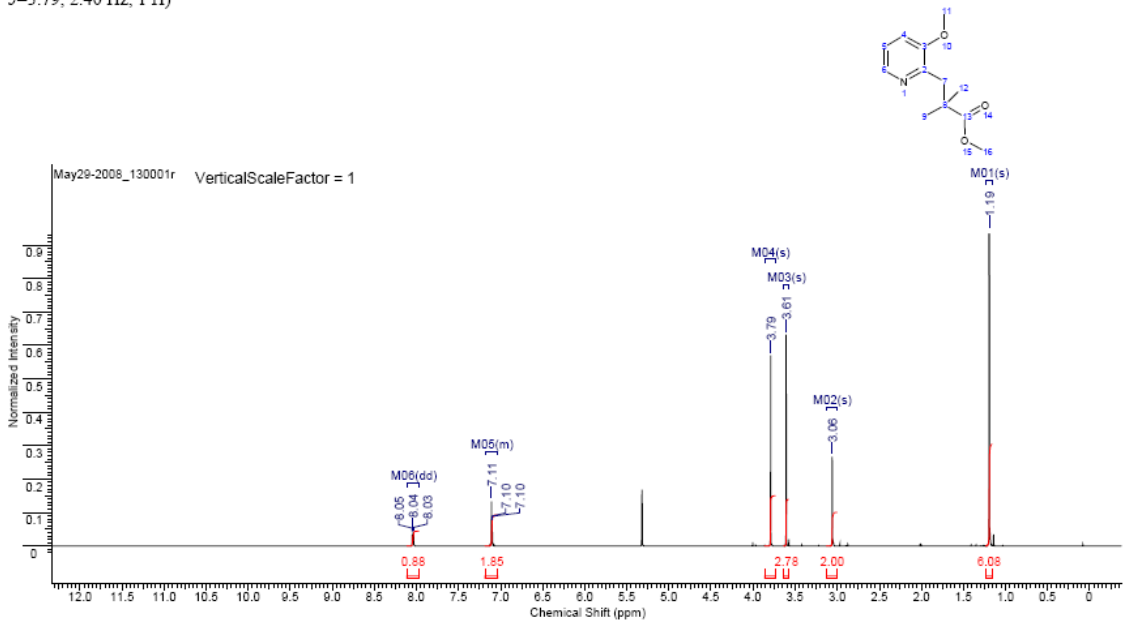


Entry 2

^{1}H NMR

SW(cyclical) (Hz)	8278.15	Solvent	DICHLOROMETHANE-d2	Spectrum Offset (Hz)	2455.7830
Sweep Width (Hz)	8278.02	Temperature (degree C)	27.000		

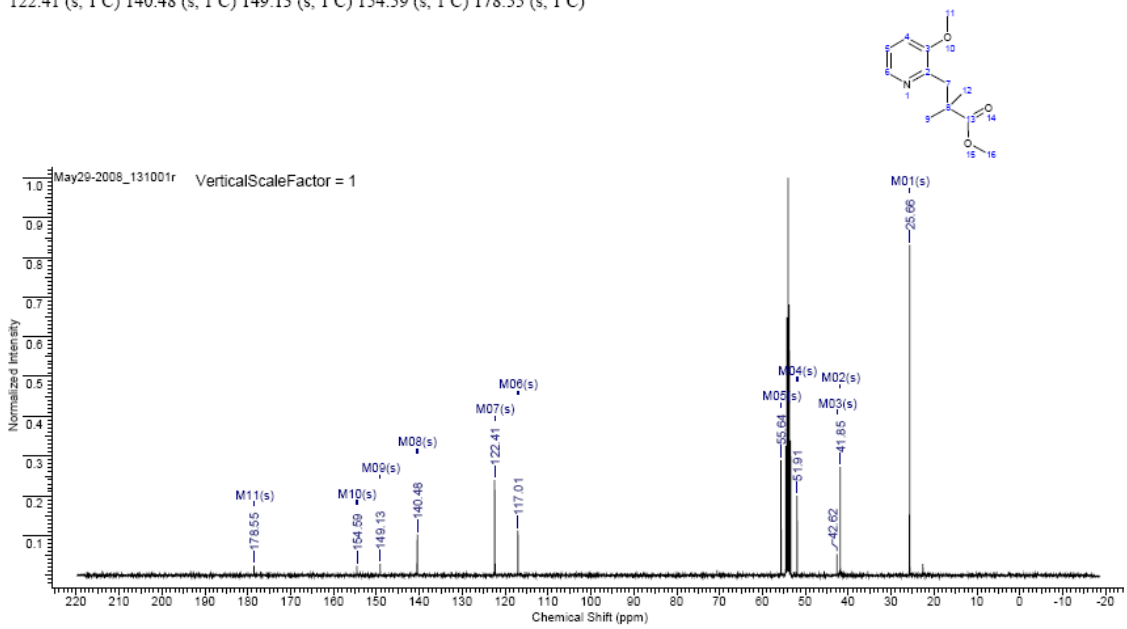
1H NMR (400 MHz, DICHLOROMETHANE-d_2) δ ppm 1.19 (s, 6 H) 3.06 (s, 2 H) 3.61 (s, 3 H) 3.79 (s, 3 H) 7.03 - 7.19 (m, 2 H) 8.04 (dd, *J*=3.79, 2.40 Hz, 1 H)



^{13}C NMR

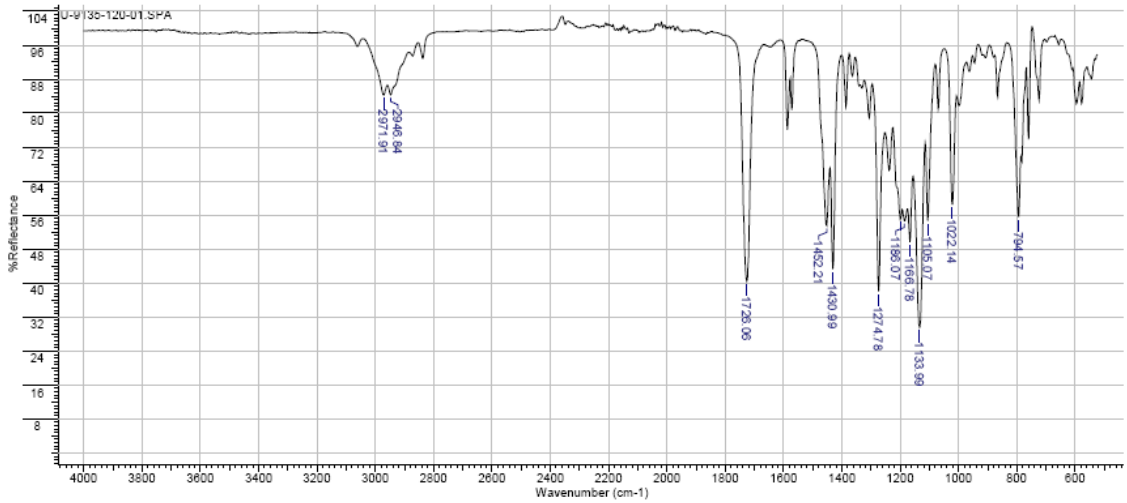
SW(cyclical) (Hz)	23980.81	*Solvent*	DICHLOROMETHANE-d2	*Spectrum Offset (Hz)*	10116.6572
Sweep Width (Hz)	23980.45	*Temperature (degree C)*	27.000		

13C NMR (101 MHz, DICHLOROMETHANE-d_2) δ ppm 25.66 (s, 1 C) 41.85 (s, 1 C) 42.62 (s, 1 C) 51.91 (s, 1 C) 55.64 (s, 1 C) 117.01 (s, 1 C) 122.41 (s, 1 C) 140.48 (s, 1 C) 149.13 (s, 1 C) 154.59 (s, 1 C) 178.55 (s, 1 C)



FT-IR

Date	22 Sep 2008 13:05:10	*Technique*	Infrared	*Spectral Region*	IR	*X Axis*	Wavenumber (cm-1)
Y Axis	%Reflectance	*Spectrum Range*	524.5687 - 3999.8375	*Points Count*	1803	*Data Spacing*	1.9286

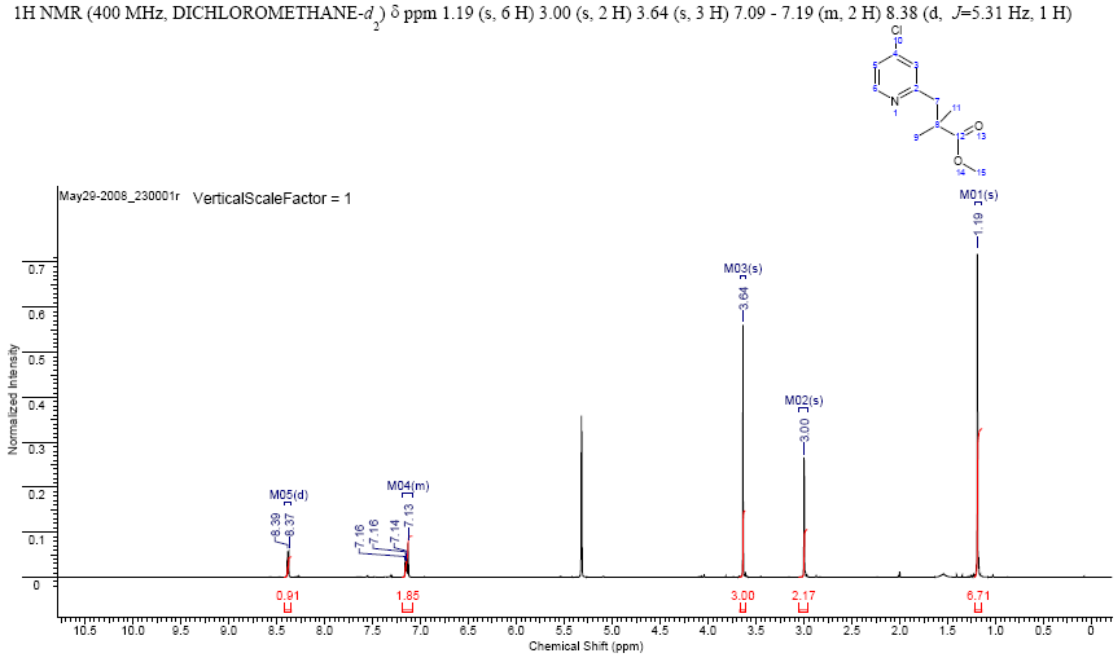


Entry 3

^{1}H NMR

SW(cyclical) (Hz)	8278.15	*Solvent*	DICHLOROMETHANE-d2	*Spectrum Offset (Hz)*	2440.2466
Sweep Width (Hz)	8278.02	*Temperature (degree C)*	27.000		

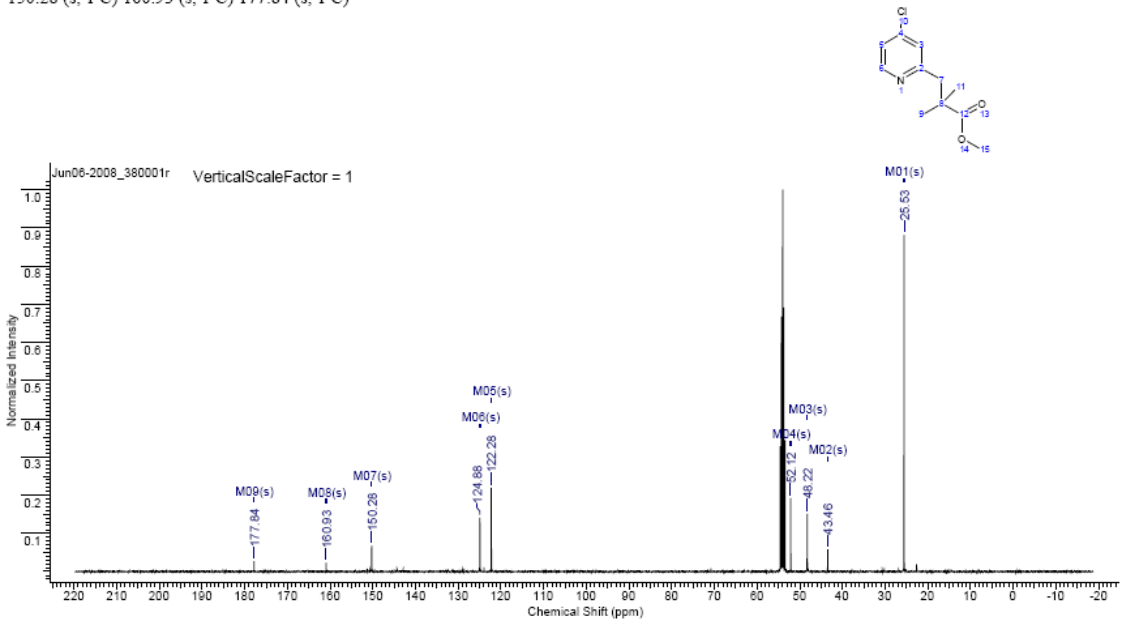
1H NMR (400 MHz, DICHLOROMETHANE-d_2) δ ppm 1.19 (s, 6 H) 3.00 (s, 2 H) 3.64 (s, 3 H) 7.09 - 7.19 (m, 2 H) 8.38 (d, *J*=5.31 Hz, 1 H)



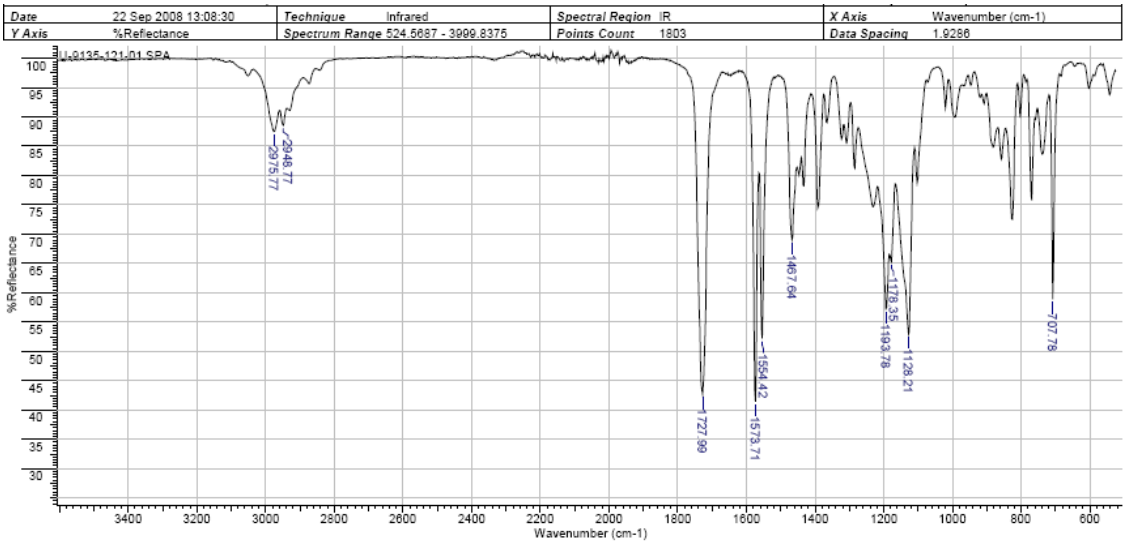
^{13}C NMR

SW(cyclical) (Hz)	23980.81	*Solvent*	DICHLOROMETHANE-d2	*Spectrum Offset (Hz)*	10111.1689
Sweep Width (Hz)	23980.45	*Temperature (degree C)*	27.000		

13C NMR (101 MHz, DICHLOROMETHANE-d_2) δ ppm 25.53 (s, 1 C) 43.46 (s, 1 C) 48.22 (s, 1 C) 52.12 (s, 1 C) 122.28 (s, 1 C) 124.88 (s, 1 C) 150.28 (s, 1 C) 160.93 (s, 1 C) 177.84 (s, 1 C)

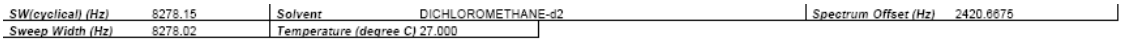


FT-IR

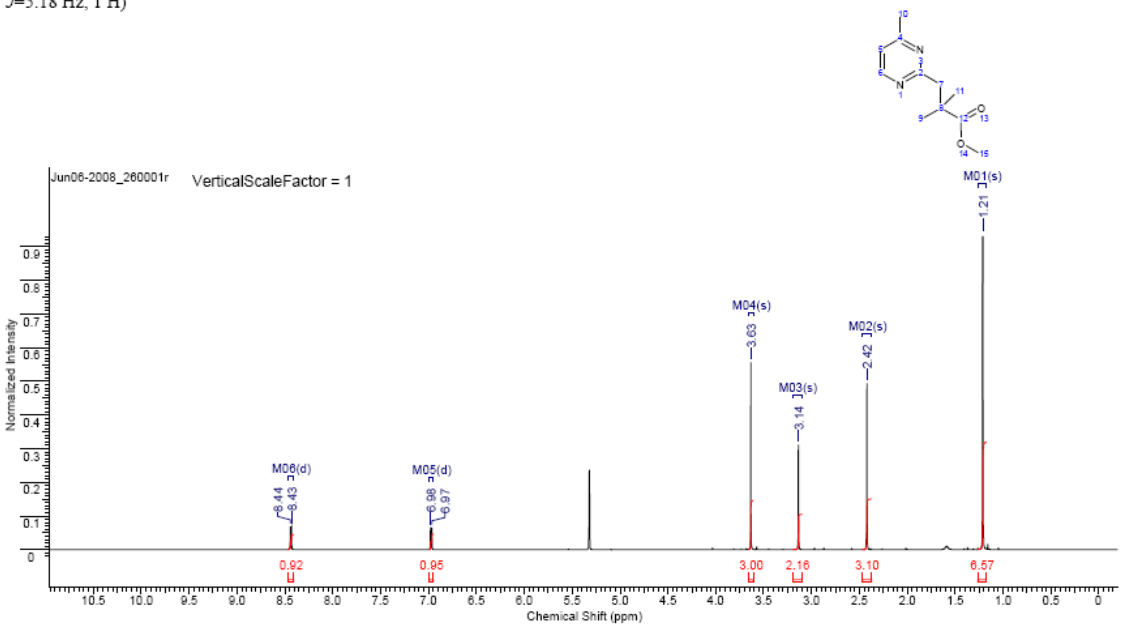


Entry 5

^{1}H NMR



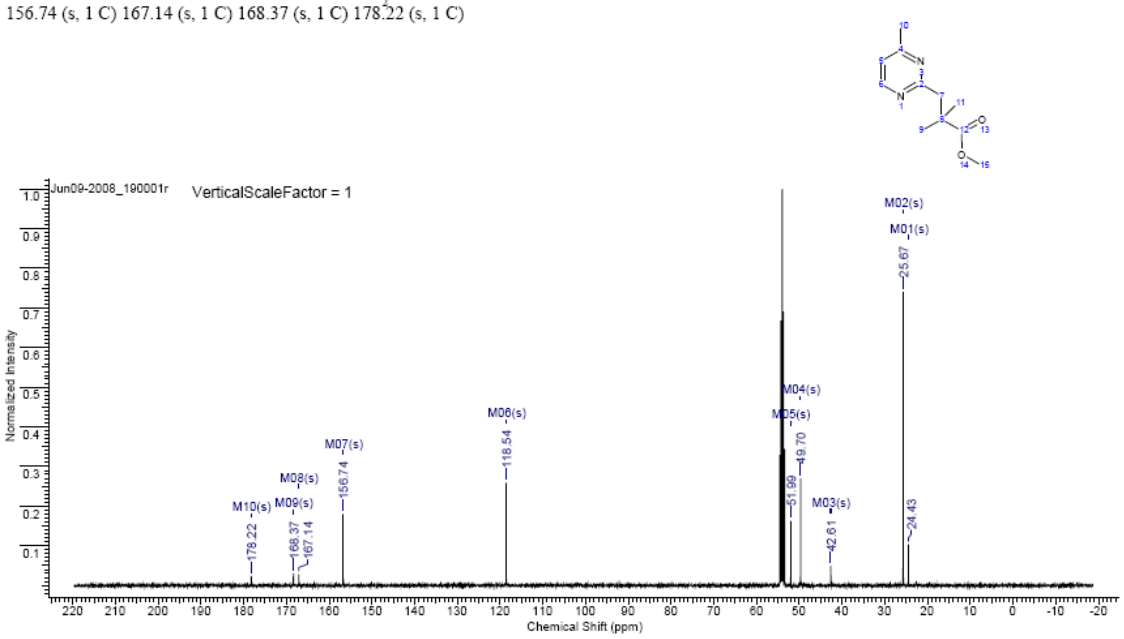
1H NMR (400 MHz, DICHLOROMETHANE-d_2) δ ppm 1.21 (s, 6 H) 2.42 (s, 3 H) 3.14 (s, 2 H) 3.63 (s, 3 H) 6.97 (d, *J*=5.05 Hz, 1 H) 8.44 (d, *J*=5.18 Hz, 1 H)



^{13}C NMR

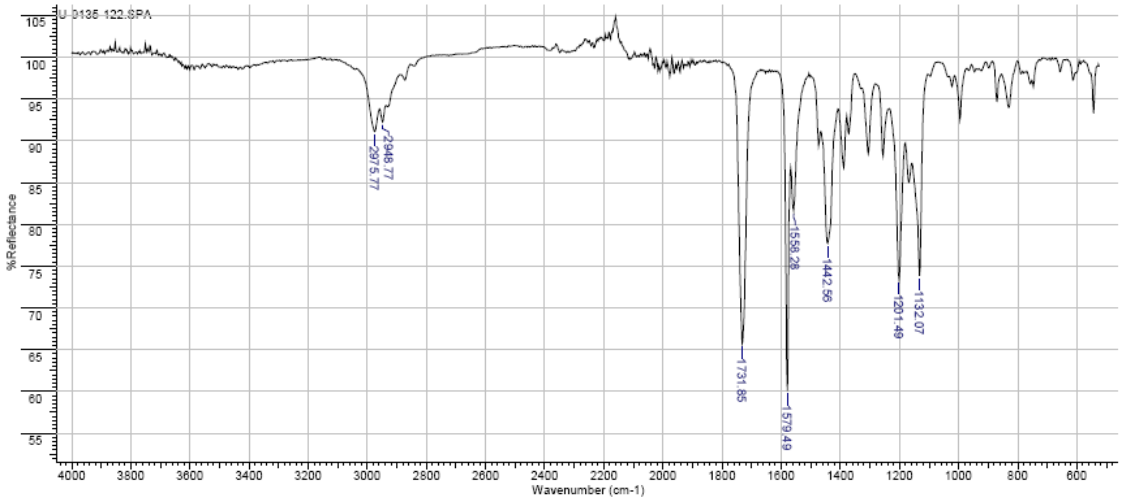
SW(cyclical) (Hz)	23980.81	*Solvent*	DICHLOROMETHANE-d2	*Spectrum Offset (Hz)*	10110.0713
Sweep Width (Hz)	23980.45	*Temperature (degree C)*	27.000		

13C NMR (101 MHz, DICHLOROMETHANE-d_2) δ ppm 24.43 (s, 1 C) 25.67 (s, 1 C) 42.61 (s, 1 C) 49.70 (s, 1 C) 51.99 (s, 1 C) 118.54 (s, 1 C) 156.74 (s, 1 C) 167.14 (s, 1 C) 168.37 (s, 1 C) 178.22 (s, 1 C)



FT-IR

Date	15 Sep 2008 14:28:44	*Technique*	Infrared	*Spectral Region*	IR	*X Axis*	Wavenumber (cm-1)
Y Axis	%Reflectance	*Spectrum Range*	524.5687 - 3999.8375	*Points Count*	1803	*Data Spacing*	1.9286

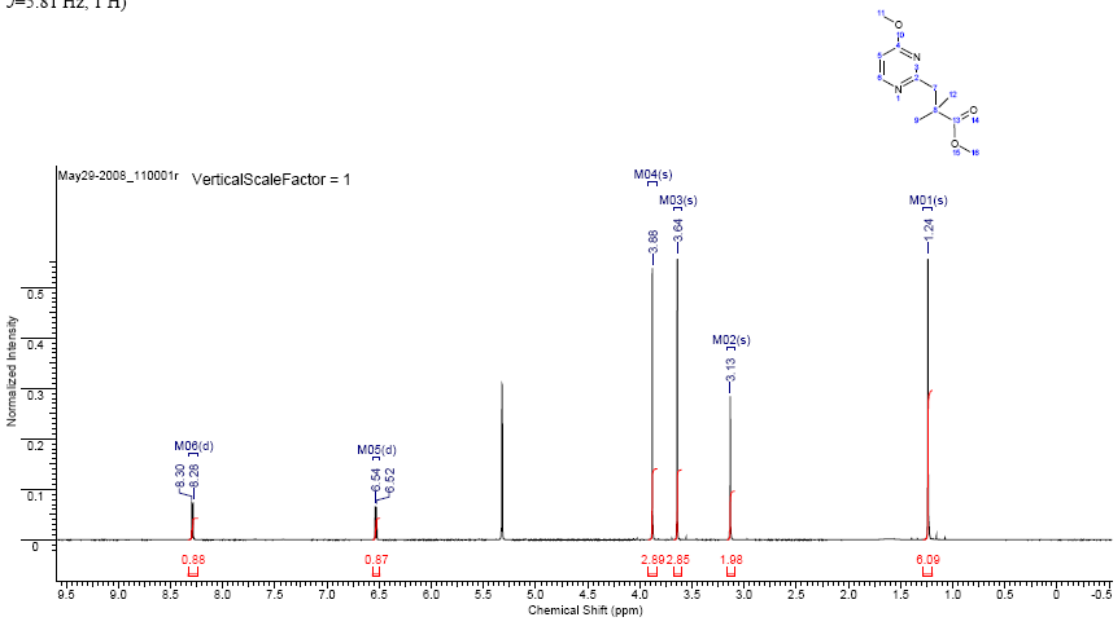


Entry 6

^{1}H NMR

SW(cyclical) (Hz)	8278.15	*Solvent*	DICHLOROMETHANE-d2	*Spectrum Offset (Hz)*	2333.3843
Sweep Width (Hz)	8278.02	*Temperature (degree C)*	27.000		

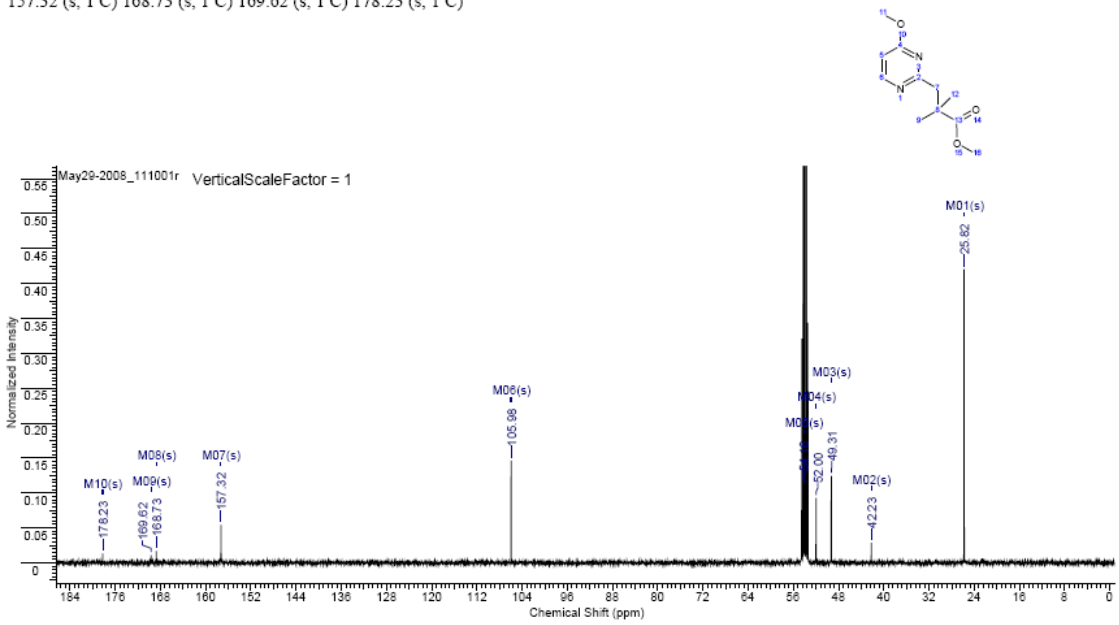
1H NMR (400 MHz, DICHLOROMETHANE-d_2) δ ppm 1.24 (s, 6 H) 3.13 (s, 2 H) 3.64 (s, 3 H) 3.88 (s, 3 H) 6.53 (d, *J*=5.81 Hz, 1 H) 8.29 (d, *J*=5.81 Hz, 1 H)



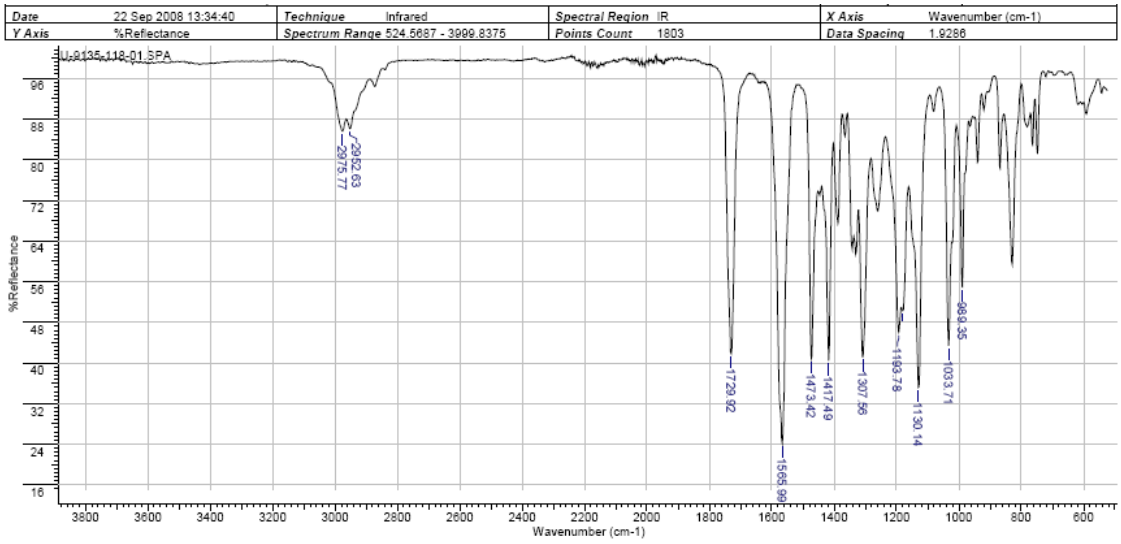
^{13}C NMR

SW(cyclical) (Hz)	23980.81	*Solvent*	DICHLOROMETHANE-d2	*Spectrum Offset (Hz)*	10109.3389
Sweep Width (Hz)	23980.45	*Temperature (degree C)*	27.000		

13C NMR (101 MHz, DICHLOROMETHANE-d_2) δ ppm 25.82 (s, 1 C) 42.23 (s, 1 C) 49.31 (s, 1 C) 52.00 (s, 1 C) 54.16 (s, 1 C) 105.98 (s, 1 C) 157.32 (s, 1 C) 168.73 (s, 1 C) 169.62 (s, 1 C) 178.23 (s, 1 C)

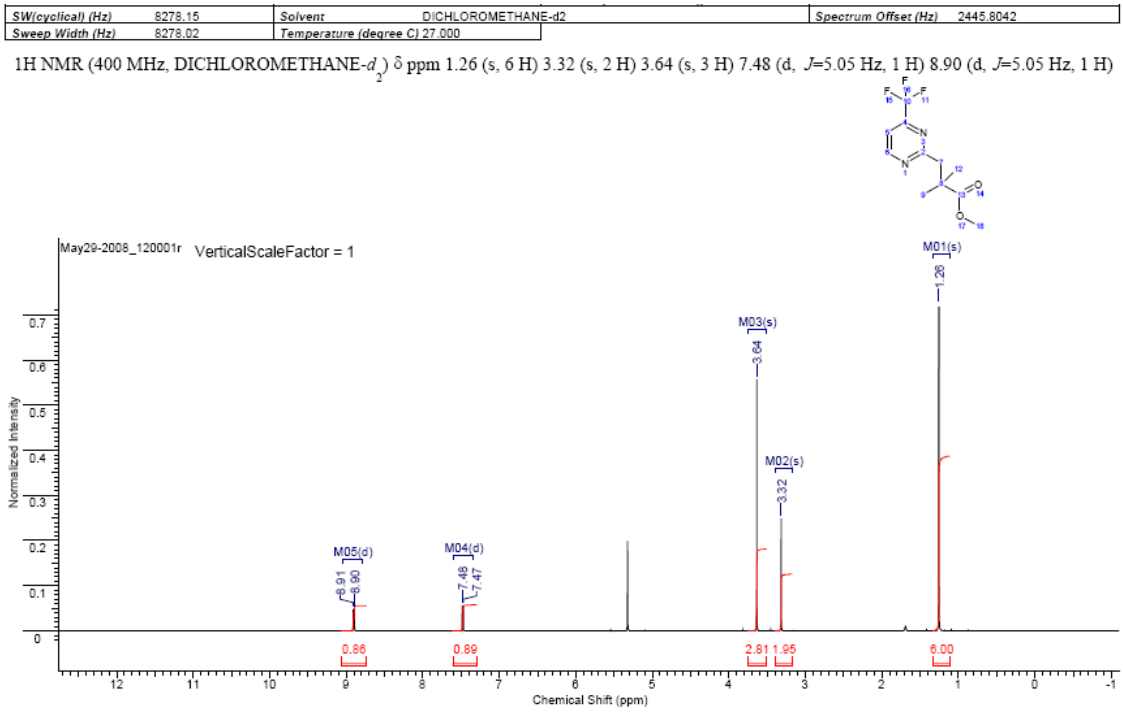


FT-IR



Entry 7

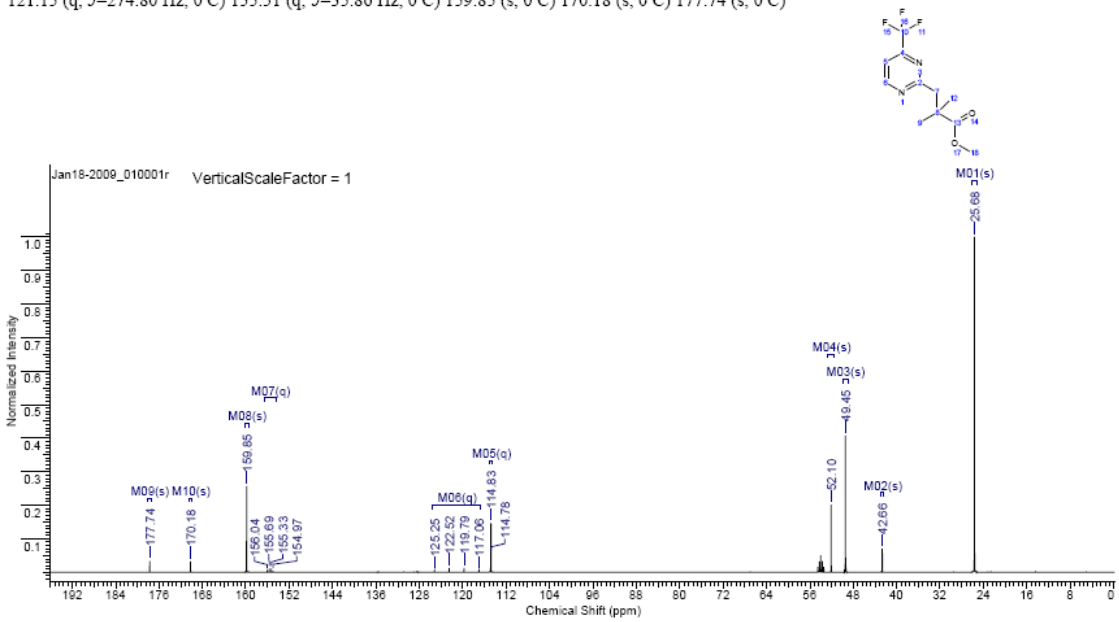
^{1}H NMR



^{13}C NMR

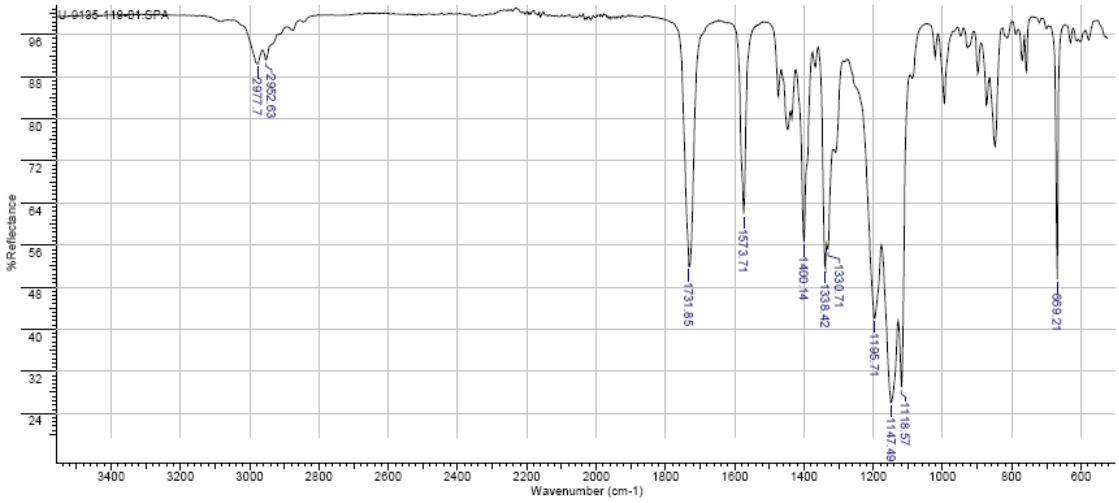
SW(cyclical) (Hz)	23980.81	Solvent	DICHLOROMETHANE-d2	Spectrum Offset (Hz)	10122.5127
Sweep Width (Hz)	23980.45	Temperature (degree C)	27.000		

13C NMR (101 MHz, DICHLOROMETHANE-d_2) δ ppm 25.68 (s, 1 C) 42.66 (s, 0 C) 49.45 (s, 0 C) 52.10 (s, 0 C) 114.82 (q, J=2.56 Hz, 0 C) 121.15 (q, J=274.80 Hz, 0 C) 155.51 (q, J=35.86 Hz, 0 C) 159.85 (s, 0 C) 170.18 (s, 0 C) 177.74 (s, 0 C)



FT-IR

Date	22 Sep 2008 13:00:20	Technique	Infrared	Spectral Region	IR
Y Axis	%Reflectance	Spectrum Range	524.5687 - 3999.8375	Points Count	1803
X Axis	Wavenumber (cm-1)	Data Spacing	1.9286		

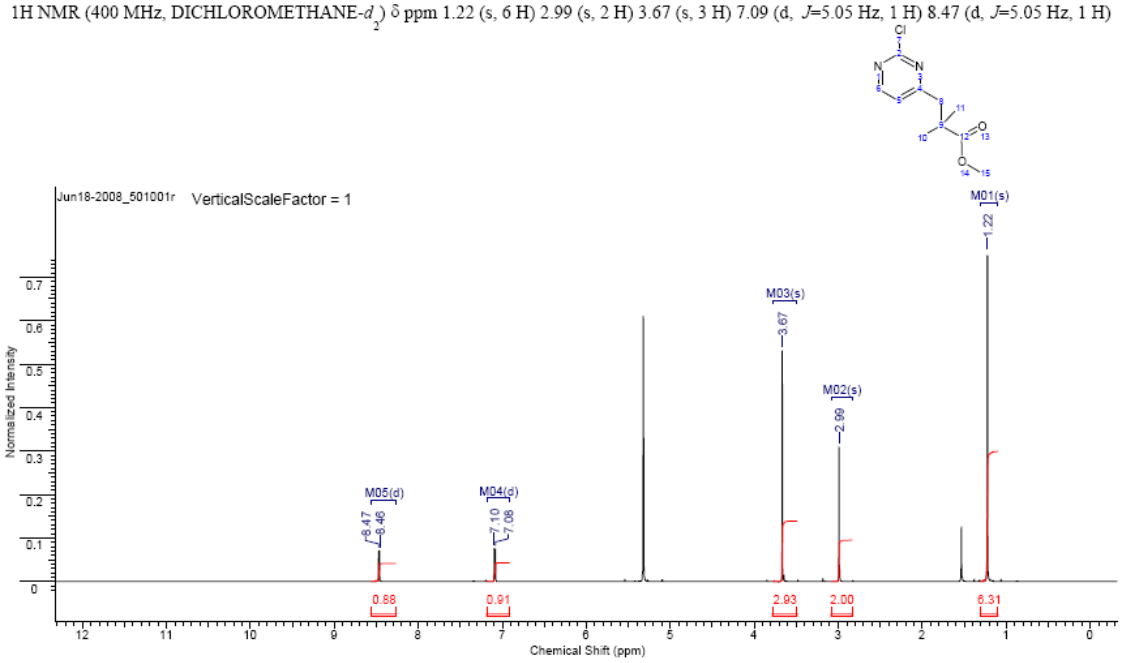


Entry 8

^{1}H NMR

SW(cyclical) (Hz)	8278.15	*Solvent*	DICHLOROMETHANE-d2	*Spectrum Offset (Hz)*	2415.1096
Sweep Width (Hz)	8278.02	*Temperature (degree C)*	27.000		

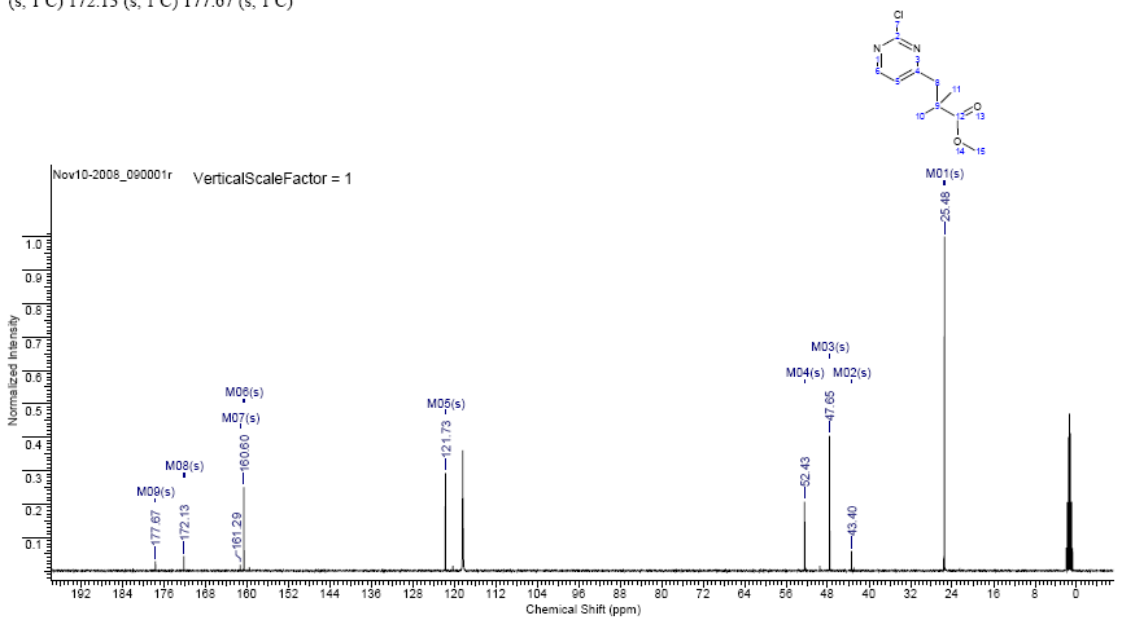
1H NMR (400 MHz, DICHLOROMETHANE-d_2) δ ppm 1.22 (s, 6 H) 2.99 (s, 2 H) 3.67 (s, 3 H) 7.09 (d, *J*=5.05 Hz, 1 H) 8.47 (d, *J*=5.05 Hz, 1 H)



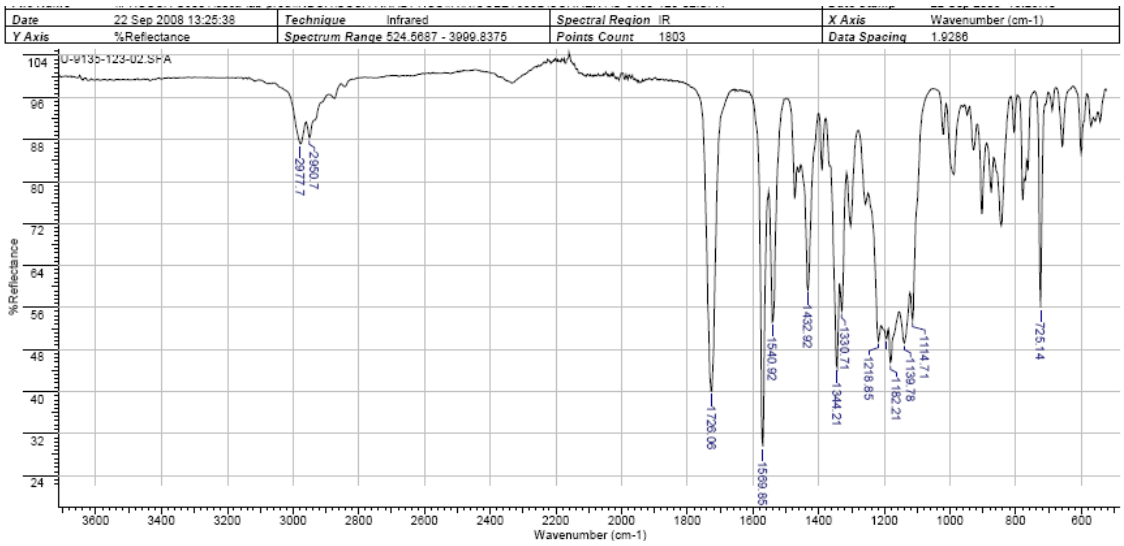
^{13}C NMR

SW(cyclical) (Hz)	23980.81	*Solvent*	ACETONITRILE-d3	*Spectrum Offset (Hz)*	10157.7773	*Sweep Width (Hz)*	23980.45
Temperature (degree C)	27.000						

13C NMR (101 MHz, ACETONITRILE-d_3) δ ppm 25.48 (s, 1 C) 43.40 (s, 1 C) 47.65 (s, 1 C) 52.43 (s, 1 C) 121.73 (s, 1 C) 160.60 (s, 1 C) 161.29 (s, 1 C) 172.13 (s, 1 C) 177.67 (s, 1 C)



FT-IR

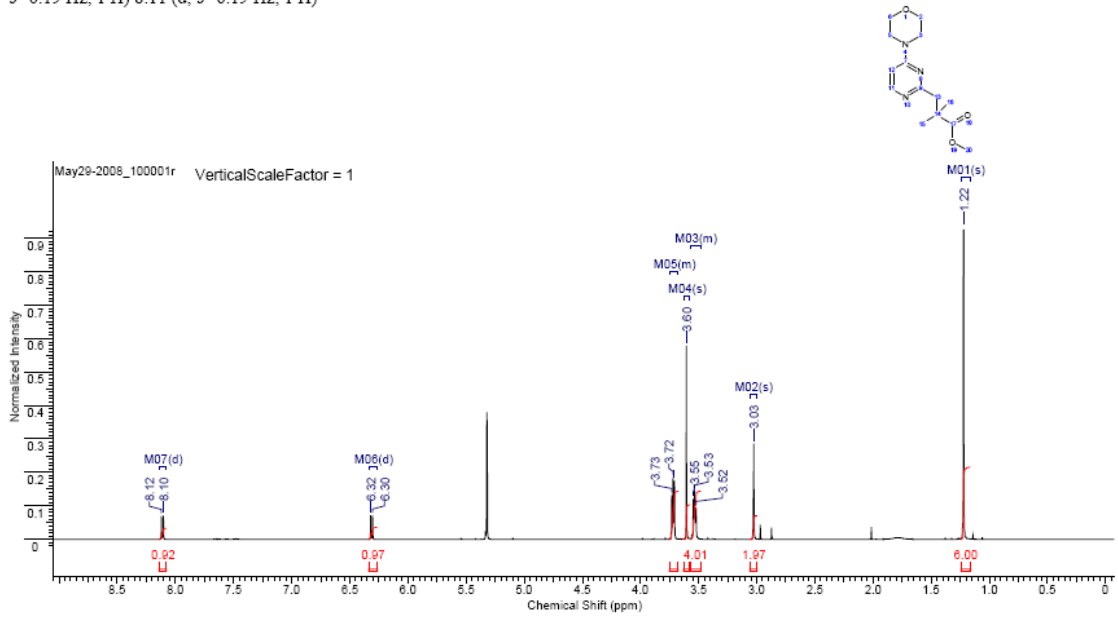


Entry 9

^{1}H NMR

SW(cyclical) (Hz)	8278.15	Solvent	DICHLOROMETHANE-d2	Spectrum Offset (Hz)	2434.0569
Sweep Width (Hz)	8278.02	Temperature (degree C)	27.000		

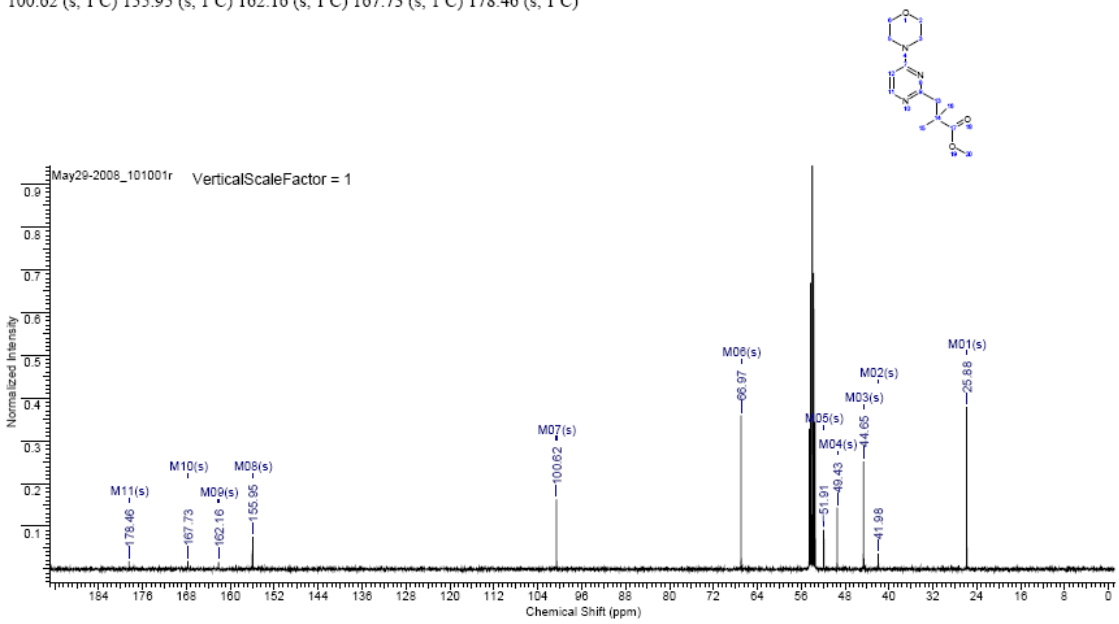
1H NMR (400 MHz, DICHLOROMETHANE-d_2) δ ppm 1.22 (s, 6 H) 3.03 (s, 2 H) 3.48 - 3.57 (m, 4 H) 3.60 (s, 3 H) 3.68 - 3.75 (m, 4 H) 6.31 (d, *J*=6.19 Hz, 1 H) 8.11 (d, *J*=6.19 Hz, 1 H)



^{13}C NMR

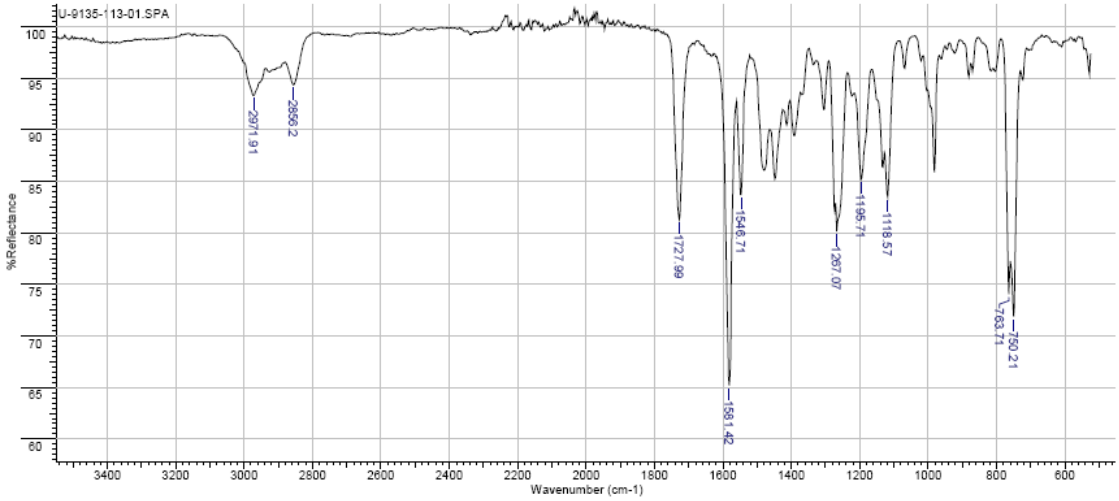
SW(cyclical) (Hz)	23980.81	*Solvent*	DICHLOROMETHANE-d2	*Spectrum Offset (Hz)*	10109.3389
Sweep Width (Hz)	23980.45	*Temperature (degree C)*	27.000		

13C NMR (101 MHz, DICHLOROMETHANE-d_2) δ ppm 25.88 (s, 1 C) 41.98 (s, 1 C) 44.65 (s, 1 C) 49.43 (s, 1 C) 51.91 (s, 1 C) 66.97 (s, 1 C) 100.62 (s, 1 C) 155.95 (s, 1 C) 162.16 (s, 1 C) 167.73 (s, 1 C) 178.46 (s, 1 C)



FT-IR

Date	22 Sep 2008 13:19:26	*Technique*	Infrared	*Spectral Region*	IR
Y Axis	%Reflectance	*Spectrum Range*	524.5687 - 3999.8375	*Points Count*	1803
X Axis	Wavenumber (cm-1)	*Data Spacing*	1.9286		

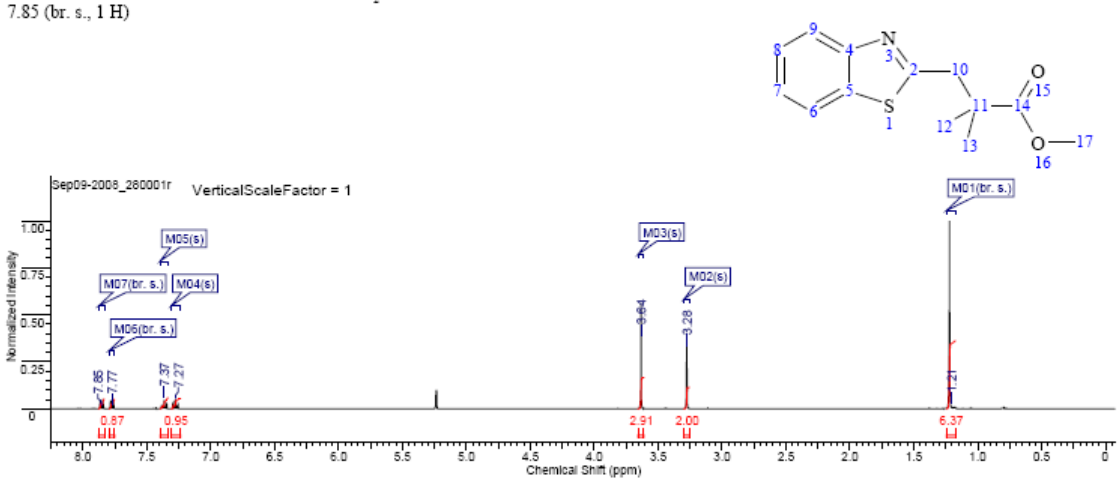


Entry 10

^{1}H NMR

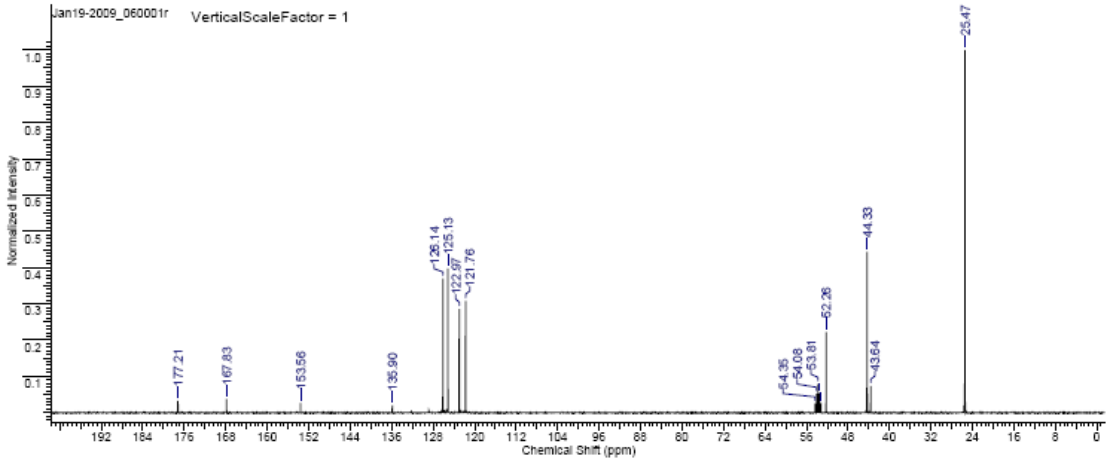
SW(cyclical) (Hz)	8278.15	Solvent	DICHLOROMETHANE-d2	Spectrum Offset (Hz)	2462.6101
Sweep Width (Hz)	8278.02	Temperature (degree C)	27.000		

1H NMR (400 MHz, DICHLOROMETHANE-d_2) δ ppm 1.21 (br. s., 6 H) 3.28 (s, 2 H) 3.64 (s, 3 H) 7.27 (s, 1 H) 7.37 (s, 1 H) 7.77 (br. s., 1 H) 7.85 (br. s., 1 H)

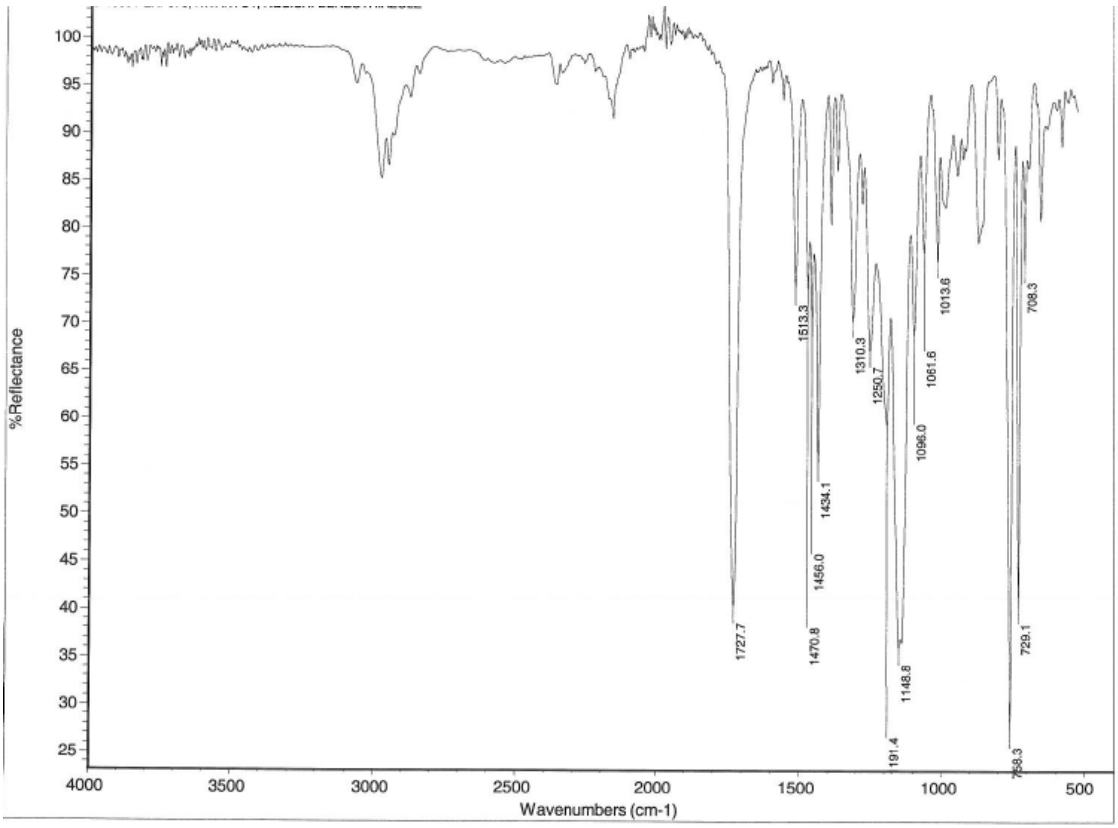


^{13}C NMR

SW(cyclical) (Hz)	23980.81	Solvent	DICHLOROMETHANE-d2	Spectrum Offset (Hz)	10101.0498
Sweep Width (Hz)	23980.45	Temperature (degree C)	27.000		



FT-IR

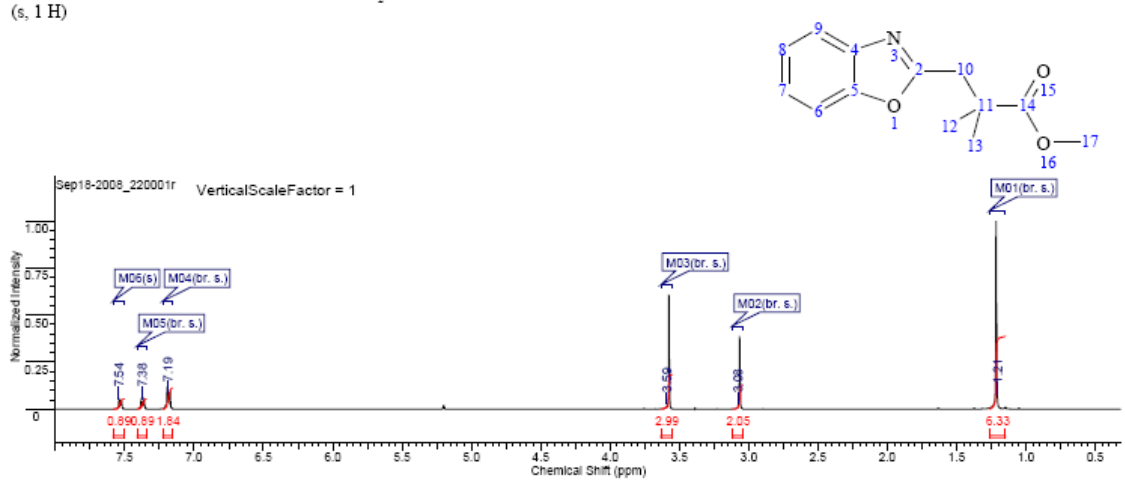


Entry 11

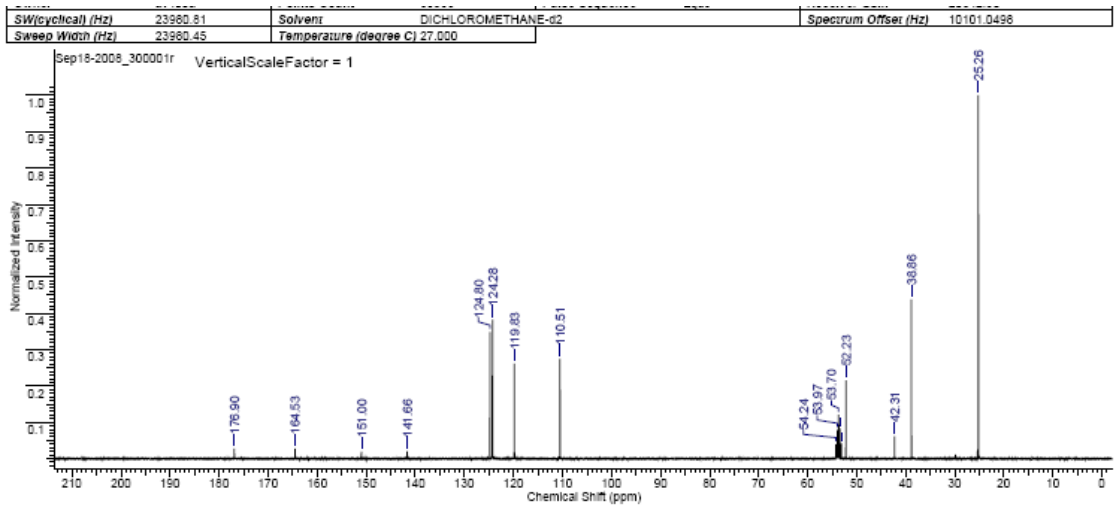
^{1}H NMR

SW(cyclical) (Hz)	8278.15	Solvent	DICHLOROMETHANE-d2	Spectrum Offset (Hz)	2470.9668
Sweep Width (Hz)	8278.02	Temperature (degree C)	27.000		

1H NMR (400 MHz, DICHLOROMETHANE-d_2) δ ppm 1.21 (br. s., 6 H) 3.08 (br. s., 2 H) 3.59 (br. s., 3 H) 7.19 (br. s., 2 H) 7.38 (br. s., 1 H) 7.54 (s, 1 H)



^{13}C NMR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Sweep Width (Hz) 23980.45
Solvent DICHLOROMETHANE-d2
Temperature (degree C) 27.000
Spectrum Offset (Hz) 10101.0498
Sep18-2008_300001r
VerticalScaleFactor = 1
Normalized Intensity
Chemical Shift (ppm)
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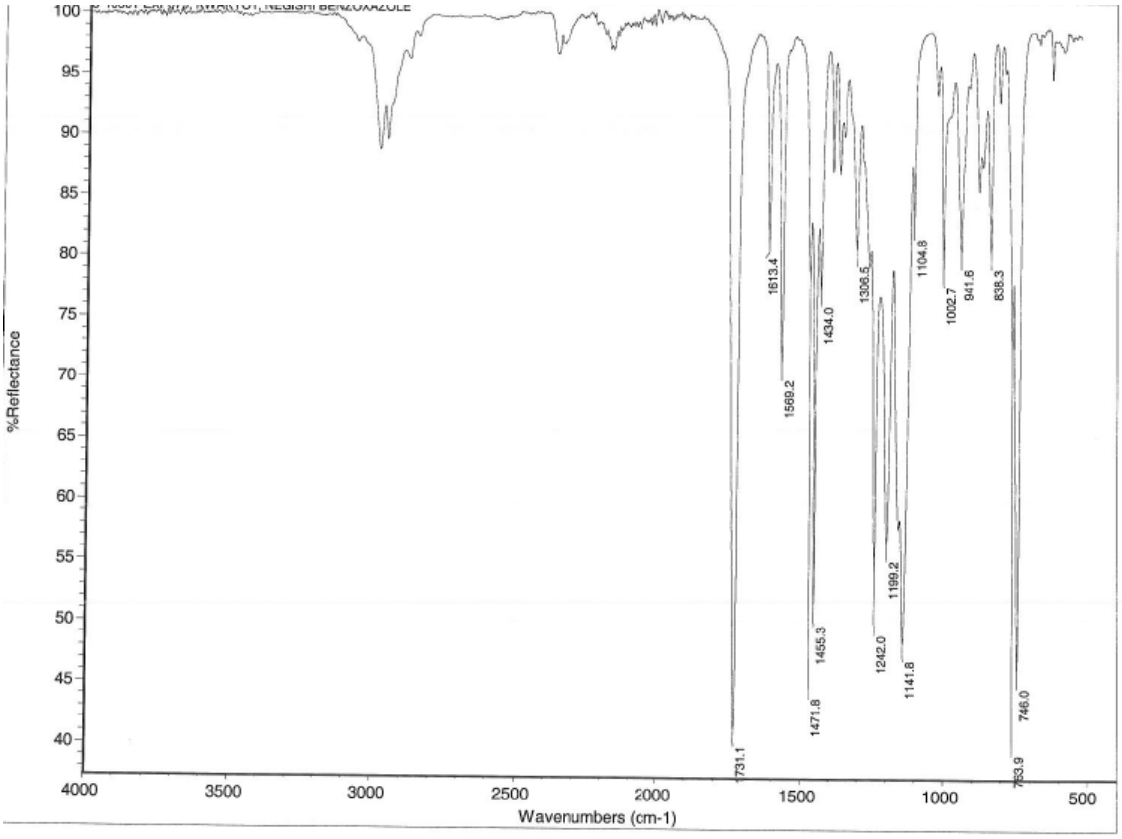
164.53
151.00
141.66
124.80
124.28
119.83
110.51
54.24
53.97
53.70
52.23
42.31
38.86
25.26

FT-IR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1306.5
1242.0
1199.2
1141.8
1104.8
1002.7
941.6
838.3
746.0
731.1
703.9