Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Iodosobenzene–Mediated Direct and Efficient Oxidation of β–Dicarbonyls to Vicinal Tricarbonyls Catalyzed by Iron(III) Salt

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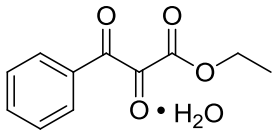
General information

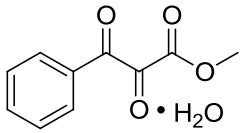
The NMR spectra were measured using Bruker AV400 and Bruker AV300 instruments with $CDCl_3$ as the solvent. IR spectra were recorded on a FT-IR Bruker EQUINOX55 spectrometer in KBr pellets. High resolution mass spectral analyses (HRMS) were performed on high resolution ESI–FTICR mass spectrometer (Varian 7.0 T). All solvents used were purified by standard methods. Petroleum ether (PE), where used, had a range of boiling point 60-90 ℃.

Typical procedure:

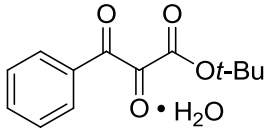


Ethyl 2,3-dioxo-3-phenylpropanoate (**2a**)

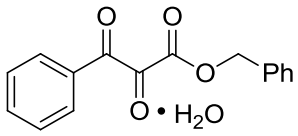
To a solution of ethyl benzoylacetate (96 mg, 0.5 mmol) in EtOAc (5 mL) in a 10 mL rounded bottom flask were added $Fe(NO_3)_3$ $9H_2O$ (40.4 mg, 20 mol%) and PhIO (165 mg, 1.5 equiv). The reaction was stirred under air at room temperature and monitored by TLC. After the completion of the reaction (3 h), the reaction mixture was diluted with EtOAc (50 mL). Then, the mixture was washed with sat. aqueous $NaHCO_3$ (5 mL) and $Na_2S_2O_3$ (5 mL). The separated aqueous phase was extracted with EtOAc (2×10 mL). The combined organic layer was washed with brine (10 mL), dried over anhydrous $MgSO_4$ and concentrated in vacuo to afford the crude product which was purified by flash column chromatograpy to give 100 mg of **2a** in 91% yield as yellowish oil. Hydrate form:keto form = 6:1. ^{1}H NMR for hydrate form (300 MHz, $CDCl_3$): δ = 1.05 (t, *J* = 7.2 Hz, 3 H), 4.20 (q, *J* = 7.2 Hz, 2 H), 5.69 (brs, 2 H), 7.46 (t, *J* = 7.5 Hz, 2 H), 7.61 (t, *J* = 7.2 Hz, 1 H), 8.10 (d, *J* = 7.5 Hz, 2 H); ^{13}C NMR for hydrate form (75 MHz, $CDCl_3$): δ = 13.5, 63.0, 91.7, 128.6, 130.1, 134.5, 135.4, 169.7, 191.6.



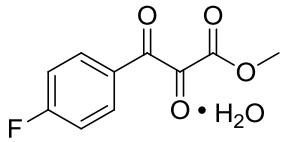
Methyl 2,3-dioxo-3-phenylpropanoate (**2b**): Yellowish oil. Hydrate form:keto form = 10:1. ^{1}H NMR for hydrate form (400 MHz, $CDCl_3$): δ = 3.71 (s, 3 H), 5.70 (brs, 2 H), 7.46 (t, *J* = 7.6 Hz, 2 H), 7.61 (t, *J* = 7.6 Hz, 1 H), 8.10 (d, *J* = 7.6 Hz, 2 H); ^{13}C NMR for hydrate form (100 MHz, $CDCl_3$): δ = 53.5, 91.8, 128.7, 130.1, 134.6, 135.5, 170.2, 191.4.



***tert*-Butyl 2,3-dioxo-3-phenylpropanoate** (**2c**): Yellowish oil. Hydrate form:keto form = 10:1. ^{1}H NMR for hydrate form (400 MHz, $CDCl_3$): δ = 1.30 (s, 9 H), 5.51 (brs, 2 H), 7.47 (t, *J* = 7.6 Hz, 2 H), 7.62 (t, *J* = 7.2 Hz, 1 H), 8.09 (d, *J* = 7.6 Hz, 2 H); ^{13}C NMR for hydrate form (100 MHz, $CDCl_3$): δ = 27.3, 84.7, 91.7, 128.6, 130.0, 134.4, 135.3, 168.8, 192.1.

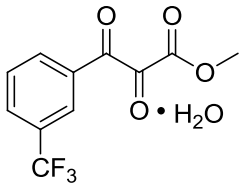


Benzyl 2,3-dioxo-3-phenylpropanoate (**2d**): Yellowish solid, melting point: 75-77 ^{o}C. Hydrate form. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 5.16 (s, 2 H), 5.29 (brs, 2 H), 7.01 (d, *J* = 7.2 Hz, 2 H), 7. 19 (t, *J* = 7.6 Hz, 1 H), 7.25 (t, *J* = 7.2 Hz, 1 H), 7.39 (t, *J* = 8.0 Hz, 2 H), 7.59 (t, *J* = 7.6 Hz, 1 H), 7.99 (d, *J* = 7.2 Hz, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 68.4, 91.6, 128.0, 128.4, 128.5, 128.7, 130.1, 131.2, 134.0, 134.6, 169.6, 191.2.

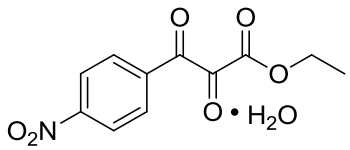


Methyl 3-(4-fluorophenyl)-2,3-dioxopropanoate (**2e**): Yellowish solid, melting point: 67-70 ^{o}C. Hydrate form:keto form = 8:1. IR (KBr): 3461, 3436, 1758, 1744, 1697, 1602, 1245, 1135, 1107, 1007 cm^{-1}. ^{1}H NMR for hydrate form (400 MHz, $CDCl_3$): δ = 3.75 (s, 3 H), 5.40 (brs, 2 H), 7.15 (t, *J* = 7.6 Hz, 2 H), 8.14 (t, *J* = 6.4 Hz, 2 H); ^{13}C NMR for hydrate form (100 MHz, $CDCl_3$): δ = 53.7, 91.8, 116.2 (d, *J* = 22 Hz), 127.7, 133.2 (d, *J* = 9.7 Hz), 166.6 (d, *J* = 256.7 Hz), 170.3, 189.9; ^{19}F NMR for

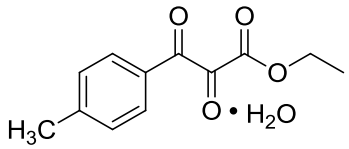
hydrate form (376 MHz, $CDCl_3$): δ = -101.4. HRMS (ESI): *m/z* calcd for $C_{10}H_9FNaO_5$ $[M + Na]^+$: 251.0332, found: 251.0330.



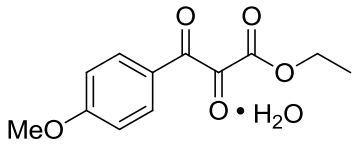
Methyl 2,3-dioxo-3-(3-(trifluoromethyl)phenyl)propanoate (**2f**): Yellowish solid, melting point: 70-73 °C. Hydrate form:keto form = 8:1. IR (KBr): 3465, 3425, 1755, 1742, 1703, 1339, 1225, 1171, 1132, 1112, 1074, 1028, 695 cm^{-1}. ^1H NMR for hydrate form (400 MHz, $CDCl_3$): δ = 3.76 (s, 3 H), 5.41 (brs, 2 H), 7.64 (t, *J* = 7.6 Hz, 1 H), 7.89 (d, *J* = 7.6 Hz, 1 H), 8.26 (d, *J* = 8.0 Hz, 1 H), 8.37 (s, 1 H); ^{13}C NMR for hydrate form (100 MHz, $CDCl_3$): δ = 53.9, 92.0, 123.4 (t, *J* = 271 Hz), 127.0 (t, *J* = 3.9 Hz), 129.5, 131.0 (t, *J* = 3.3 Hz), 132.0, 133.3, 169.9, 190.6; ^{19}F NMR for hydrate form (376 MHz, $CDCl_3$): δ = -63.0. HRMS (ESI): *m/z* calcd for $C_{11}H_7F_3NaO_4$ $[M + Na]^+$: 283.0194, found: 283.0190.



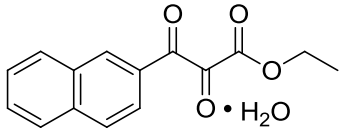
Ethyl 3-(4-nitrophenyl)-2,3-dioxopropanoate (**2g**): Yellowish solid, melting point: 81-84 °C. Hydrate form:keto form = 10:1. ^1H NMR for hydrate form (400 MHz, $CDCl_3$): δ = 1.02 (t, *J* = 6.8 Hz, 3 H), 4.15 (t, *J* = 7.2 Hz, 2 H), 5.49 (brs, 2 H), 8.18-8.24 (m, 4 H); ^{13}C NMR for hydrate form (100 MHz, $CDCl_3$): δ = 13.6, 63.4, 92.2, 123.7, 131.2, 136.1, 150.8, 169.0, 190.7.



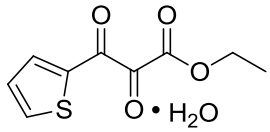
Ethyl 2,3-dioxo-3-(*p*-tolyl)propanoate (**2h**): Yellowish oil. Hydrate form:keto form = 6:1. ^1H NMR for hydrate form (300 MHz, $CDCl_3$): δ = 1.09 (t, *J* = 7.2 Hz, 3 H), 2.42 (s, 3 H), 4.21 (q, *J* = 7.2 Hz, 2 H), 5.50 (brs, 2 H), 7.26 (d, *J* = 8.1 Hz, 2 H), 7.98 (d, *J* = 8.4 Hz, 2 H); ^{13}C NMR for hydrate form (75 MHz, $CDCl_3$): δ = 13.6, 21.8, 63.0, 91.6, 128.8, 129.4, 130.3, 145.8, 170.0, 191.1.



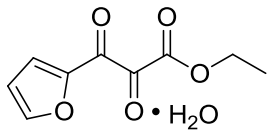
Ethyl 3-(4-methoxyphenyl)-2,3-dioxopropanoate (**2i**): Yellowish oil. Hydrate form:keto form = 6:1. ^{1}H NMR for hydrate form (400 MHz, $CDCl_3$): δ = 1.10 (t, *J* = 7.2 Hz, 3 H), 3.87 (s, 3 H), 4.21 (q, *J* = 7.2 Hz, 2 H), 5.57 (brs, 2 H), 6.93 (d, *J* = 8.8 Hz, 2 H), 8.08 (d, *J* = 8.8 Hz, 2 H); ^{13}C NMR for hydrate form (100 MHz, $CDCl_3$): δ = 13.7, 55.5, 63.0, 91.6, 114.0, 124.1, 132.8, 164.7, 170.2, 189.8.



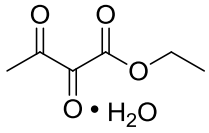
Ethyl 3-(naphthalen-2-yl)-2,3-dioxopropanoate (**2j**): Yellowish solid, melting point: 68-70 ^{o}C. Hydrate form:keto form = 7:1. IR (KBr): 3431, 3406, 1740, 1687, 1624, 1298, 1127, 1105, 1030, 754 cm^{-1}. ^{1}H NMR for hydrate form (400 MHz, $CDCl_3$): δ = 1.05 (t, *J* = 7.2 Hz, 3 H), 4.21 (t, *J* = 7.2 Hz, 2 H), 5.46 (brs, 2 H), 7.56 (t, *J* = 7.2 Hz, 1 H), 7.64 (t, *J* = 7.2 Hz, 1 H), 7.89 (t, *J* = 8.8 Hz, 2 H), 7.96 (d, *J* = 8.0 Hz, 1 H), 8.08 (dd, *J* = 8.8, 1.2 Hz, 1 H), 8.67 (s, 1 H); ^{13}C NMR for hydrate form (100 MHz, $CDCl_3$): δ = 13.6, 63.2, 91.8, 124.8, 127.1, 127.8, 128.6, 129.5, 130.1, 132.3, 132.9, 136.1, 170.0, 191.5. HRMS (ESI): *m/z* calcd for $C_{15}H_{14}NaO_5$ [hydrate form + Na]$^{+}$: 297.0739, found: 297.0736; HRMS (ESI): *m/z* calcd for $C_{15}H_{12}NaO_4$ [keto form + Na]$^{+}$: 279.0633, found: 279.0632.



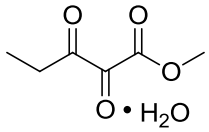
Ethyl 2,3-dioxo-3-(thiophen-2-yl)propanoate (**2k**): Yellowish oil. Hydrate form:keto form = 7:1. IR (KBr): 3408, 2987, 1748, 1667, 1410, 1246, 1136, 1104, 1058, 742 cm^{-1}. ^{1}H NMR for hydrate form (400 MHz, $CDCl_3$): δ = 1.15 (t, *J* = 7.2 Hz, 3 H), 4.24 (q, *J* = 7.2 Hz, 2 H), 5.35 (brs, 2 H), 7.16 (t, *J* = 4.0 Hz, 1 H), 7.78 (d, *J* = 5.2 Hz, 1 H), 7.94 (d, *J* = 4.0 Hz, 1 H); ^{13}C NMR for hydrate form (100 MHz, $CDCl_3$): δ = 13.7, 63.4, 92.1, 128.6, 136.2, 136.5, 138.3, 169.7, 184.9. HRMS (ESI): *m/z* calcd for $C_9H_{10}NaO_5S$ [hydrate form + Na]$^{+}$: 253.0147, found: 253.0142; HRMS (ESI): *m/z* calcd for $C_9H_8NaO_4S$ [keto form + Na]$^{+}$: 235.0041, found: 235.0040.



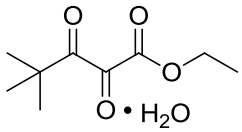
Ethyl 3-(furan-2-yl)-2,3-dioxopropanoate (**2l**): Yellowish oil. Hydrate form:keto form = 17:1. IR (KBr): 3423, 1747, 1683, 1464, 1258, 1135, 1114, 1035, 775 cm^{-1}. ^{1}H NMR for hydrate form (400 MHz, $CDCl_3$): δ = 1.19 (t, *J* = 7.2 Hz, 3 H), 4.27 (q, *J* = 7.2 Hz, 2 H), 5.33 (brs, 2 H), 6.10 (dd, *J* = 3.6, 1.6 Hz, 1 H), 7.47 (d, *J* = 3.6 Hz, 1 H), 7.71 (d, *J* = 0.8 Hz, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.8, 63.4, 91.1, 112.9, 122.7, 148.1, 148.6, 169.3, 180.1. HRMS (ESI): *m/z* calcd for $C_9H_{10}NaO_6$ [hydrate form + Na]$^+$: 237.0375, found: 237.0373; HRMS (ESI): *m/z* calcd for $C_9H_8NaO_5$ [keto form + Na]$^+$: 219.0269, found: 219.0268; HRMS (ESI): *m/z* calcd for $C_9H_9O_5$ [keto form + H]$^+$: 197.0450, found: 197.0452.



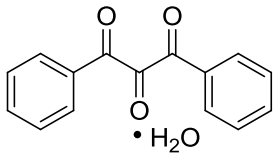
Ethyl 2,3-dioxobutanoate (**2m**): Yellowish oil. Hydrate form. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.32 (t, *J* = 7.2 Hz, 3 H), 2.30 (s, 3 H), 4.33 (q, *J* = 7.2 Hz, 2 H), 5.04 (brs, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.9, 23.1, 63.4, 92.4, 168.9, 200.8.



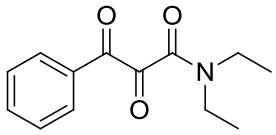
Methyl 2,3-dioxopentanoate (**2n**): Yellowish oil. Hydrate form. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.14 (t, *J* = 7.2 Hz, 3 H), 2.64 (q, *J* = 7.2 Hz, 2 H), 3.86 (s, 3 H), 5.09 (brs, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 7.3, 29.1, 53.8, 92.4, 169.6, 203.9.



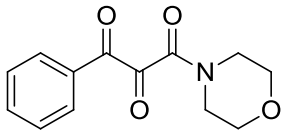
Ethyl 4,4-dimethyl-2,3-dioxopentanoate (**2o**): Yellowish oil. Hydrate form:keto form = 1.2:1. ^{1}H NMR for keto form (400 MHz, $CDCl_3$): δ = 1.30 (s, 9 H), 1.38 (t, *J* = 7.2 Hz, 3 H), 4.39 (q, *J* = 7.2 Hz, 2 H); ^{1}H NMR for hydrate form (400 MHz, $CDCl_3$): δ = 1.26 (s, 9 H), 1.31 (t, *J* = 7.2 Hz, 3 H), 4.30 (q, *J* = 7.2 Hz, 2 H), 5.20 (brs, 2 H); ^{13}C NMR for keto and hydrate form (100 MHz, $CDCl_3$): δ = 13.9, 25.5, 27.2, 42.6, 43.0, 63.1, 92.0, 160.4, 169.3, 184.4, 206.6, 208.3.



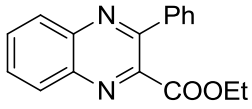
1,3-Diphenylpropane-1,2,3-trione (**2p**). Yellowish solid, melting point: 111-114 ^{o}C. Hydrate form:keto form = 1.1:1. ^{1}H NMR for keto and hydrate form (400 MHz, $CDCl_3$): δ = 5.91 (s, 1 H), 7.36 (t, *J* = 7.6 Hz, 2 H), 7.52 (t, *J* = 7.6 Hz, 1 H), 7.57 (t, *J* = 7.6 Hz, 2 H), 7.72 (t, *J* = 7.6 Hz, 1 H), 7.94 (d, *J* = 7.6 Hz, 2 H), 8.09 (d, *J* = 7.2 Hz, 2 H); ^{13}C NMR for keto and hydrate form (100 MHz, $CDCl_3$): δ = 94.0, 128.8, 129.1, 130.2, 130.2, 132.0, 132.1, 134.7, 135.4, 188.2, 192.4, 194.0.



***N*,*N*-Diethyl-2,3-dioxo-3-phenylpropanamide** (**2q**). Yellowish oil. Hydrate form:keto form = 1:10. ^{1}H NMR for keto form (400 MHz, $CDCl_3$): δ = 1.24 (t, *J* = 7.2 Hz, 3 H), 1.31 (t, *J* = 7.2 Hz, 3 H), 3.44-3.53 (m, 4 H), 7.53 (t, *J* = 7.6 Hz, 2 H), 7.67 (t, *J* = 7.6 Hz, 1 H), 8.06 (d, *J* = 7.2 Hz, 2 H); ^{13}C NMR for keto form (100 MHz, $CDCl_3$): δ = 12.5, 14.4, 39.5, 42.2, 128.9, 130.3, 132.0, 135.1, 165.3, 185.5, 192.2.

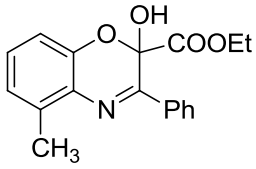


1-Morpholino-3-phenylpropane-1,2,3-trione (**2r**). Yellowish solid, melting point: 88-91 ^{o}C. Hydrate form:keto form = 1:2. IR (KBr): 3323, 2865, 1690, 1640, 1240, 1134, 1095, 1030, 875 cm^{-1}. ^{1}H NMR for keto form (400 MHz, $CDCl_3$): δ = 3.34-3.36 (m, 1 H), 3.60-3.63 (m, 3 H), 3.78-3.81 (m, 4 H), 7.54 (t, *J* = 7.6 Hz, 2 H), 7.67-7.61 (m, 1 H), 8.06 (d, *J* = 8.0 Hz, 2 H); ^{1}H NMR for hydrate form (400 MHz, $CDCl_3$): δ = 3.24-3.26 (m, 2 H), 3.54-3.56 (m, 2 H), 3.71-3.74 (m, 4 H), 5.95 (s, 2 H), 7.46-7.50 (m, 2 H), 7.61-7.65 (m, 1 H), 8.02-8.05 (m, 2 H); ^{13}C NMR for keto and hydrate form (100 MHz, $CDCl_3$): δ = 42.0, 44.0, 46.1, 46.2, 66.0, 66.3, 66.4, 66.8, 91.3, 128.9, 128.9, 130.0, 131.6, 134.8, 135.3, 164.1, 167.3, 185.2, 191.8, 194.1. HRMS (ESI): *m/z* calcd for $C_{13}H_{13}NNaO_4$ $[M + Na]^+$: 270.0742, found: 270.0740.

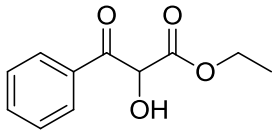


Ethyl 3-phenylquinoxaline-2-carboxylate (**3a**): Colorless soild, melting point: 55-56 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.18 (t, *J* = 7.2 Hz, 3 H), 4.34 (q, *J* = 7.2

Hz, 2 H), 7.50-7.54 (m, 3 H), 7.74-7.76 (m, 2 H), 7.81-7.89 (m, 2 H), 8.18-8.24 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.7, 62.4, 128.5, 128.6, 129.3, 129.6, 130.5, 131.7, 137.8, 139.9, 142.2, 145.7, 152.2, 166.6.



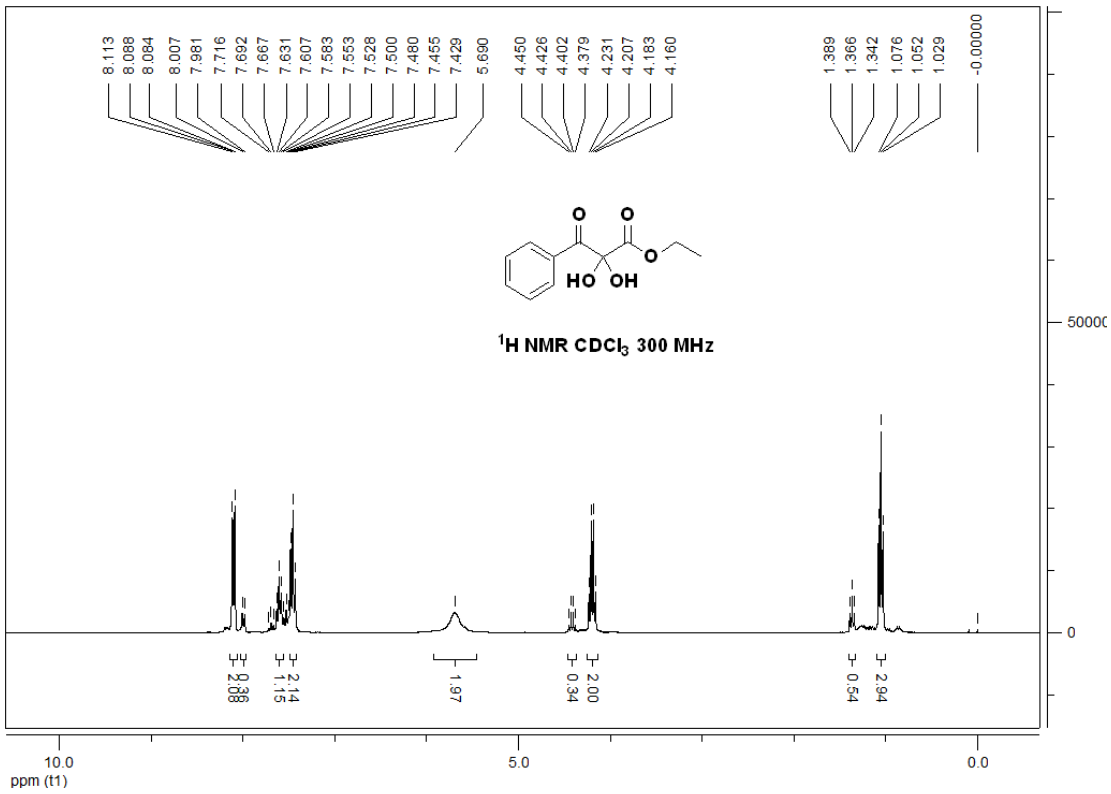
Ethyl 2-hydroxy-5-methyl-3-phenyl-2*H*-benzo[*b*][1,4]oxazine-2-carboxylate (**3b**): Colorless solid, melting point: 84-85 ^{o}C. IR (KBr): 3452, 2980, 1731, 1265, 1128, 1056, 744 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.85 (t, *J* = 7.2 Hz, 3 H), 2.58 (s, 3 H), 4.01-4.09 (m, 1 H), 4.15-4.23 (m, 1 H), 4.97 (s, 1 H), 6.85 (d, *J* = 8.0 Hz, 1 H), 6.98 (d, *J* = 7.2 Hz, 1 H), 7.14 (t, *J* = 8.0 Hz, 1 H), 7.38-7.43 (m, 3 H), 7.86 (d, *J* = 8.0 Hz, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.2, 16.8, 63.5, 89.0, 113.8, 124.3, 127.2, 128.4, 128.7, 129.6, 130.3, 136.0, 137.2, 141.9, 152.0, 169.3. HRMS (MALDI): *m/z* calcd for $C_{18}H_{18}NO_4$ $[M + H]^+$: 312.1236, found: 312.1229.

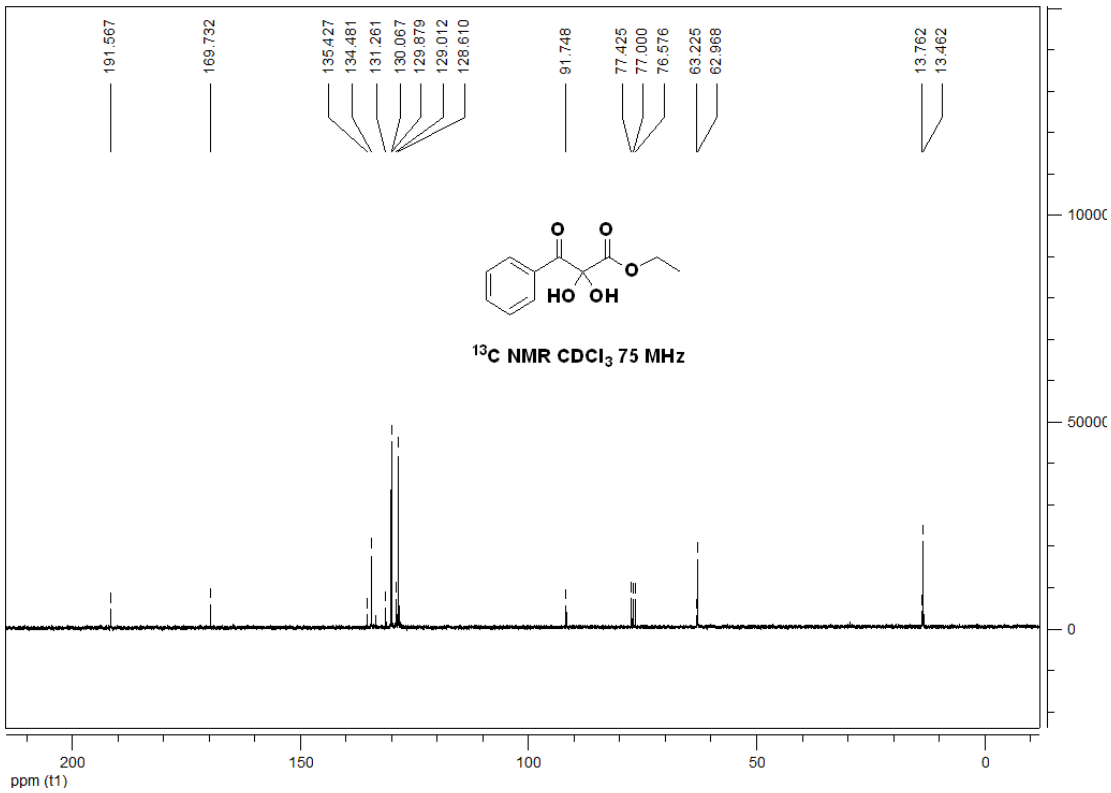


Ethyl 2-hydroxy-3-oxo-3-phenylpropanoate (**4a**): Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.15 (t, *J* = 7.2 Hz, 3 H), 4.17 (q, *J* = 7.2 Hz, 2 H), 4.43 (brs, 1 H), 5.62 (s, 1 H), 7.51 (t, *J* = 7.6 Hz, 2 H), 7.64 (t, *J* = 7.6 Hz, 1 H), 8.08 (d, *J* = 7.6 Hz, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.7, 62.1, 74.4, 128.6, 129.3, 132.9, 134.5, 168.5, 193.8.

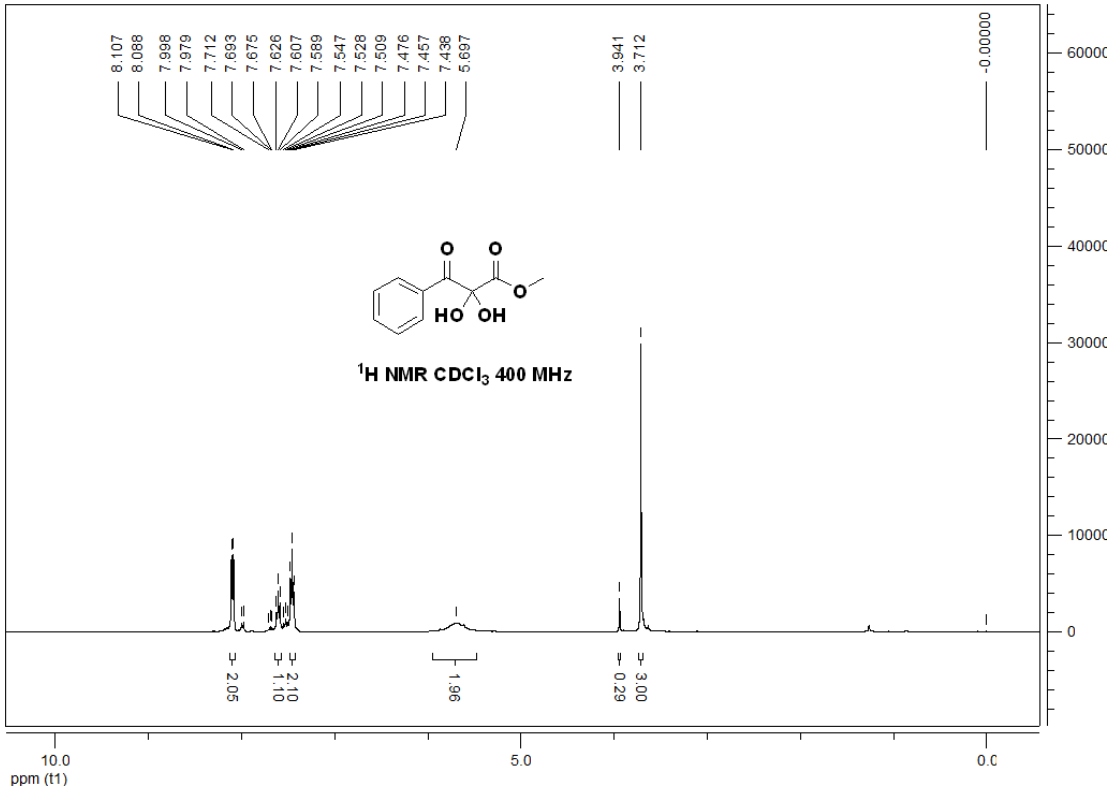
NMR and HRMS Spectra:

Ethyl 2,3-dioxo-3-phenylpropanoate (2a)

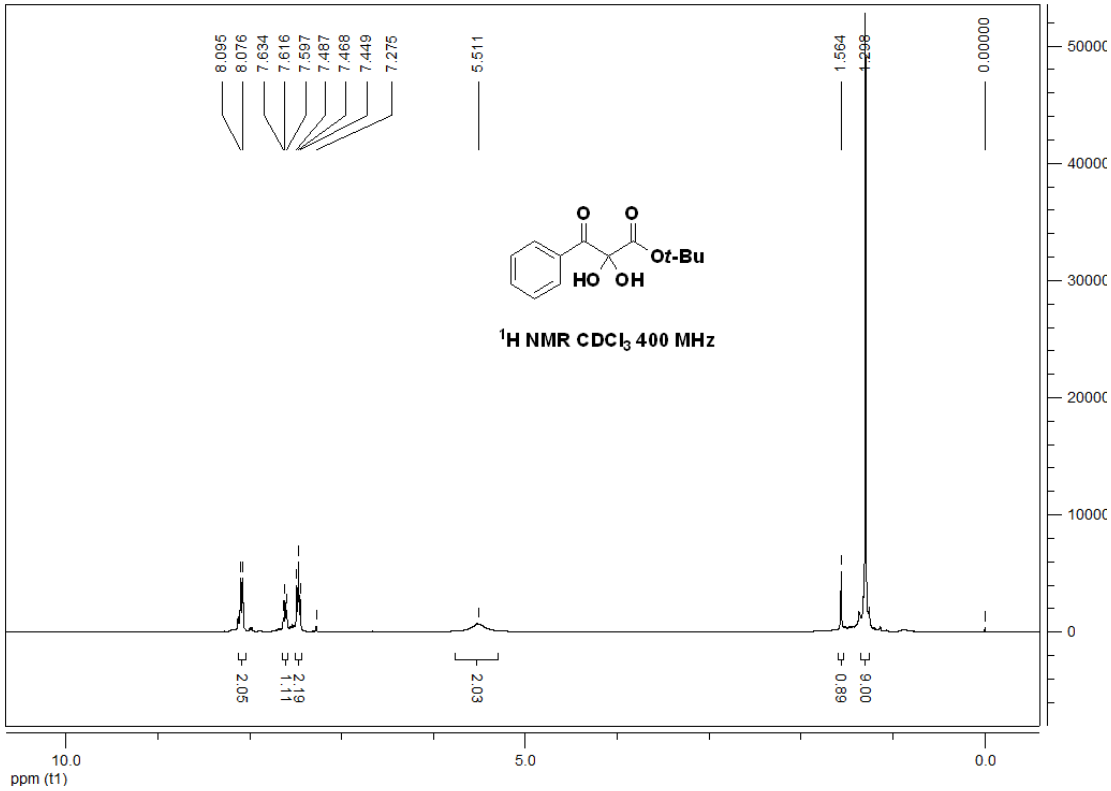


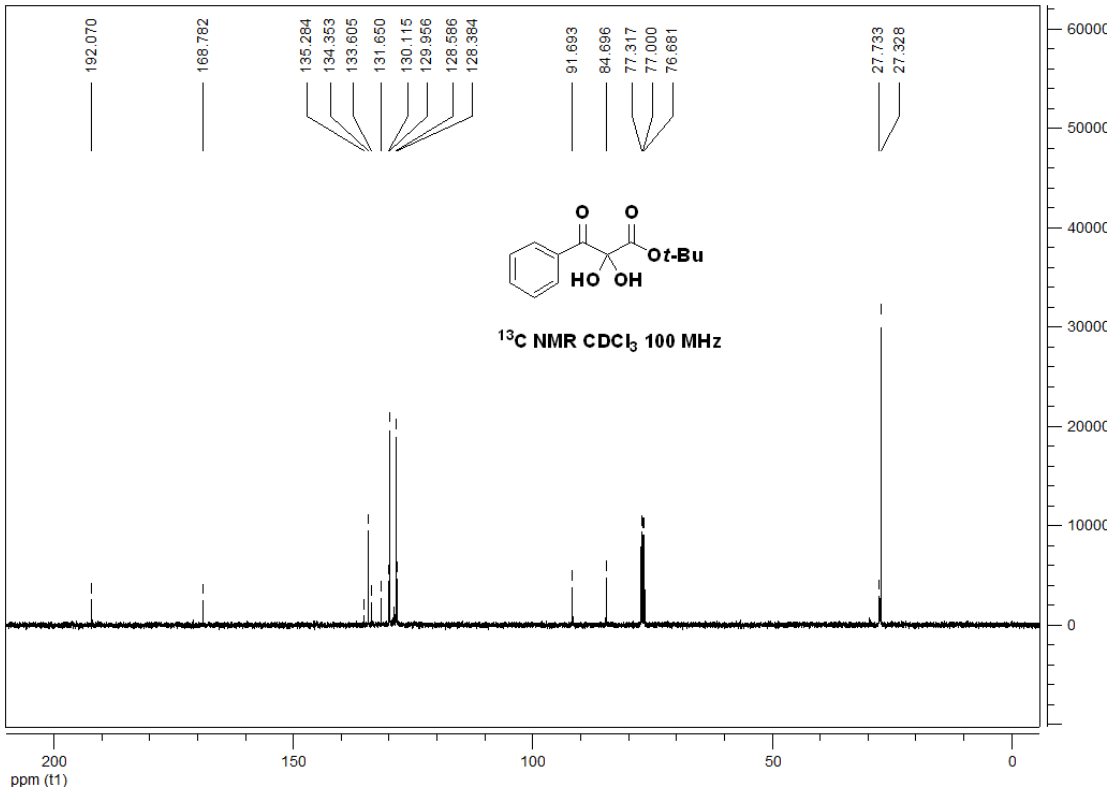


Methyl 2,3-dioxo-3-phenylpropanoate (2b)

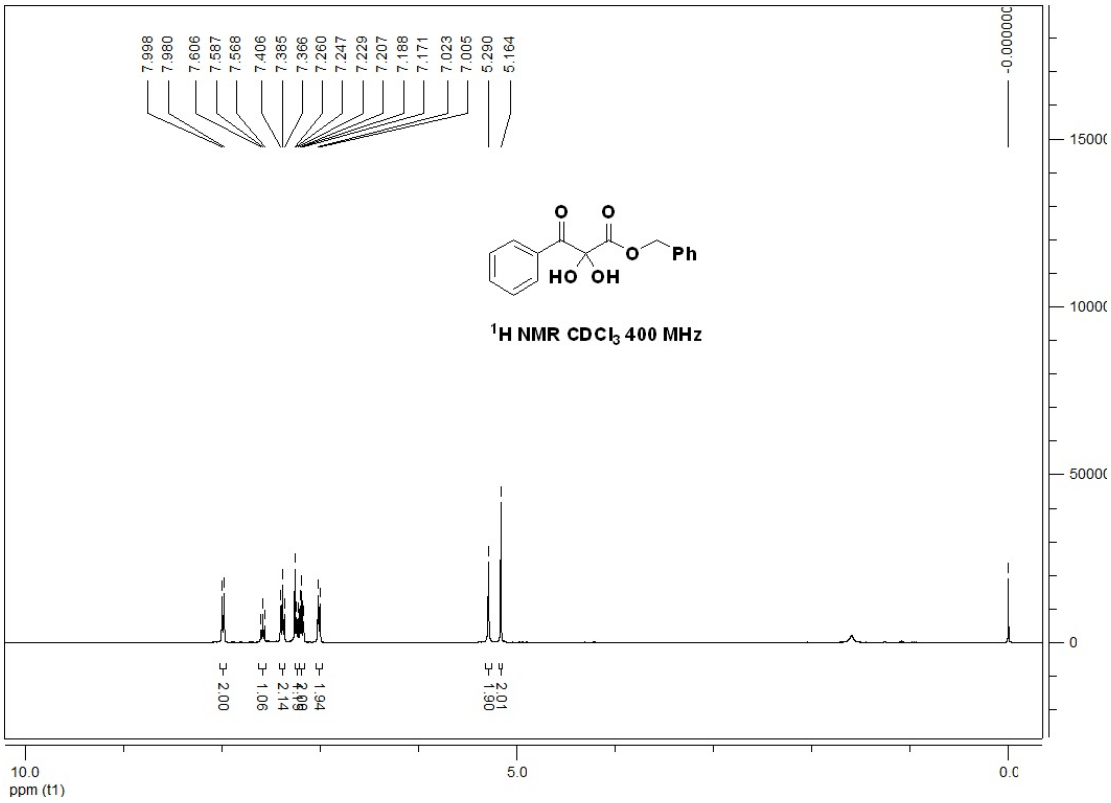


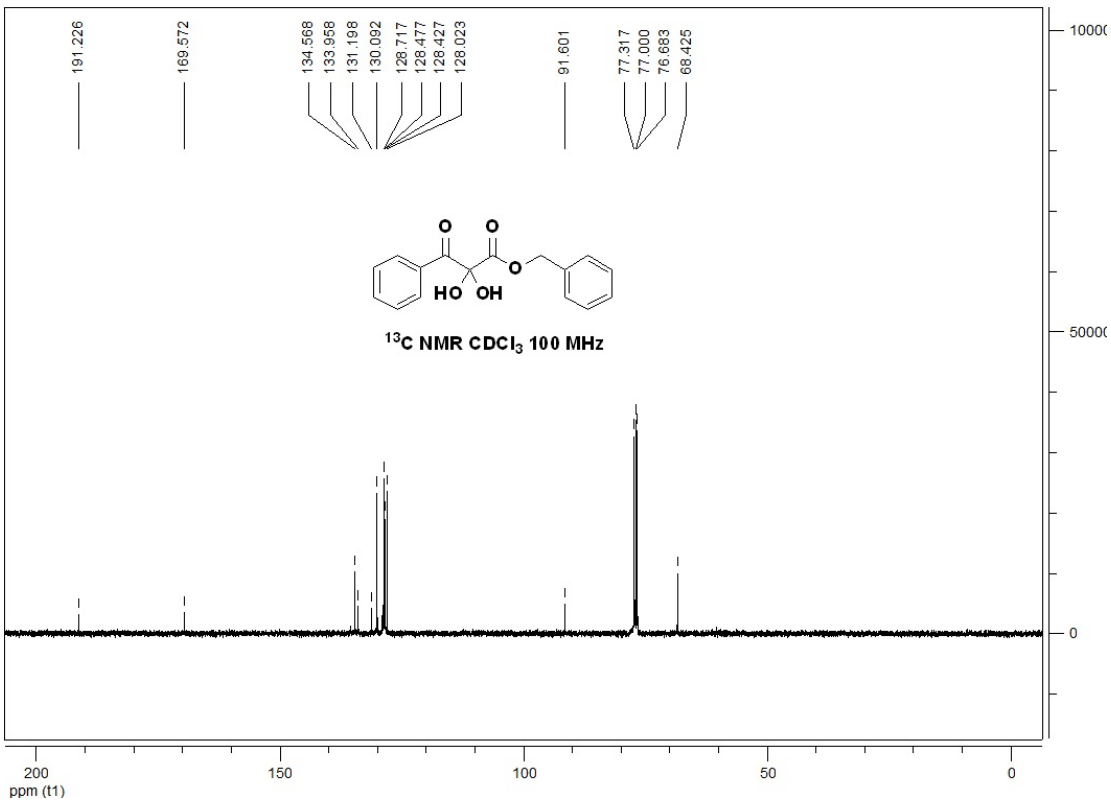
tert-Butyl 2,3-dioxo-3-phenylpropanoate (2c)



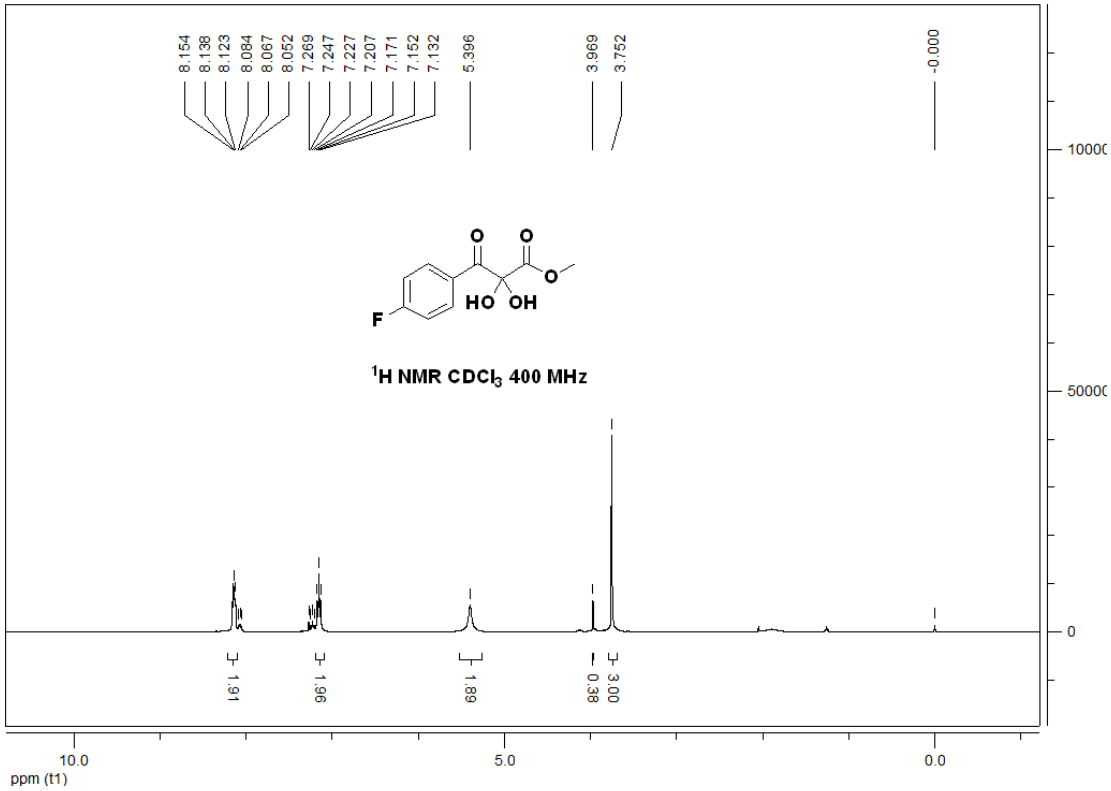


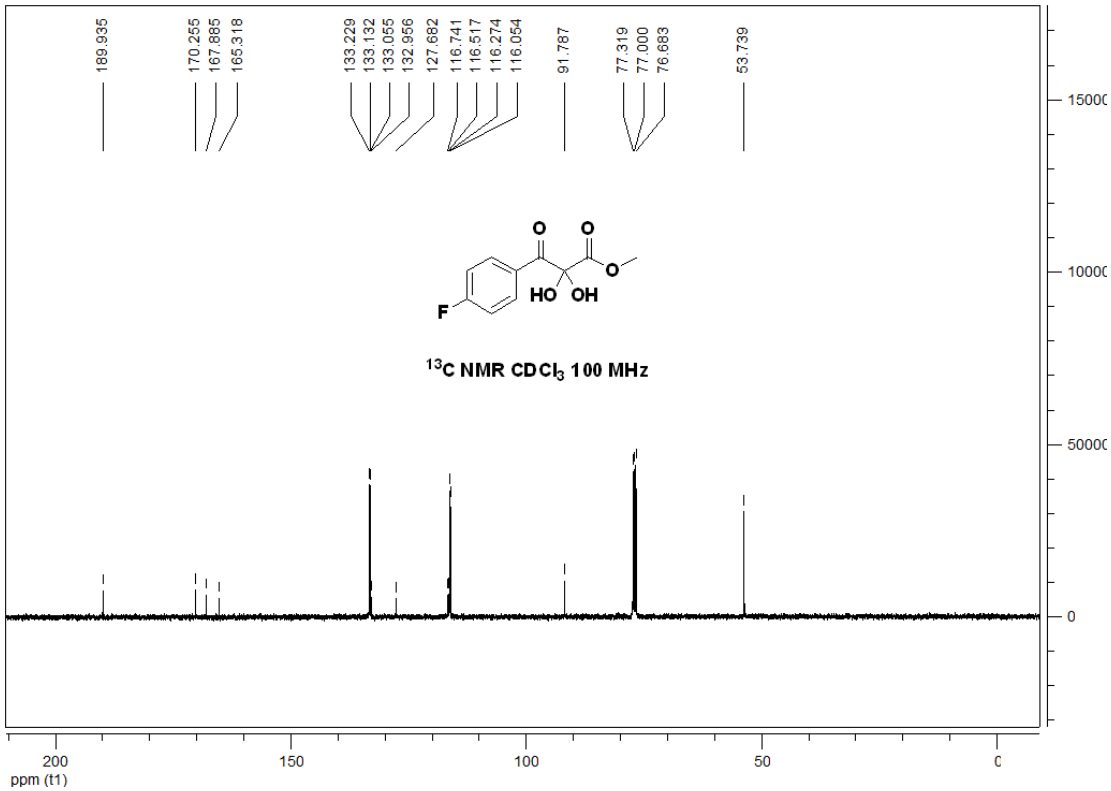
Benzyl 2,3-dioxo-3-phenylpropanoate (2d)

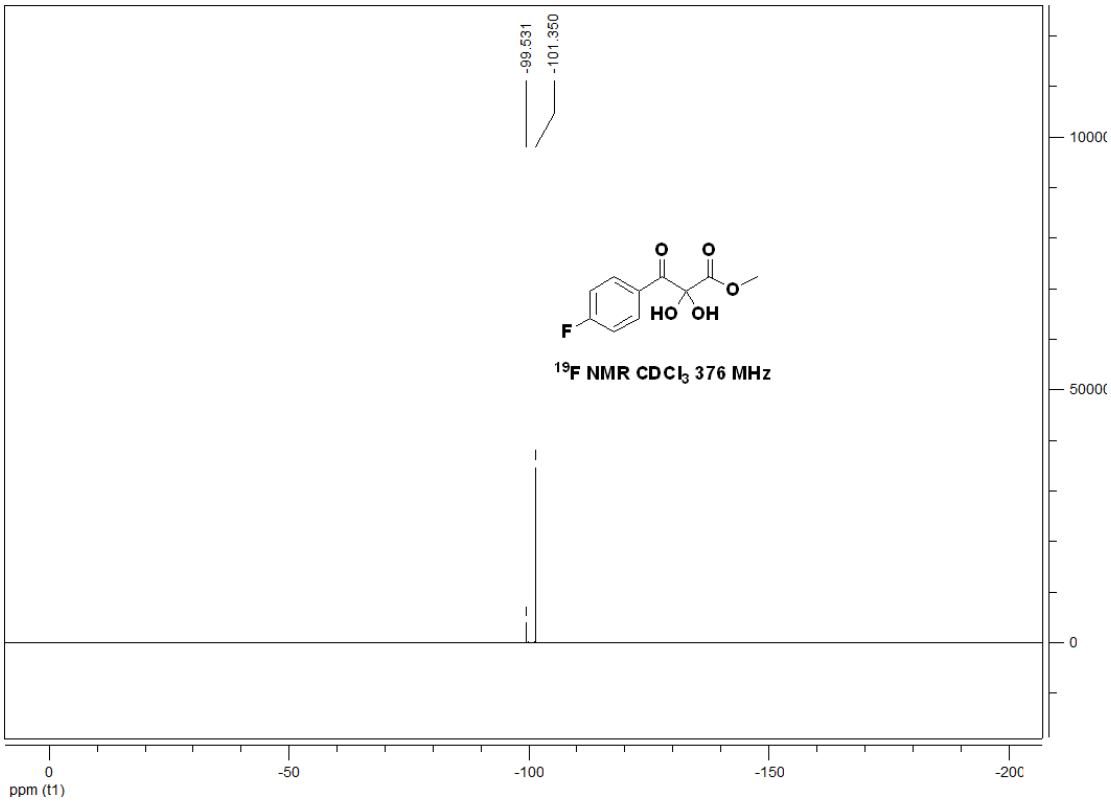


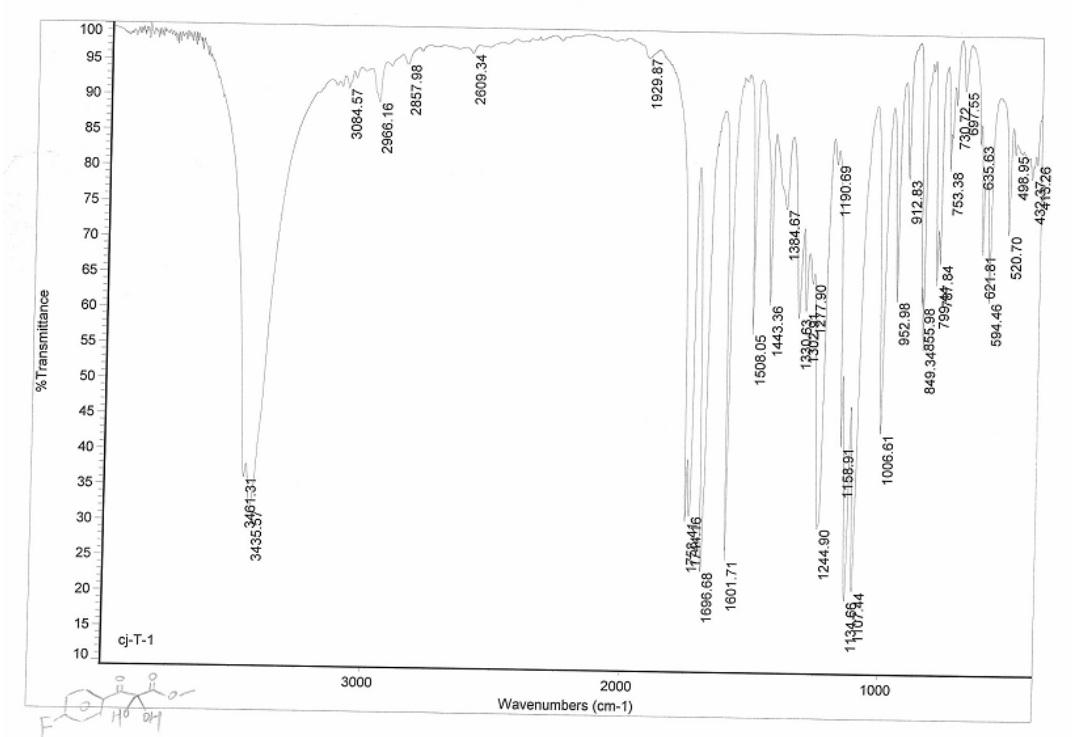


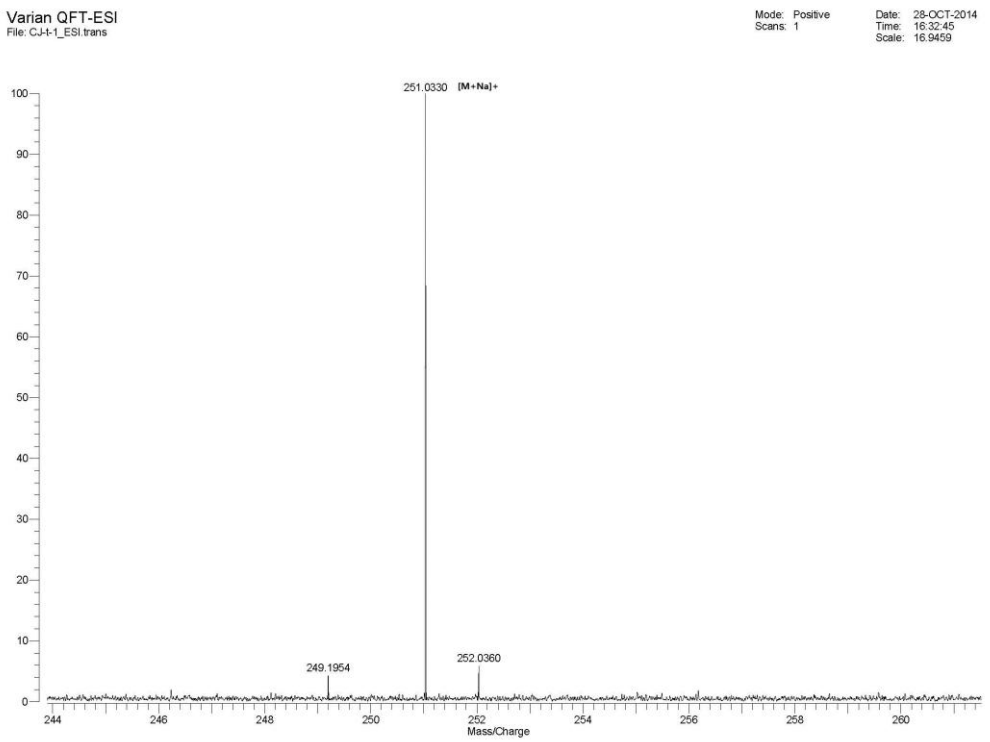
Methyl 3-(4-fluorophenyl)-2,3-dioxopropanoate (2e)


8.154
8.138
8.123
8.084
8.067
8.052
7.269
7.247
7.227
7.207
7.171
7.152
7.132
5.396
3.969
3.752
-0.000
O
O
O
HO
OH
F
^{1}H NMR CDCl$_3$ 400 MHz
1.91
1.96
1.89
0.38
3.00
10000
5000
0
10.0
5.0
0.0
ppm (t1)

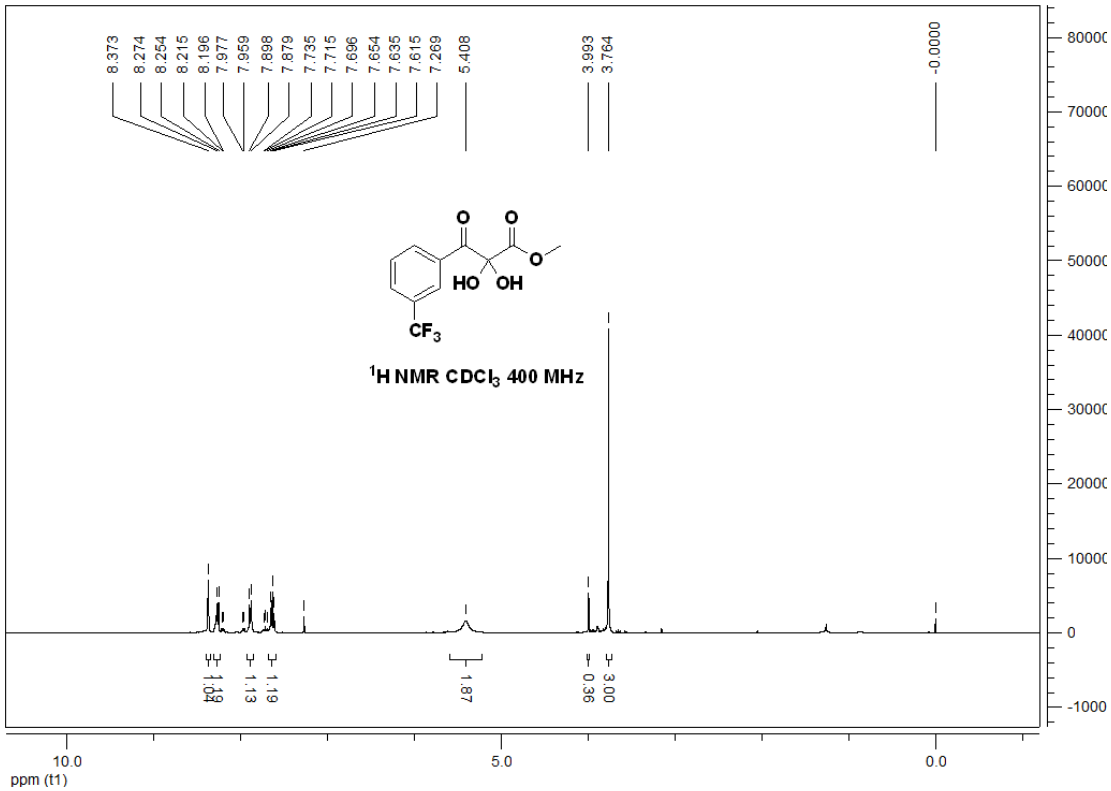

189.935
170.255
167.885
165.318
133.229
133.132
133.055
132.956
127.682
116.741
116.517
116.274
116.054
91.787
77.319
77.000
76.683
53.739
O
O
O
HO
OH
F
^{13}C NMR CDCl$_3$ 100 MHz
15000
10000
5000
0
200
150
100
50
0
ppm (t1)

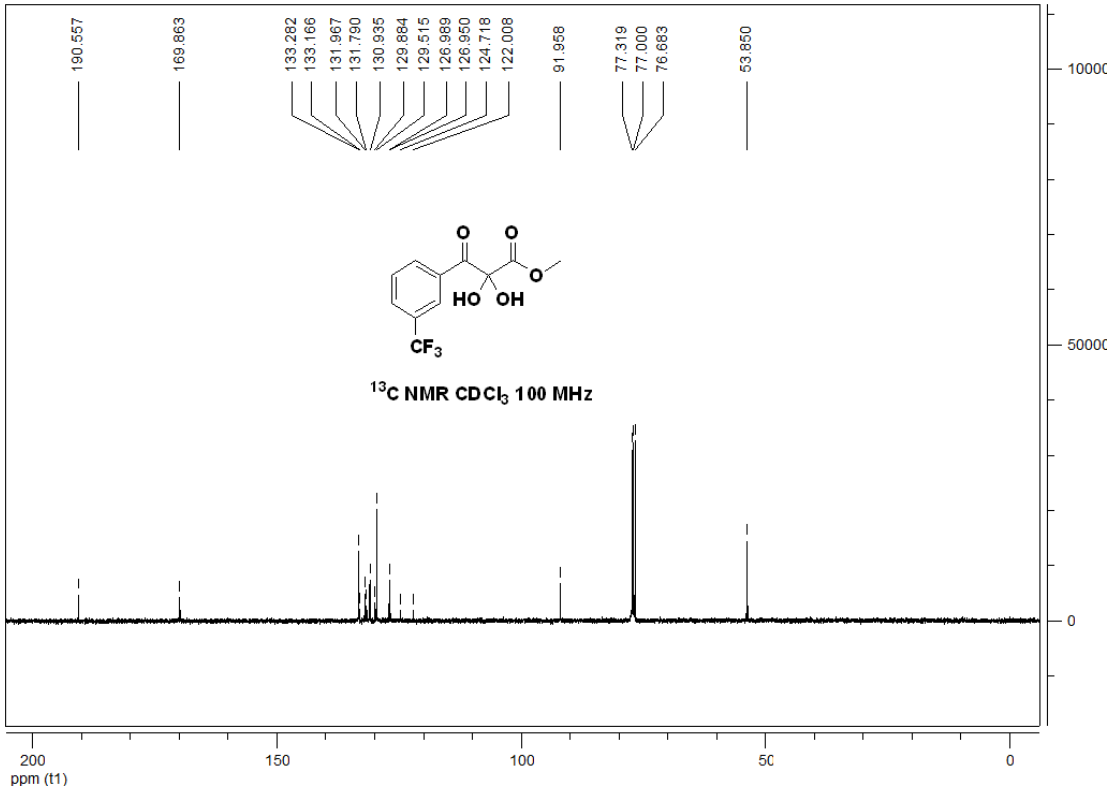

-99.531
-101.350
O
O
O
HO
OH
F
^{19}F NMR CDCl$_3$ 376 MHz
10000
50000
0
0
-50
-100
-150
-200
ppm (t1)

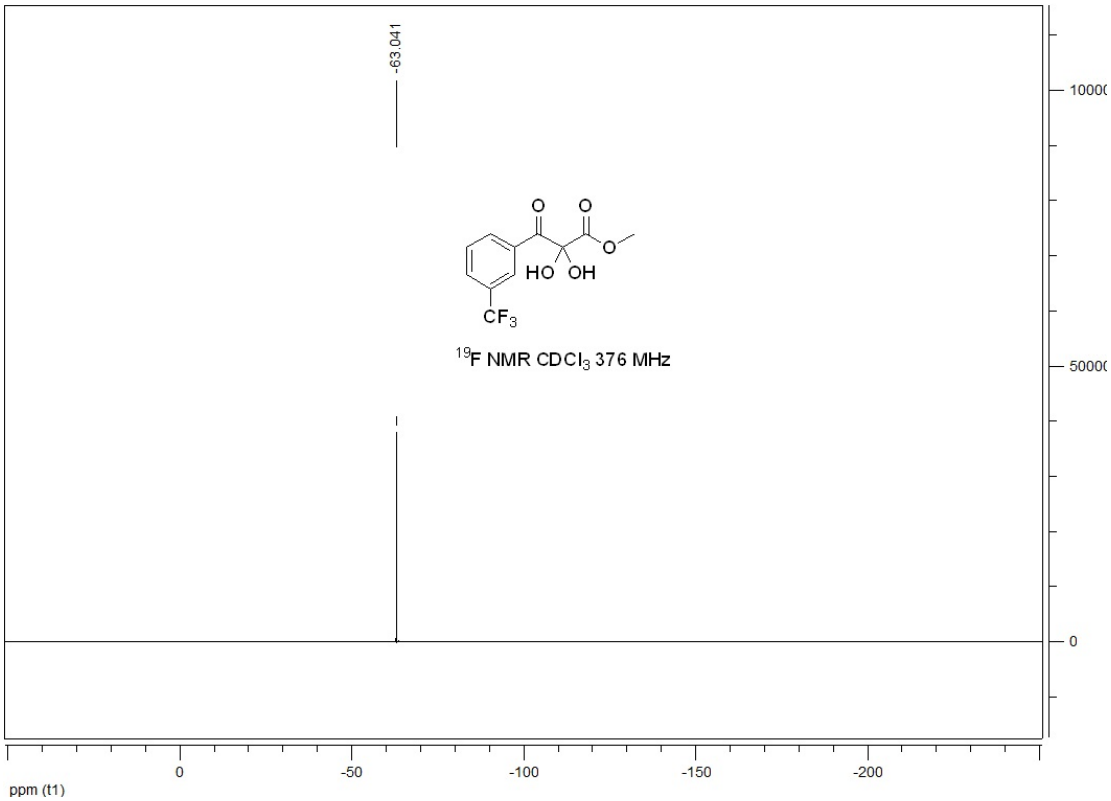

%Transmittance
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
3435.37
3461.31
3084.57
2966.16
2857.98
2609.34
1929.87
1758.46
1754.16
1696.68
1601.71
1506.05
1443.36
1384.67
1330.63
1302.31
1217.90
1244.90
1190.69
1158.91
1134.66
1110.44
1006.61
952.98
912.83
849.34
855.98
799.41
797.84
753.38
730.72
697.55
635.63
621.81
594.46
520.70
498.95
432.37
413.26
cj-T-1
3000
2000
1000
Wavenumbers (cm-1)

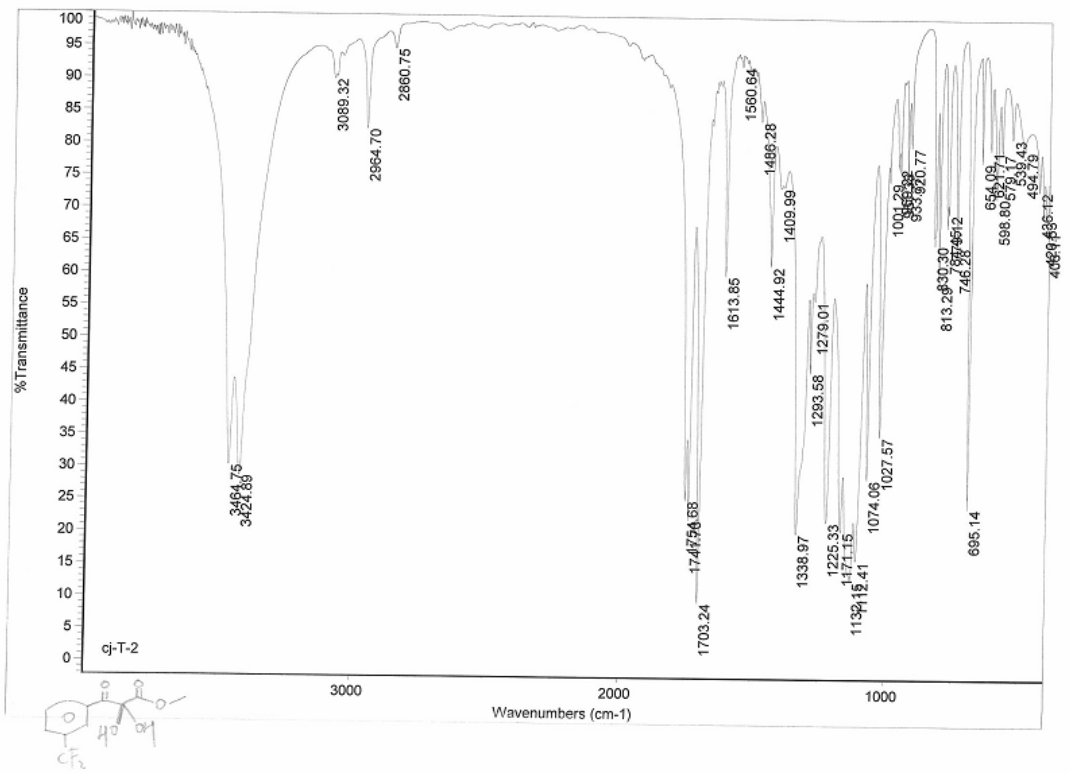


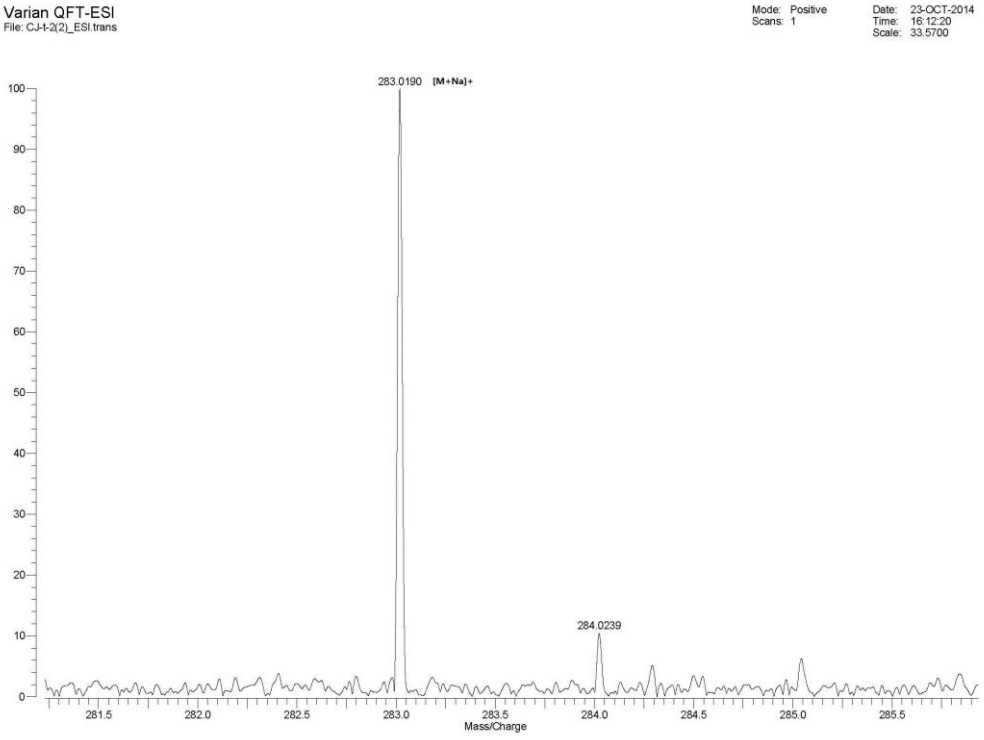
Methyl 2,3-dioxo-3-(3-(trifluoromethyl)phenyl)propanoate (2f)



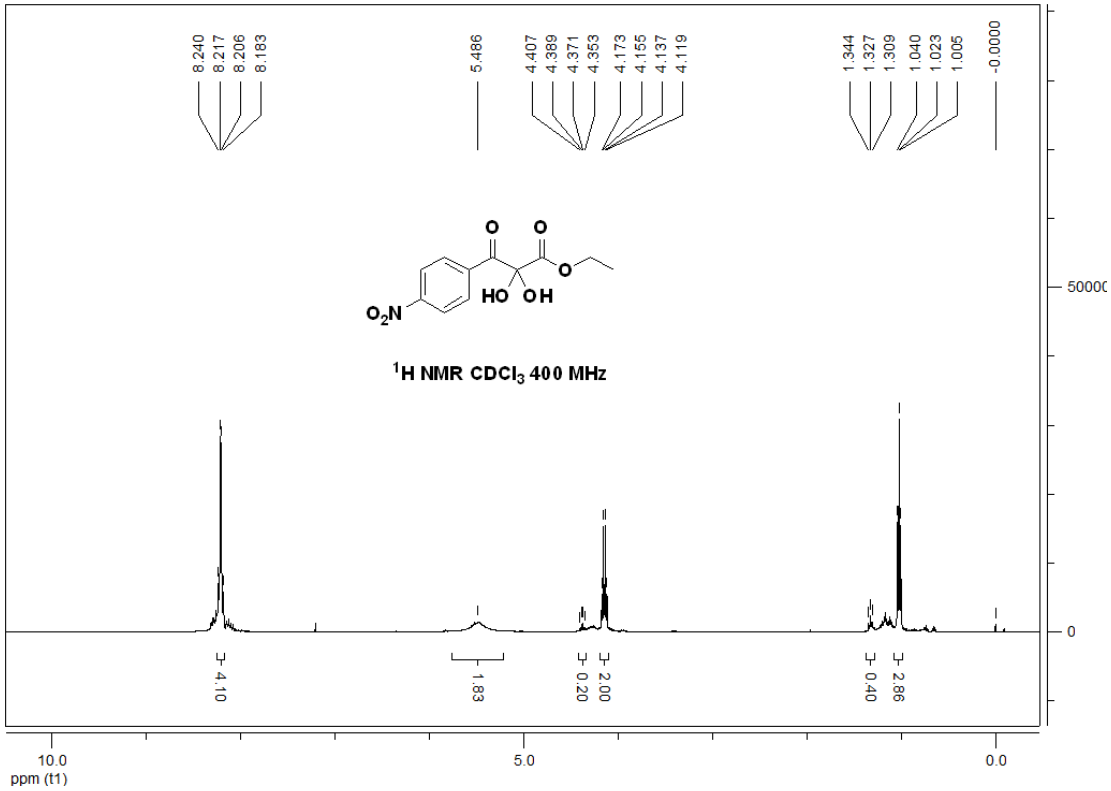

190.557
169.863
133.282
133.166
131.967
131.790
130.935
129.884
129.515
126.989
126.950
124.718
122.008
91.958
77.319
77.000
76.683
53.850
HO OH
CF3
13C NMR CDCl3 100 MHz
200
150
100
50
0
ppm (t1)

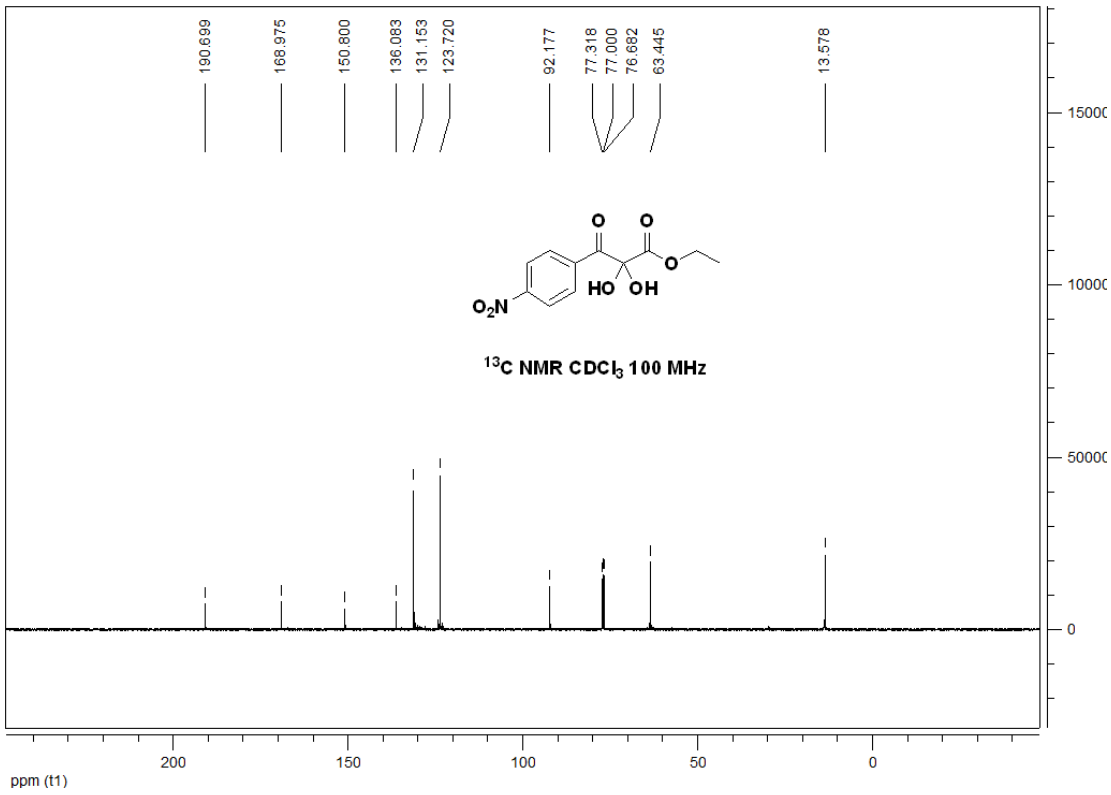

-63.041
HO OH
CF3
19F NMR CDCl3 376 MHz
0
-50
-100
-150
-200
ppm (t1)


%Transmittance
Wavenumbers (cm-1)
cj-T-2
3089.32
2964.70
2860.75
3464.75
3424.89
1703.24
1613.85
1560.64
1486.28
1444.92
1409.99
1338.97
1293.58
1279.01
1225.33
1171.15
1074.06
1027.57
695.14

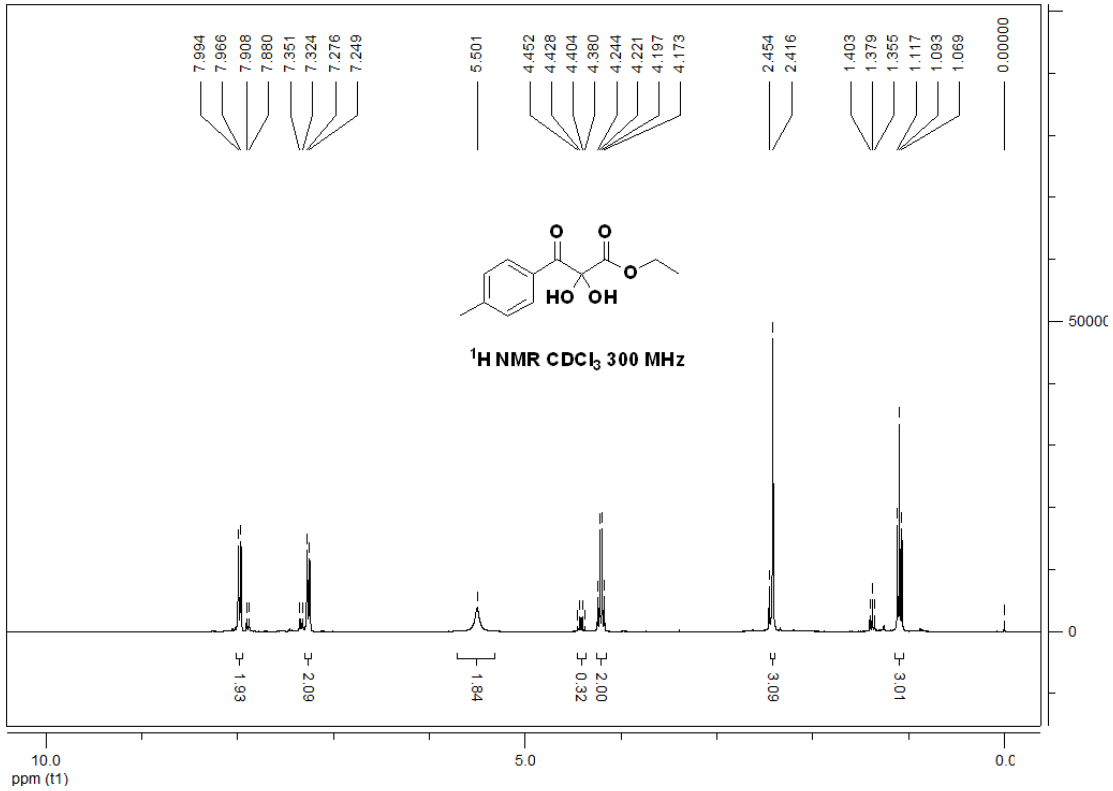

Varian QFT-ESI
File: CJ-I-2(2)_ESI.trans
Mode: Positive
Scans: 1
Date: 23-OCT-2014
Time: 16:12:20
Scale: 33.5700
283.0190 [M+Na]+
284.0239
Mass/Charge

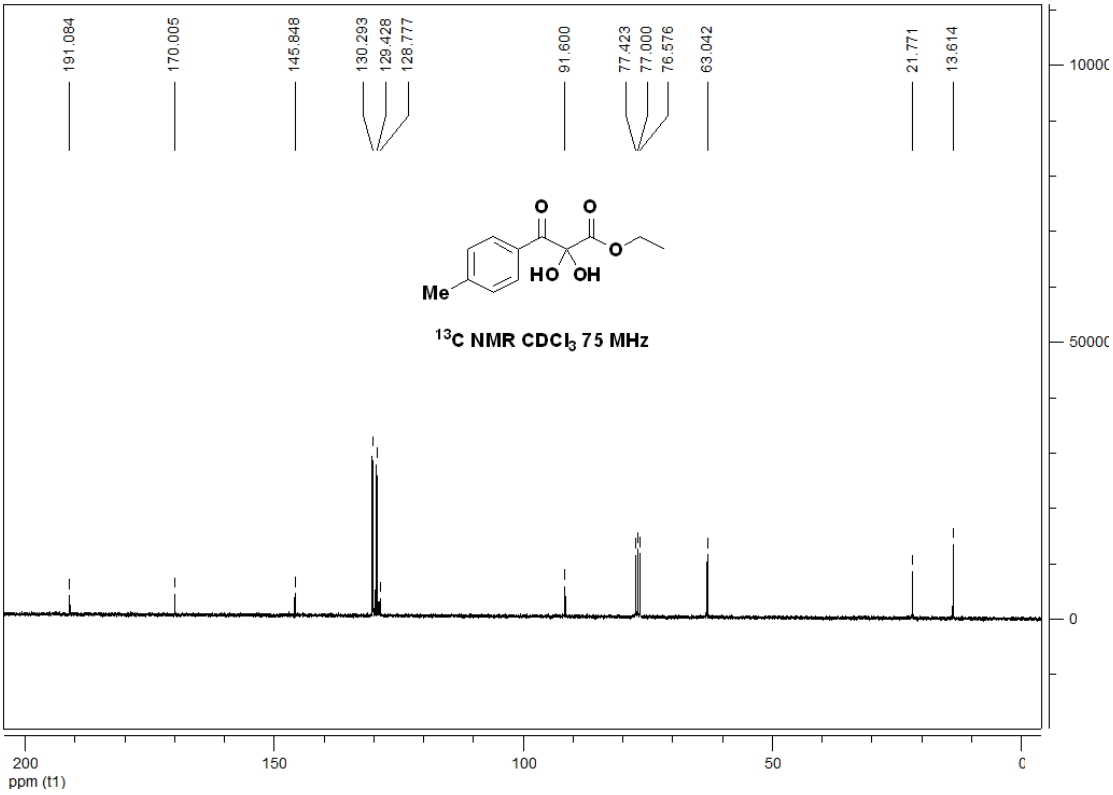
Ethyl 3-(4-nitrophenyl)-2,3-dioxopropanoate (2g)



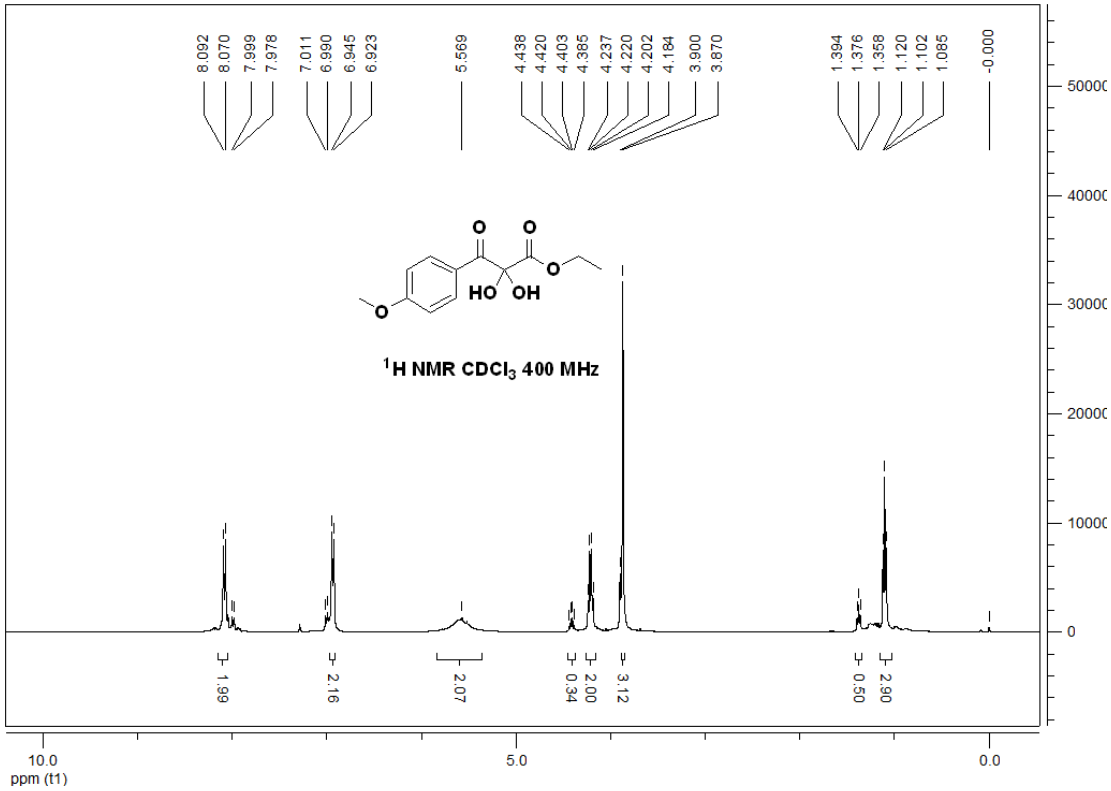


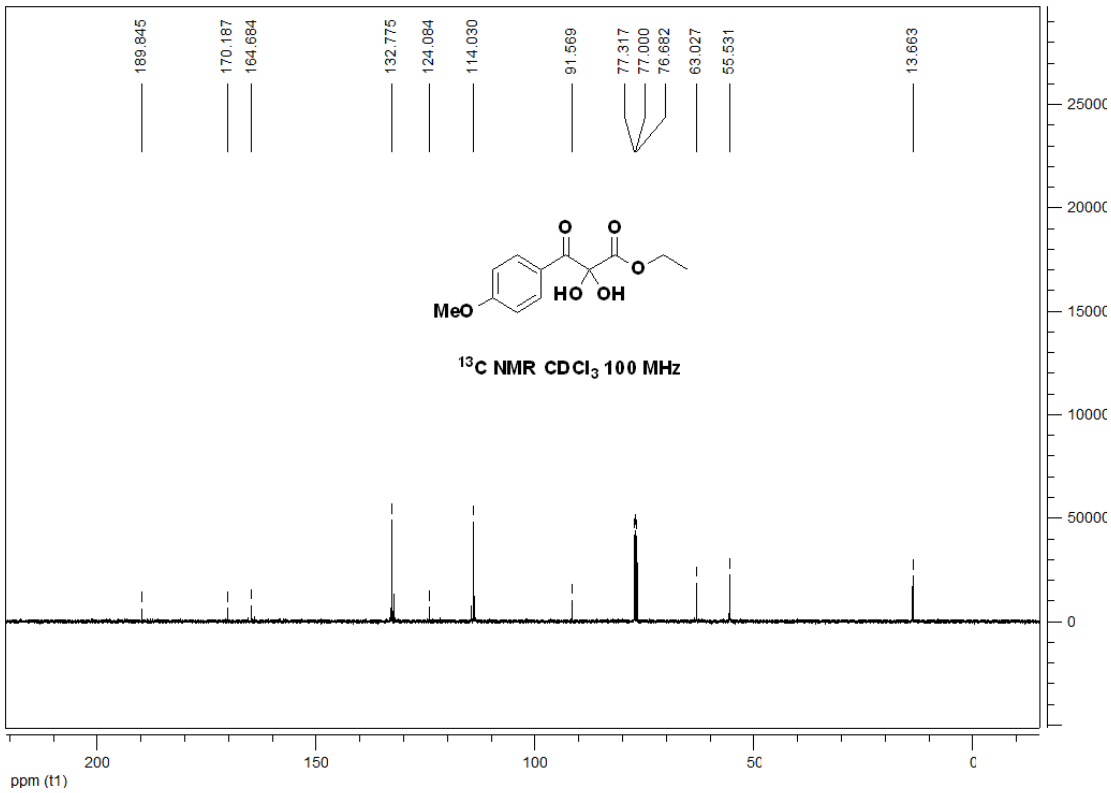
Ethyl 2,3-dioxo-3-(*p*-tolyl)propanoate (2h)



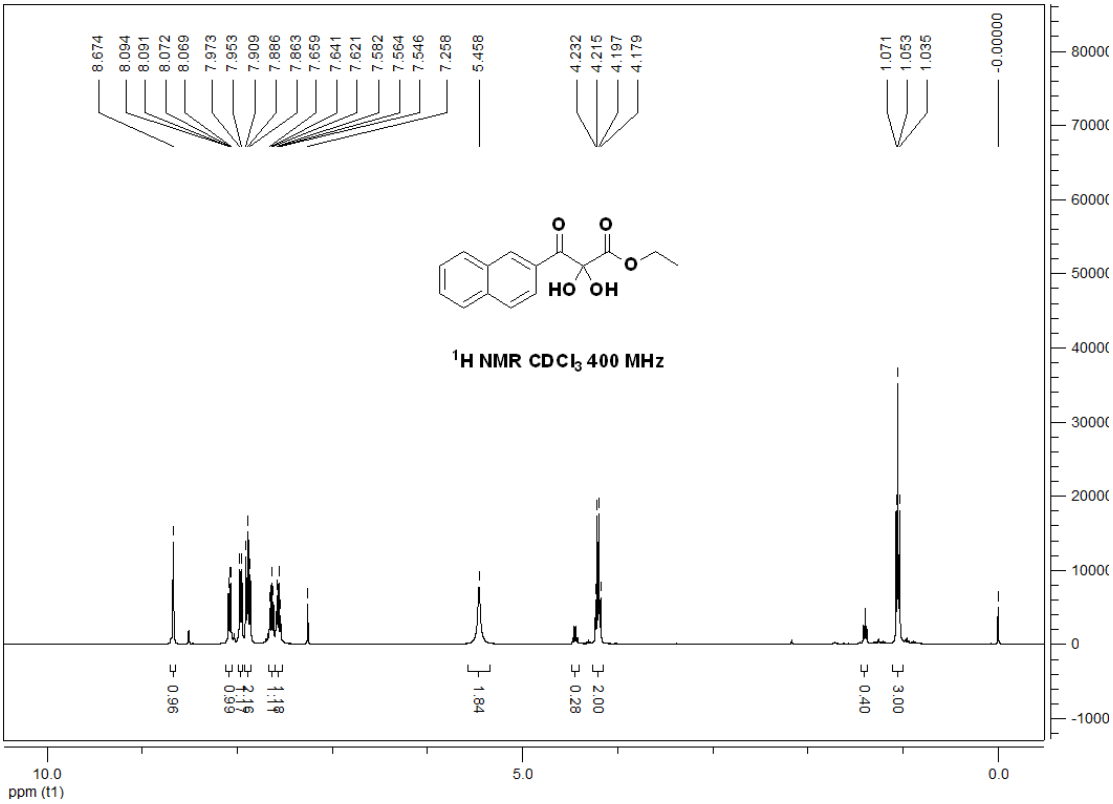


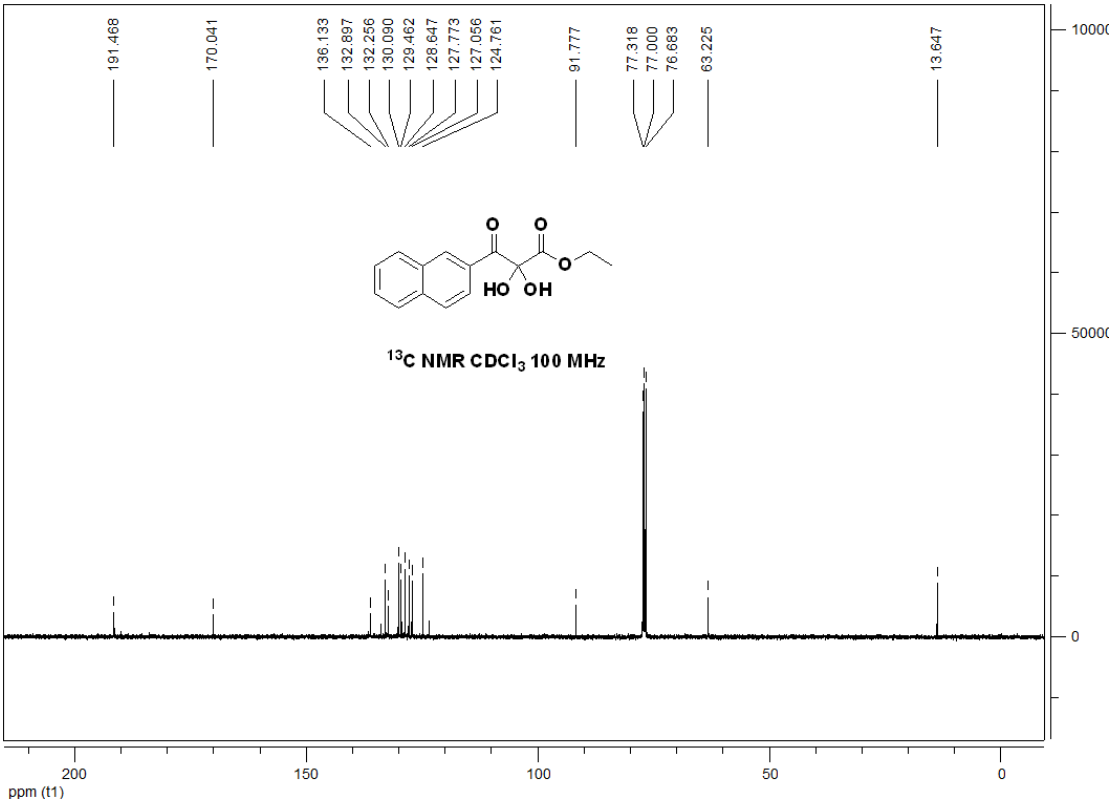
Ethyl 3-(4-methoxyphenyl)-2,3-dioxopropanoate (2i)

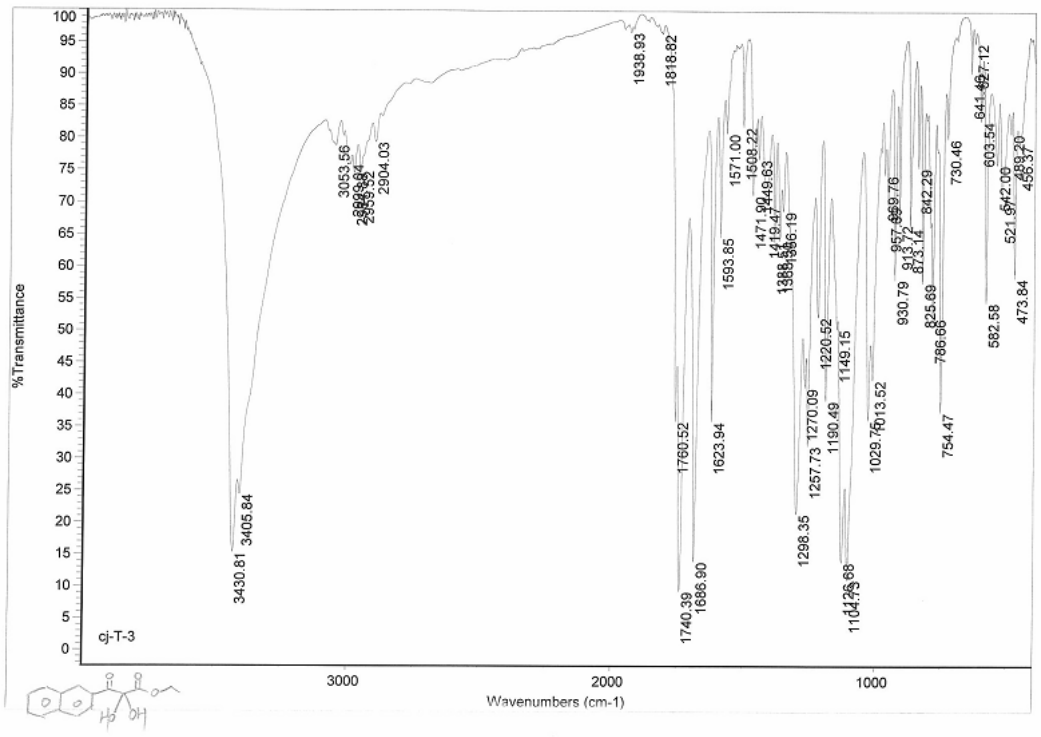


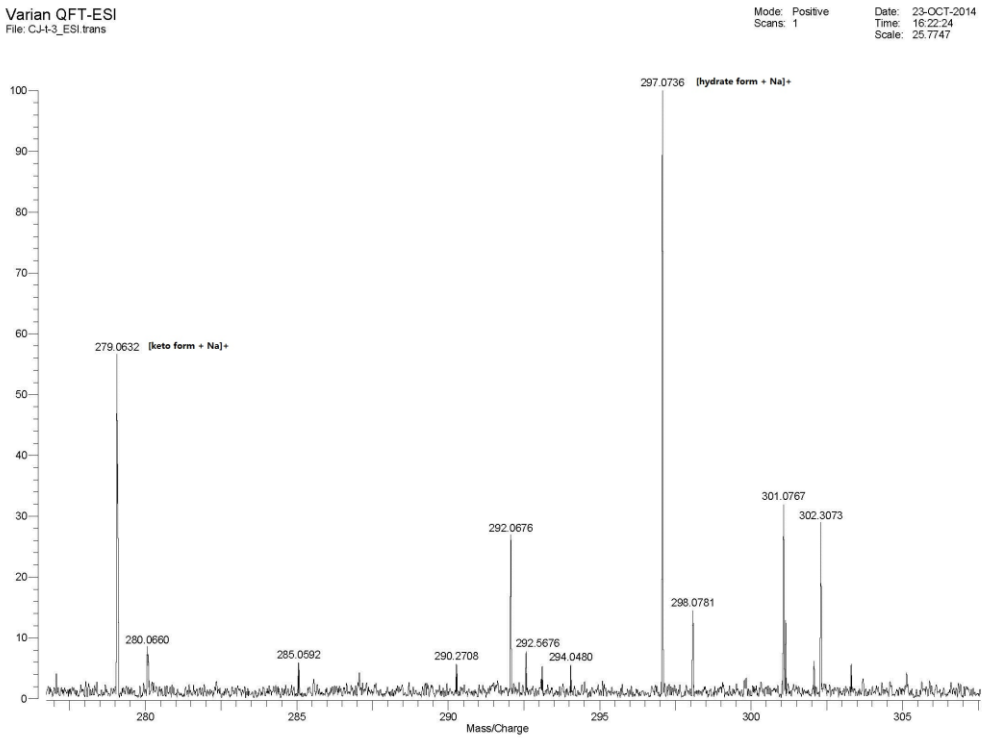


Ethyl 3-(naphthalen-2-yl)-2,3-dioxopropanoate (2j)






%Transmittance
Wavenumbers (cm-1)
cj-T-3
3430.81
3405.84
3053.56
2904.03
1938.93
1818.82
1760.52
1740.39
1686.90
1623.94
1593.85
1571.00
1508.22
1449.63
1298.35
1257.73
1220.52
1190.49
1149.15
1029.76
1013.52
930.79
842.29
825.69
786.66
754.47
730.46
603.54
582.58
473.84


Varian QFT-ESI
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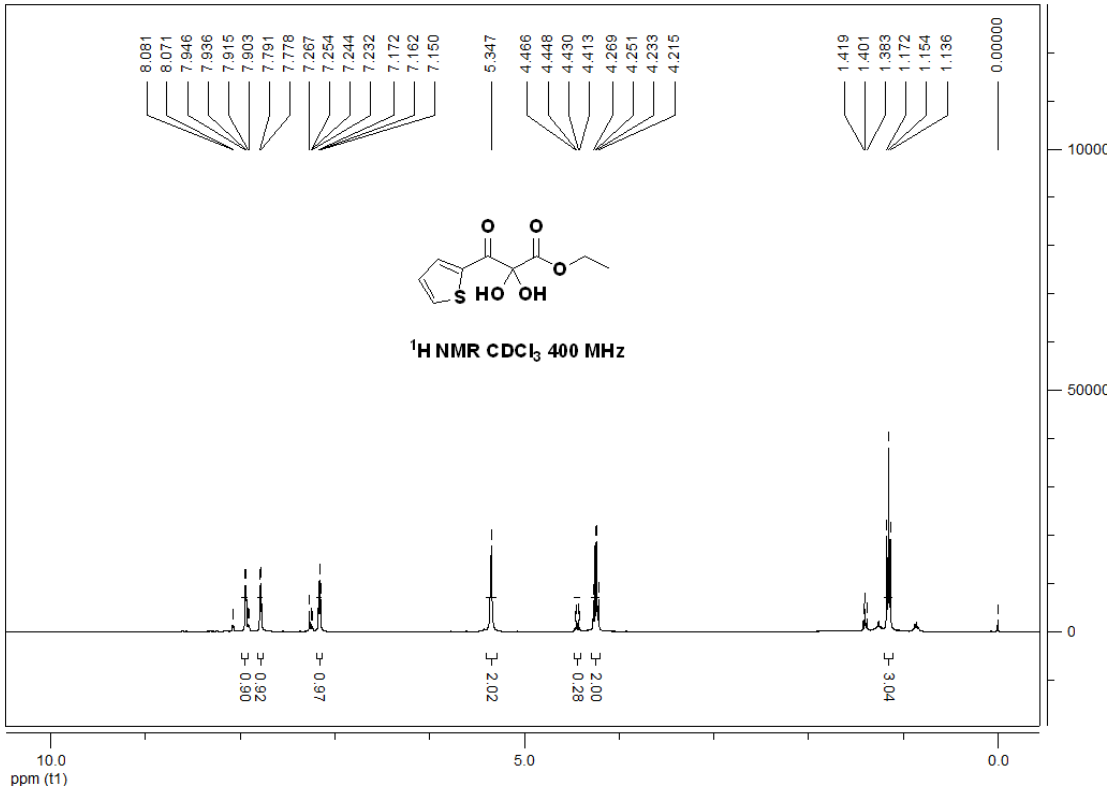
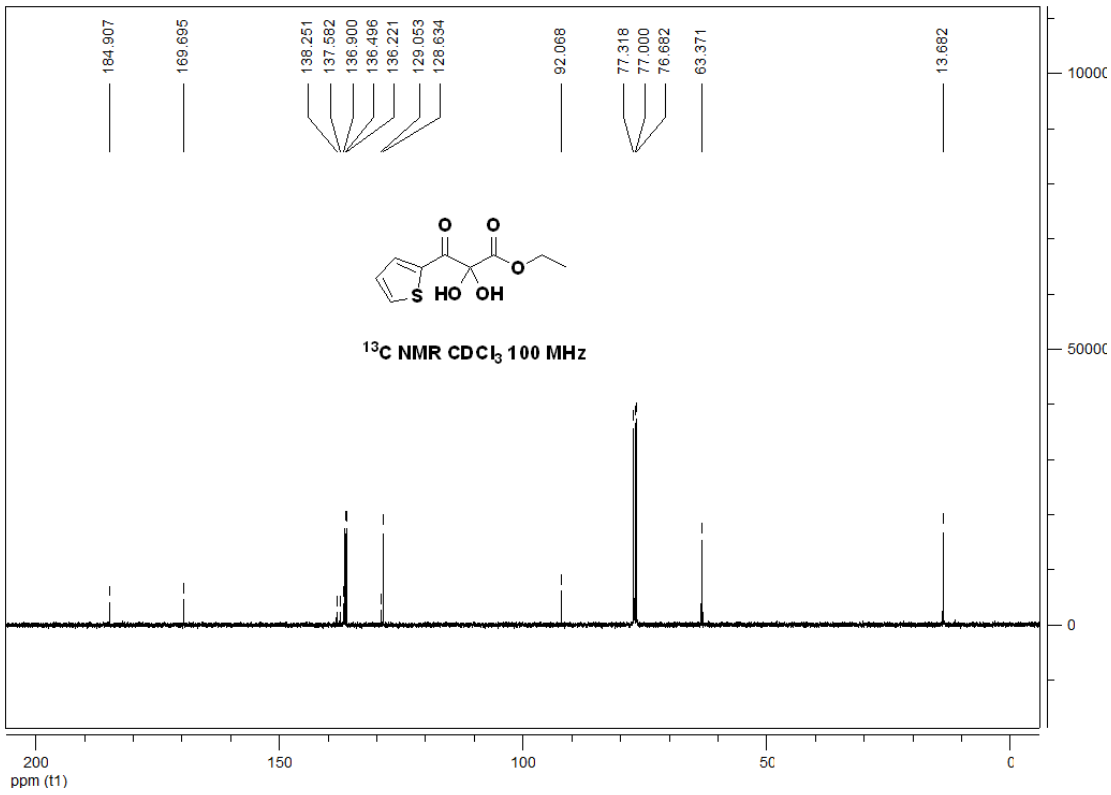
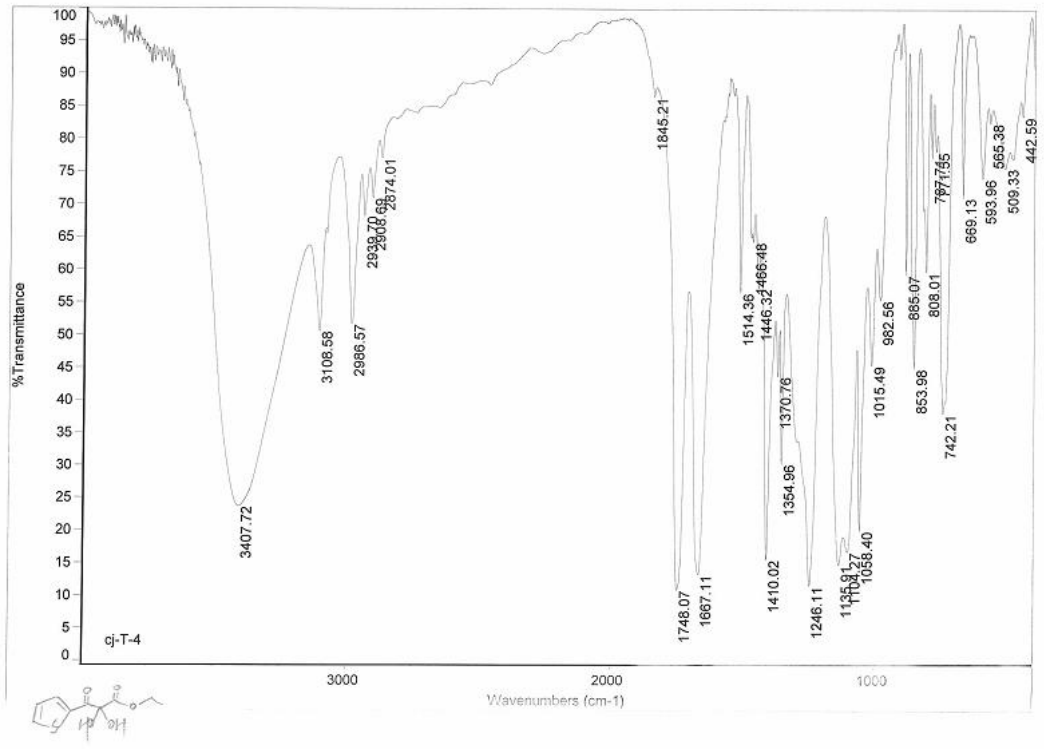
Mode: Positive
Scans: 1
Date: 23-OCT-2014
Time: 16:22:24
Scale: 25.7747
297.0736 [hydrate form + Na]+
279.0632 [keto form + Na]+
301.0767
302.3073
292.0676
298.0781
280.0660
285.0592
290.2708
292.5676
294.0480
Mass/Charge

Ethyl 2,3-dioxo-3-(thiophen-2-yl)propanoate (2k)






%Transmittance
Wavenumbers (cm-1)
cj-T-4
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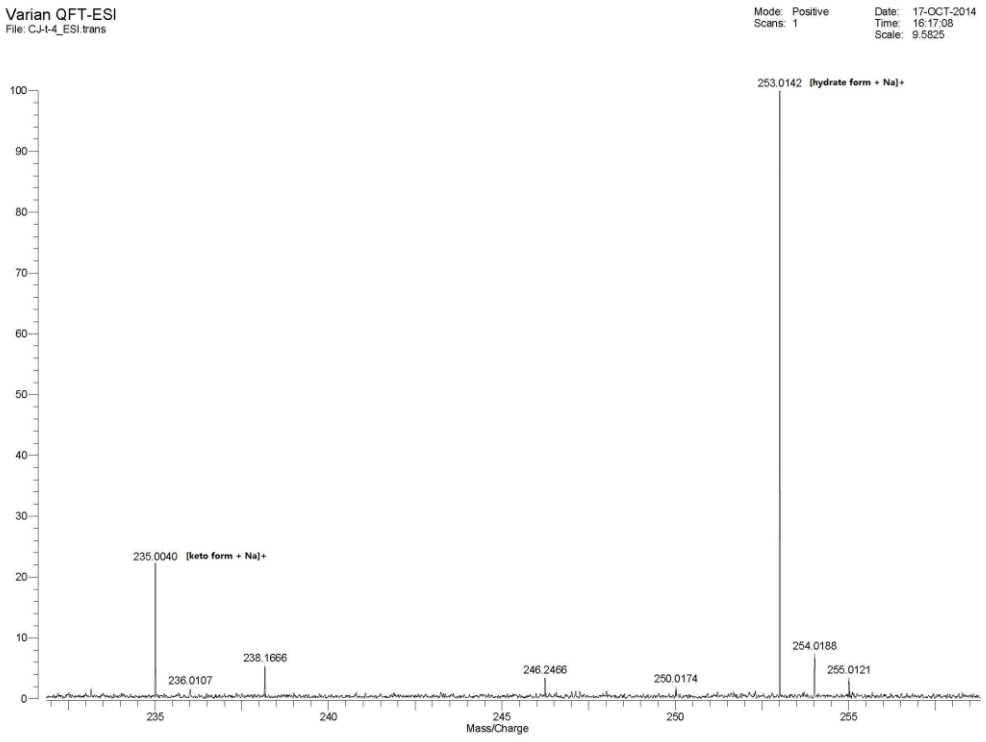
3108.58
2986.57
2939.70
2908.68
2874.01
1845.21
1748.07
1667.11
1514.36
1466.48
1446.32
1410.02
1370.76
1354.96
1246.11
1135.91
1104.27
1058.40
1015.49
982.56
885.07
853.98
808.01
787.75
742.21
669.13
593.96
565.38
509.33
442.59


Varian QFT-ESI
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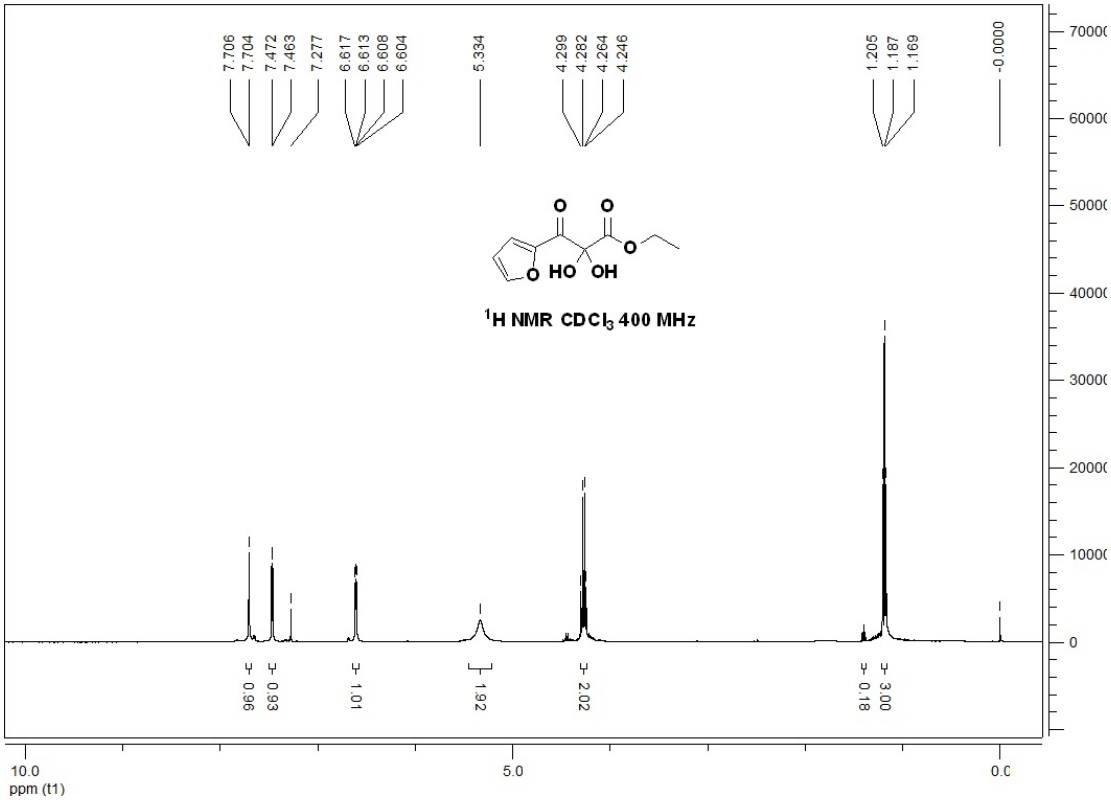
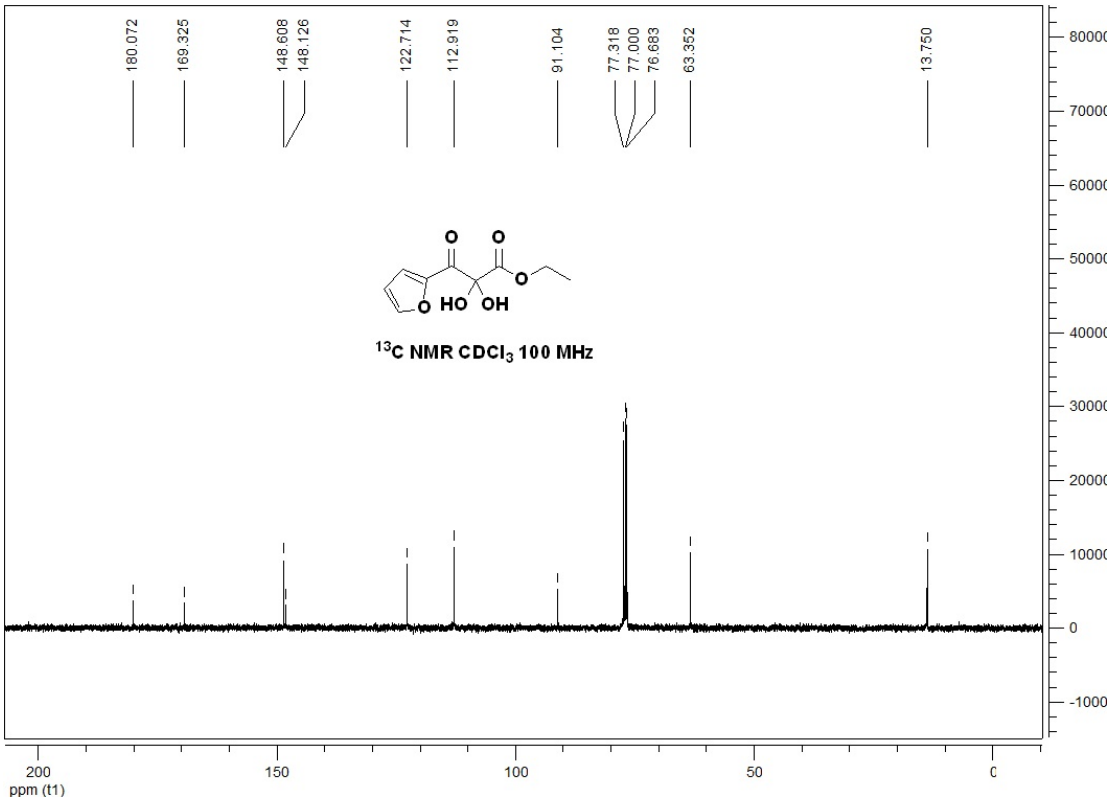
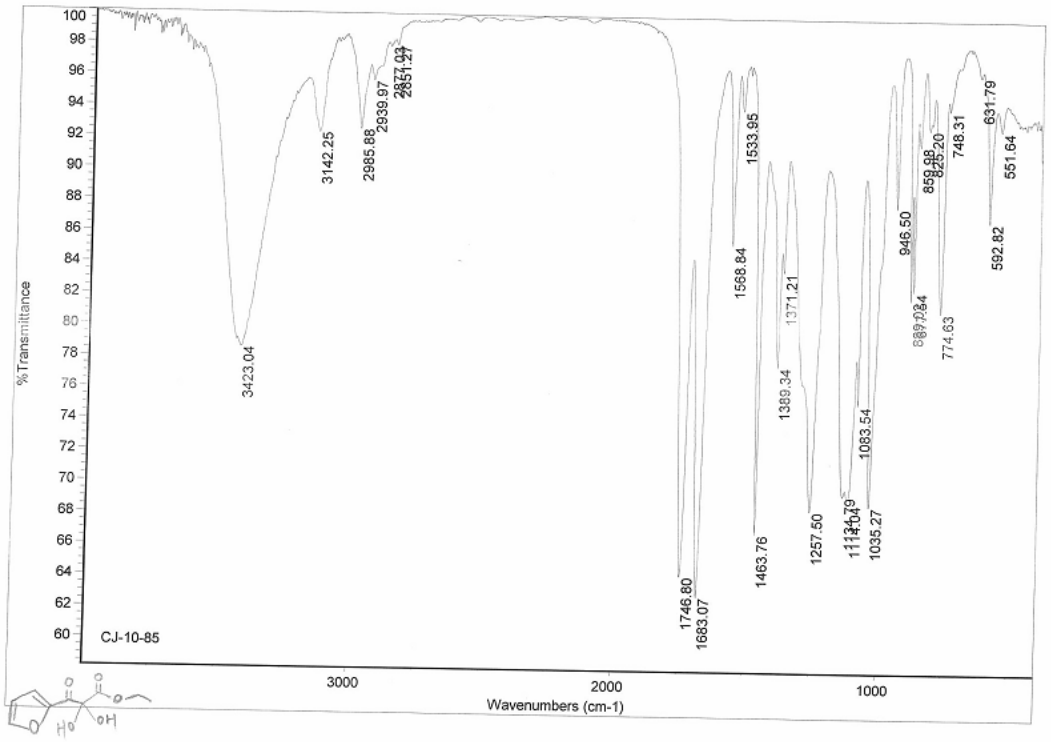
Mode: Positive
Scans: 1
Date: 17-OCT-2014
Time: 16:17:08
Scale: 9.5825
253.0142 [hydrate form + Na]+
235.0040 [keto form + Na]+
238.1666
236.0107
246.2466
250.0174
254.0188
255.0121
Mass/Charge

Ethyl 3-(furan-2-yl)-2,3-dioxopropanoate (2l)






%Transmittance
Wavenumbers (cm-1)
CJ-10-85
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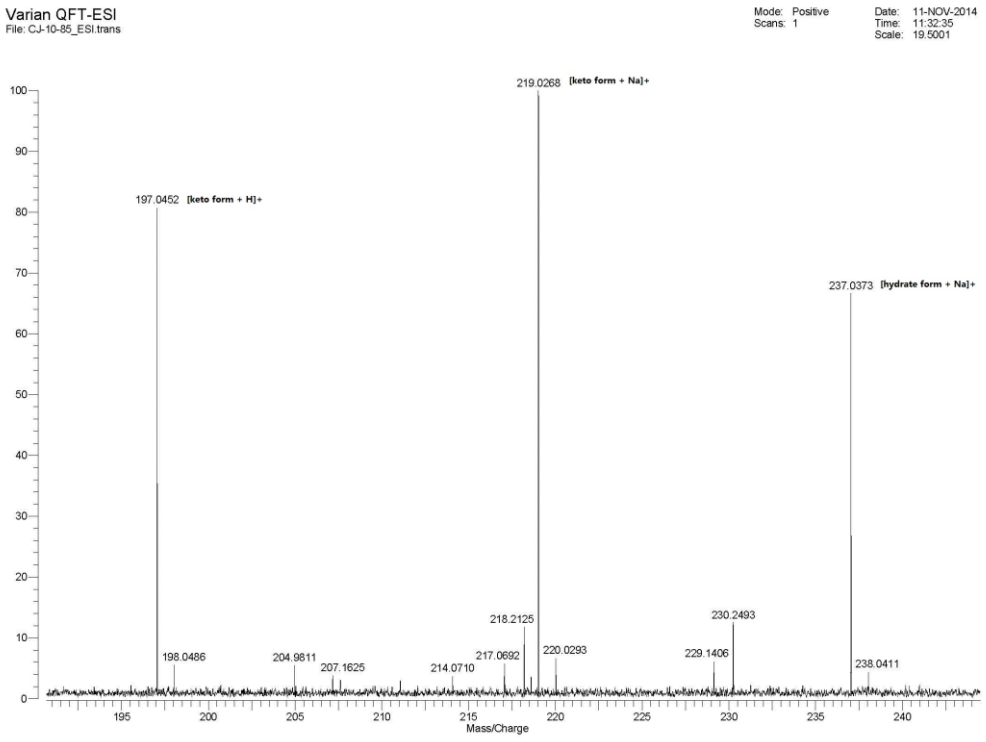
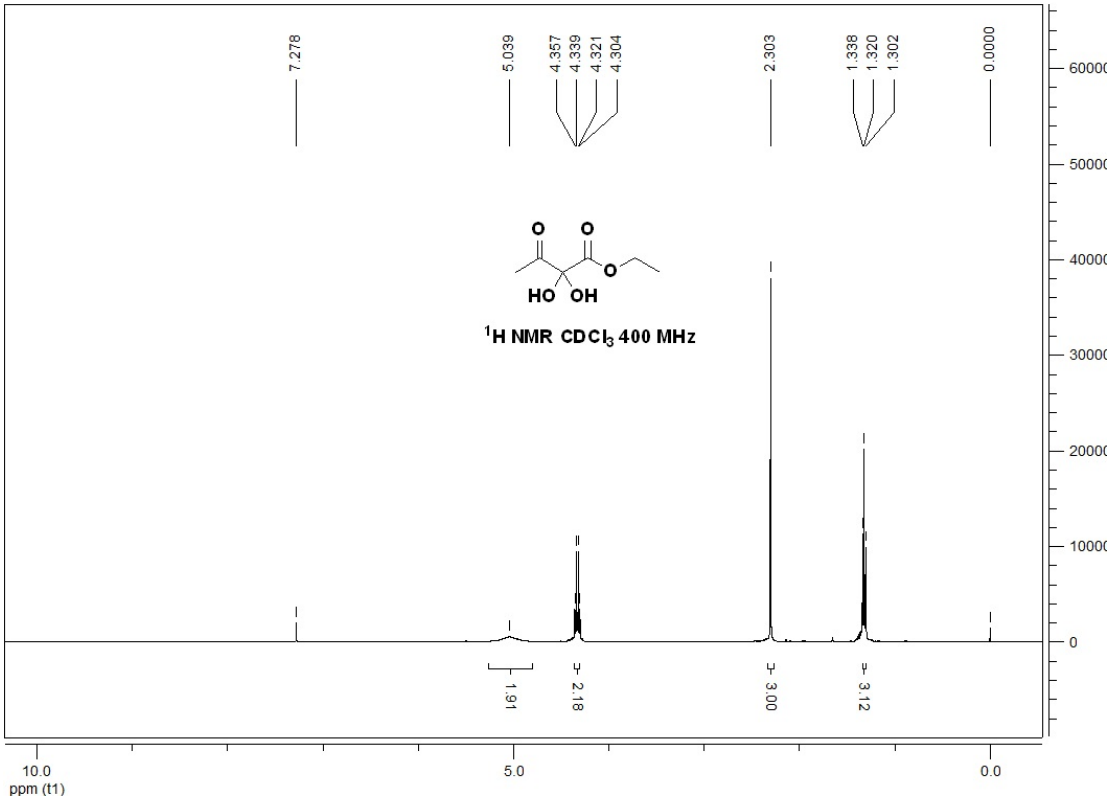
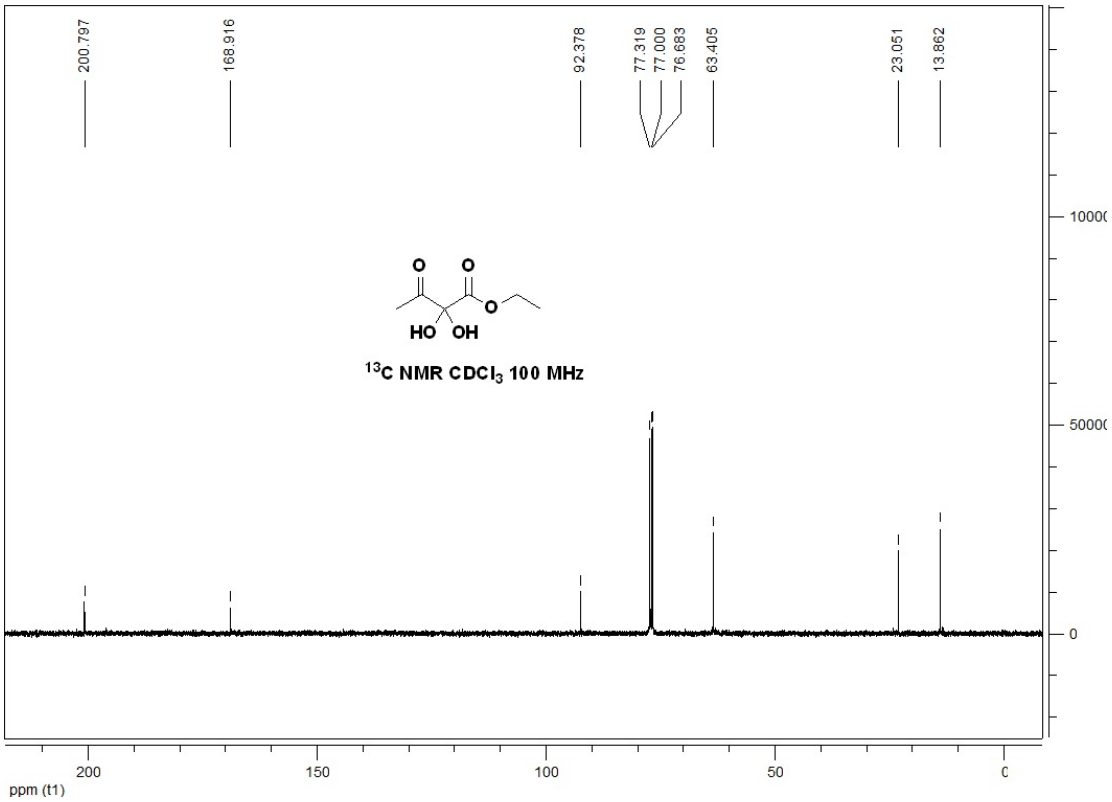
3142.25
2985.88
2939.97
2857.03
2851.27
1746.80
1683.07
1568.84
1533.95
1463.76
1389.34
1371.21
1257.50
1134.79
1114.04
1083.54
1035.27
946.50
889.04
859.08
825.20
774.63
748.31
631.79
592.82
551.64

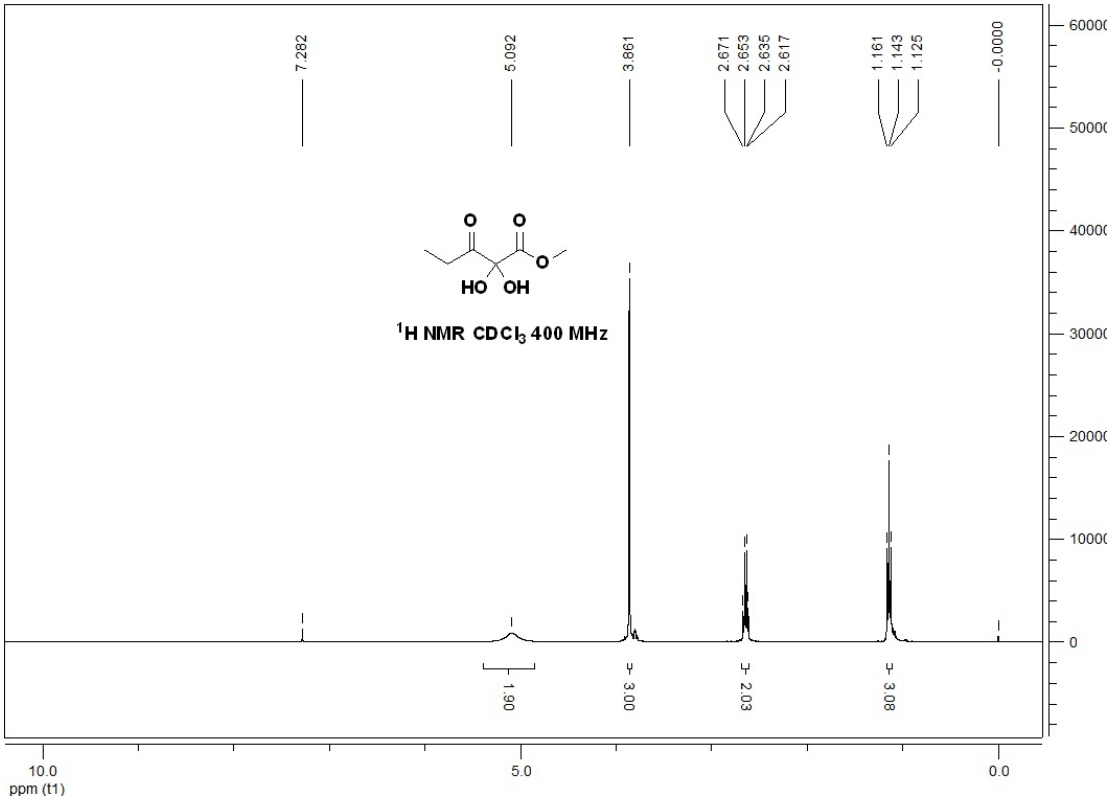

Varian QFT-ESI
File: CJ-10-85_ESI.trans
Mode: Positive
Scans: 1
Date: 11-NOV-2014
Time: 11:32:35
Scale: 19.5001
219.0268 [keto form + Na]+
197.0452 [keto form + H]+
237.0373 [hydrate form + Na]+
198.0486
204.9811
207.1625
214.0710
217.0692
218.2125
220.0293
229.1406
230.2493
238.0411
Mass/Charge

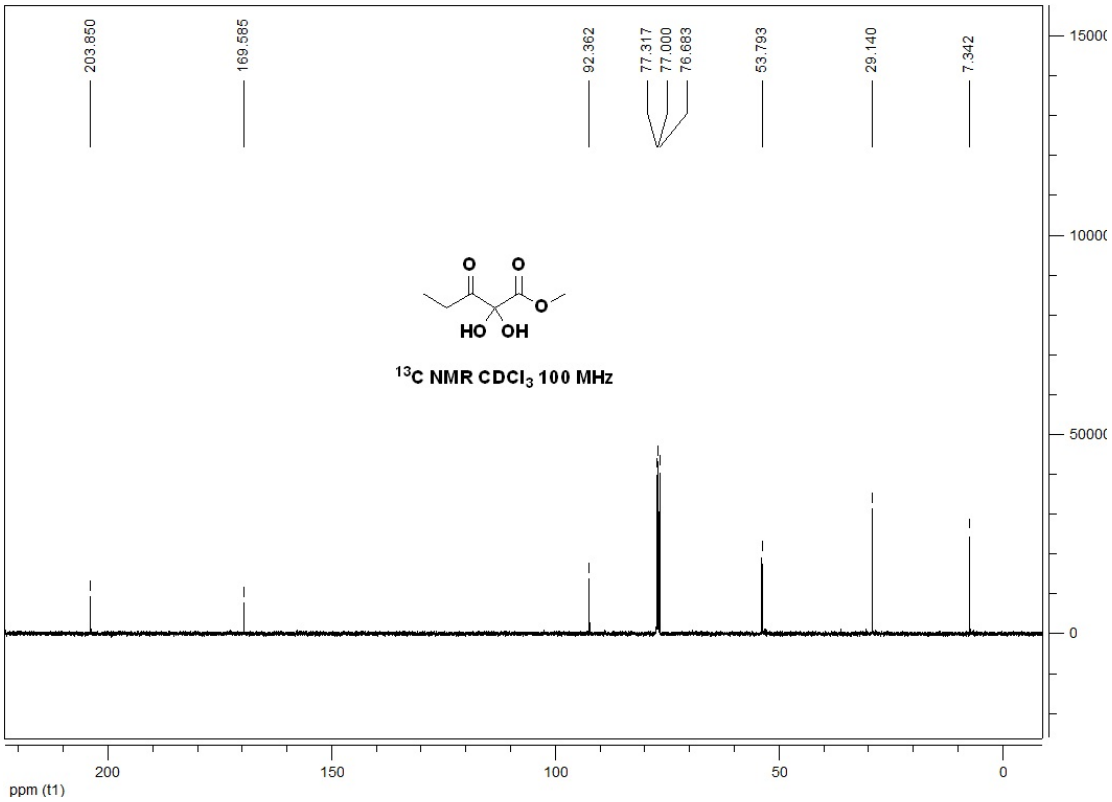
Ethyl 2,3-dioxobutanoate (2m)



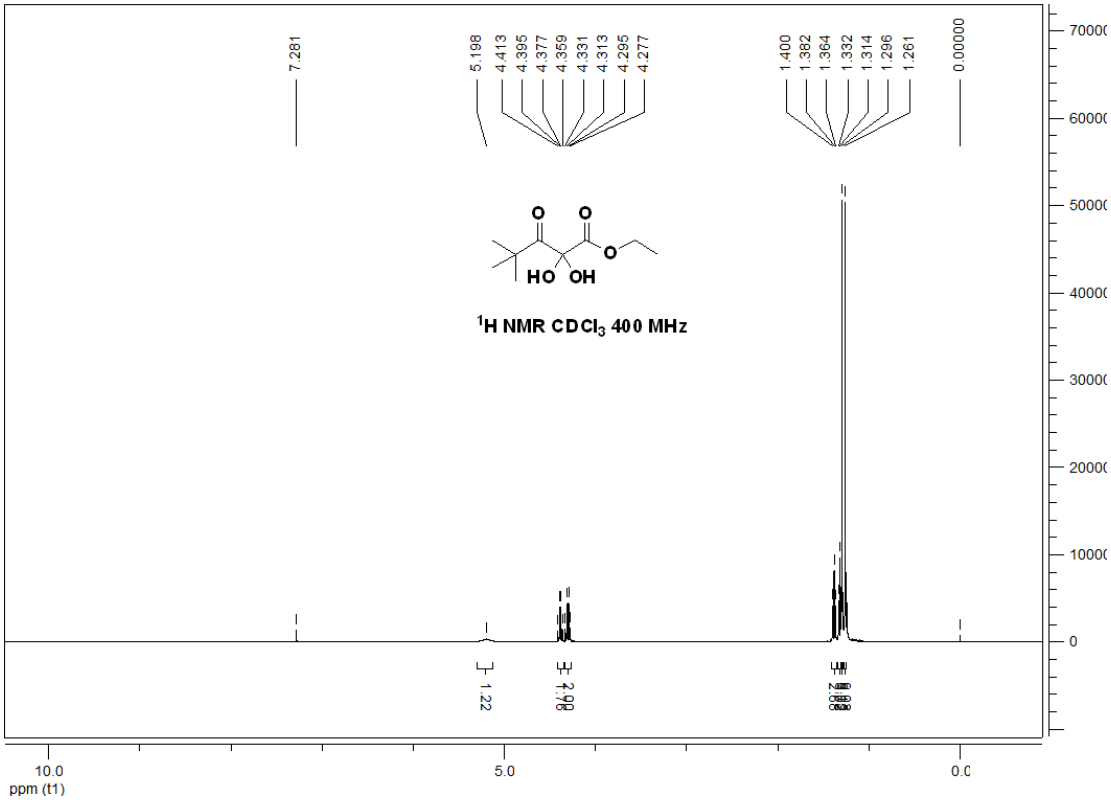


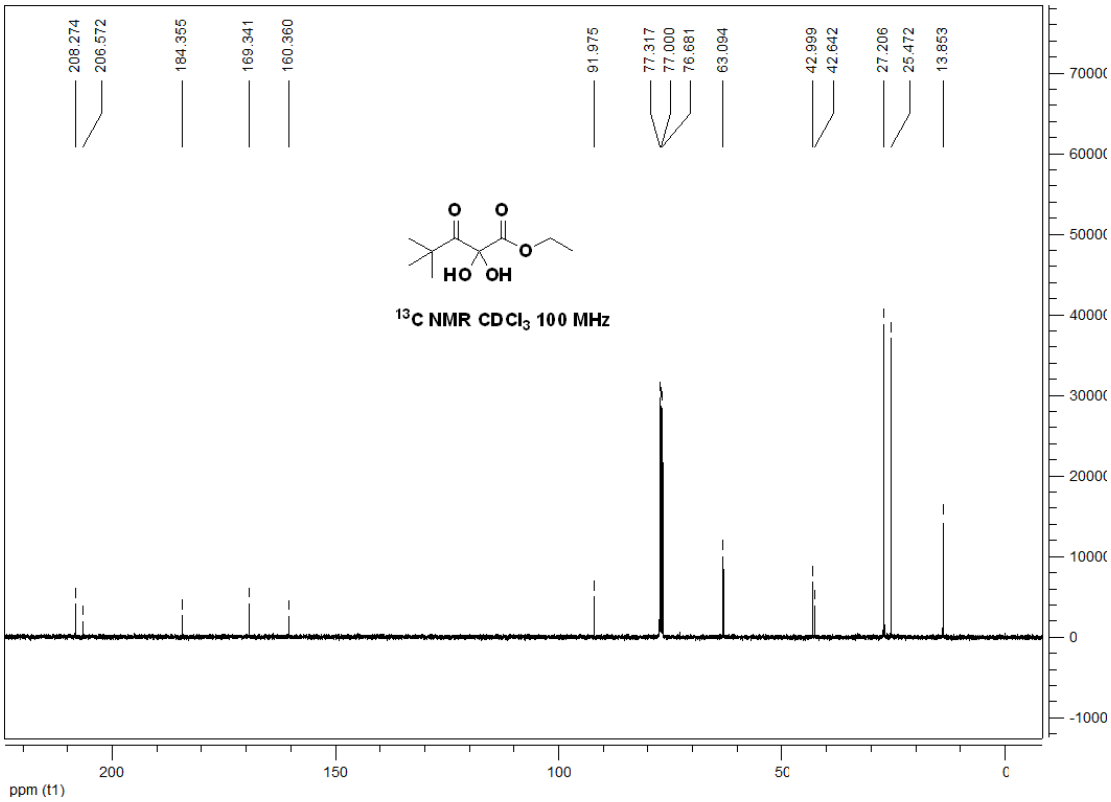
Methyl 2,3-dioxopentanoate (2n)



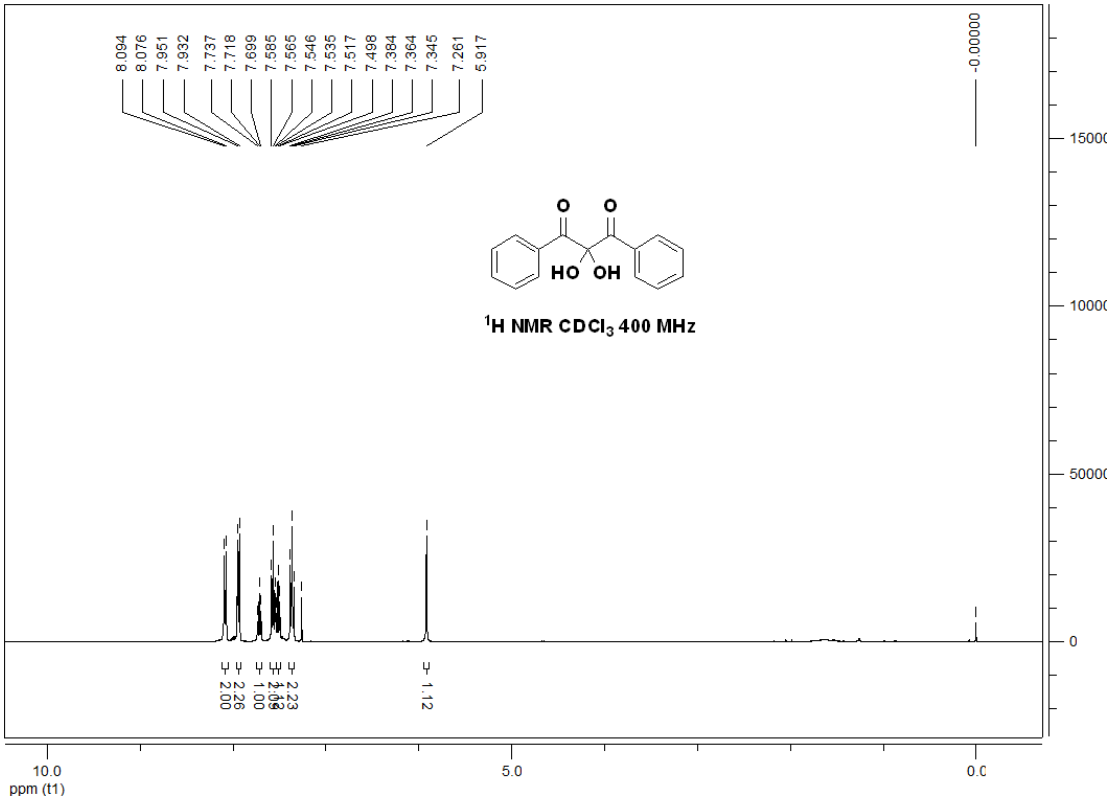


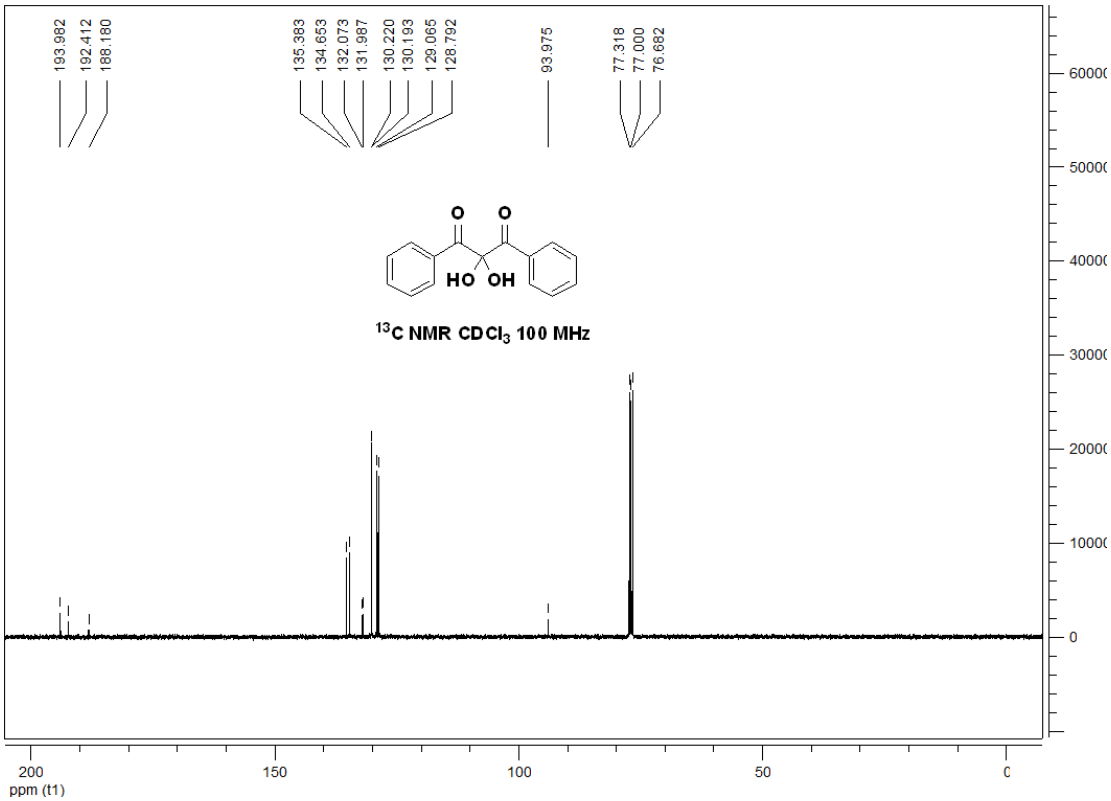
Ethyl 4,4-dimethyl-2,3-dioxopentanoate (2o)



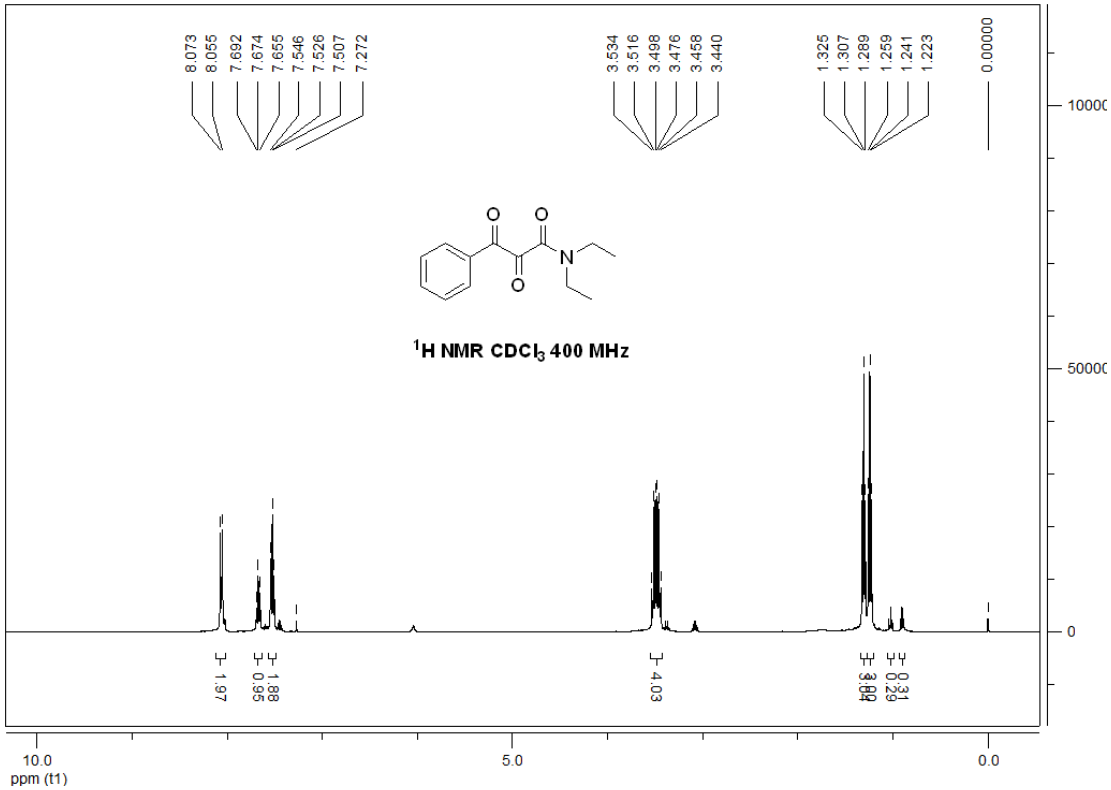


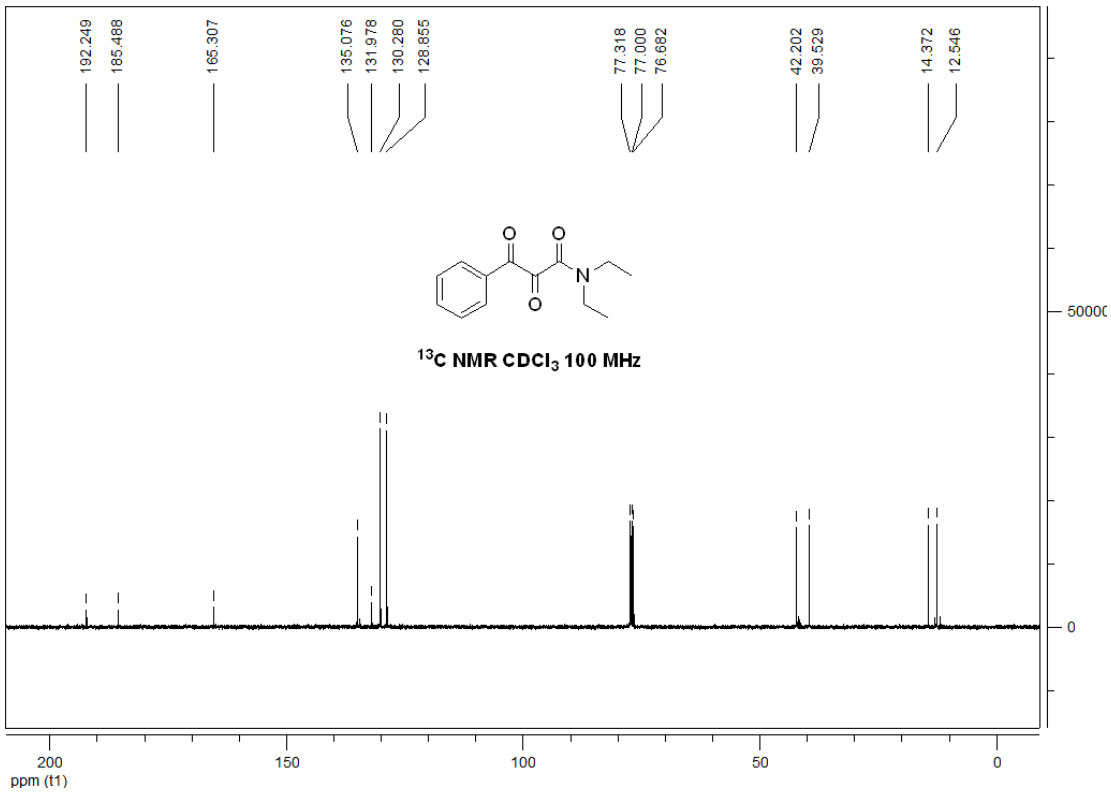
1,3-Diphenylpropane-1,2,3-trione (2p)



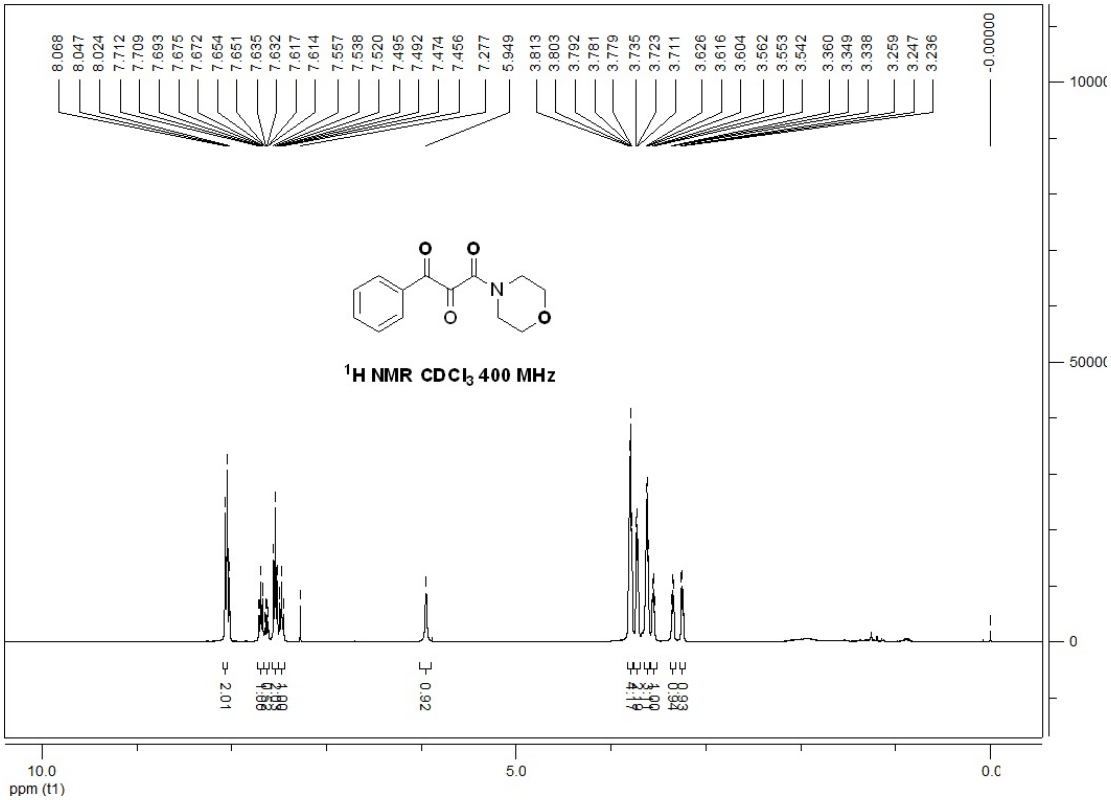


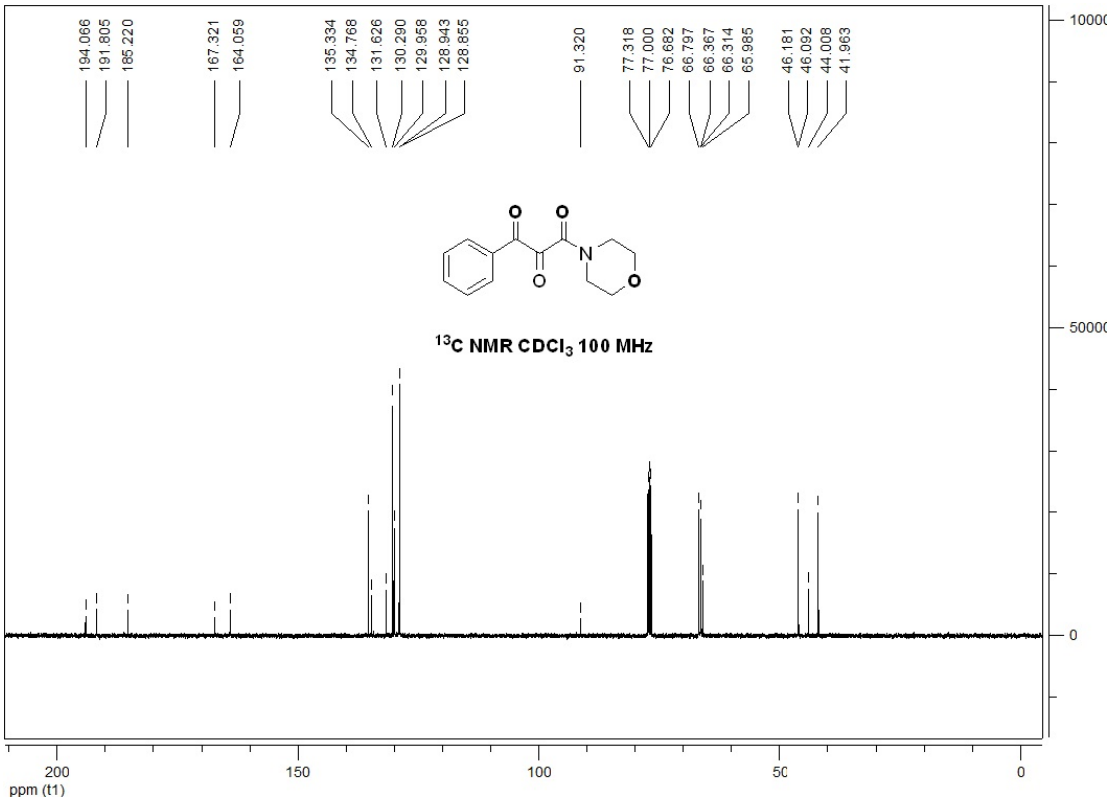
N,*N*-Diethyl-2,3-dioxo-3-phenylpropanamide (2q)

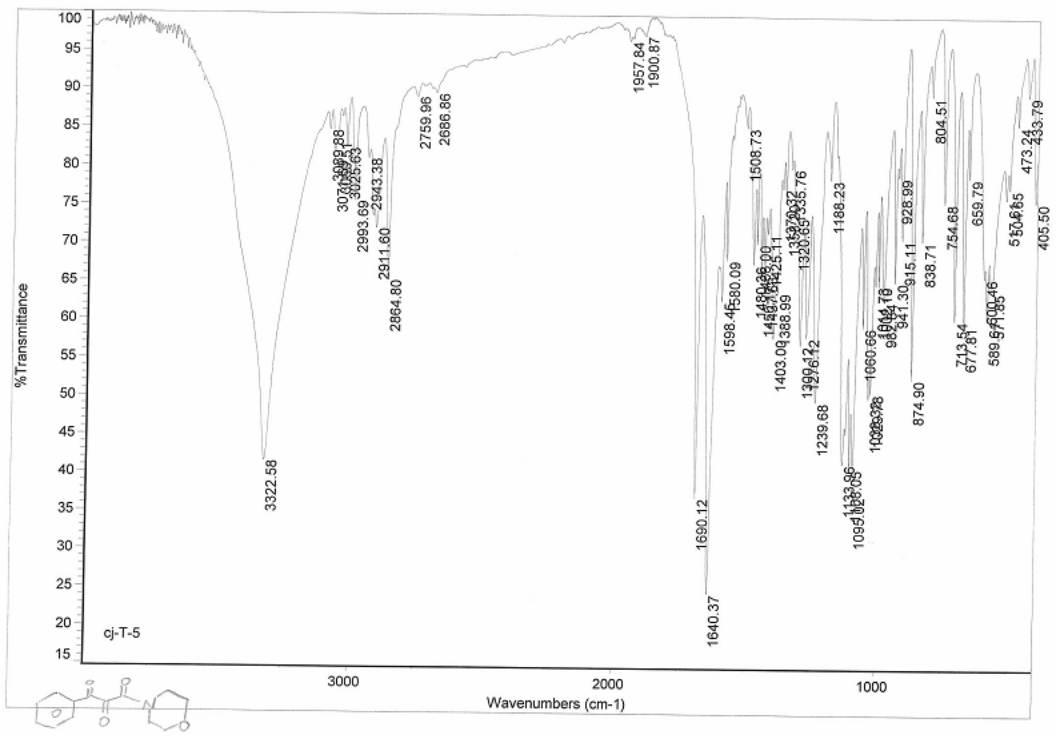




1-Morpholino-3-phenylpropane-1,2,3-trione (2r)






%Transmittance
Wavenumbers (cm-1)
cj-T-5
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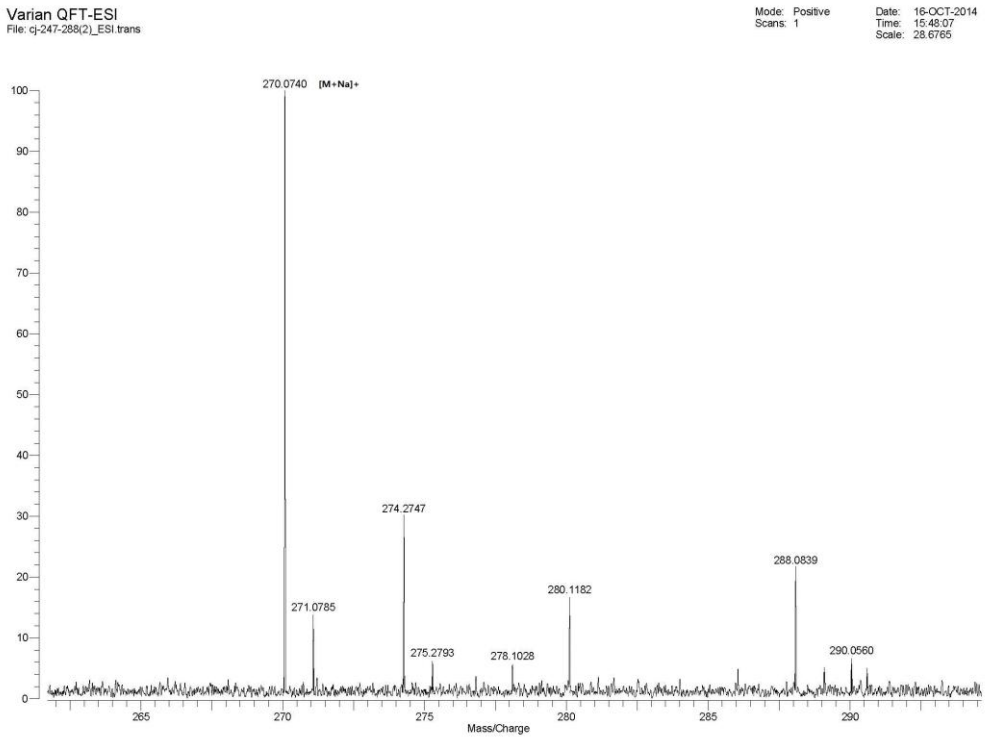
2993.69
2943.38
2911.60
2864.80
2759.96
2686.86
1957.84
1900.87
1690.12
1640.37
1598.45
1580.09
1508.73
1403.09
1388.99
1320.65
1239.68
1188.23
1060.66
941.30
915.11
874.90
838.71
804.51
754.68
713.54
677.81
659.79
589.97
550.65
504.85
473.24
433.79
405.50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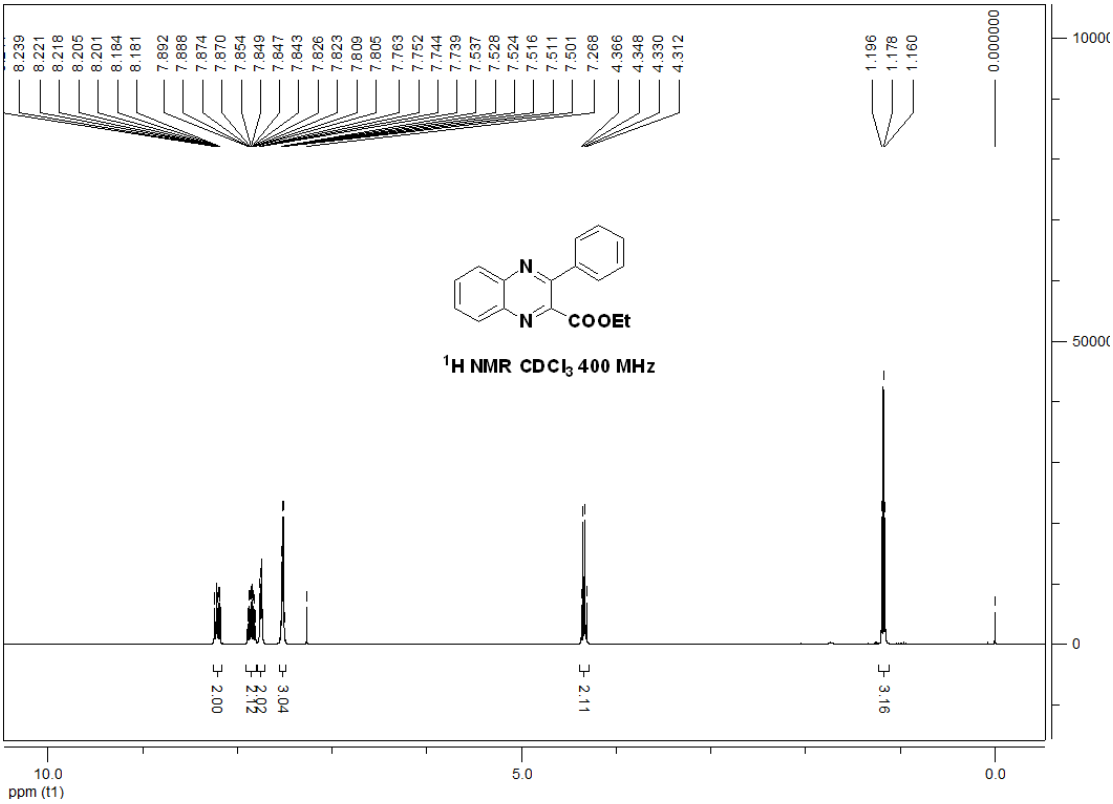
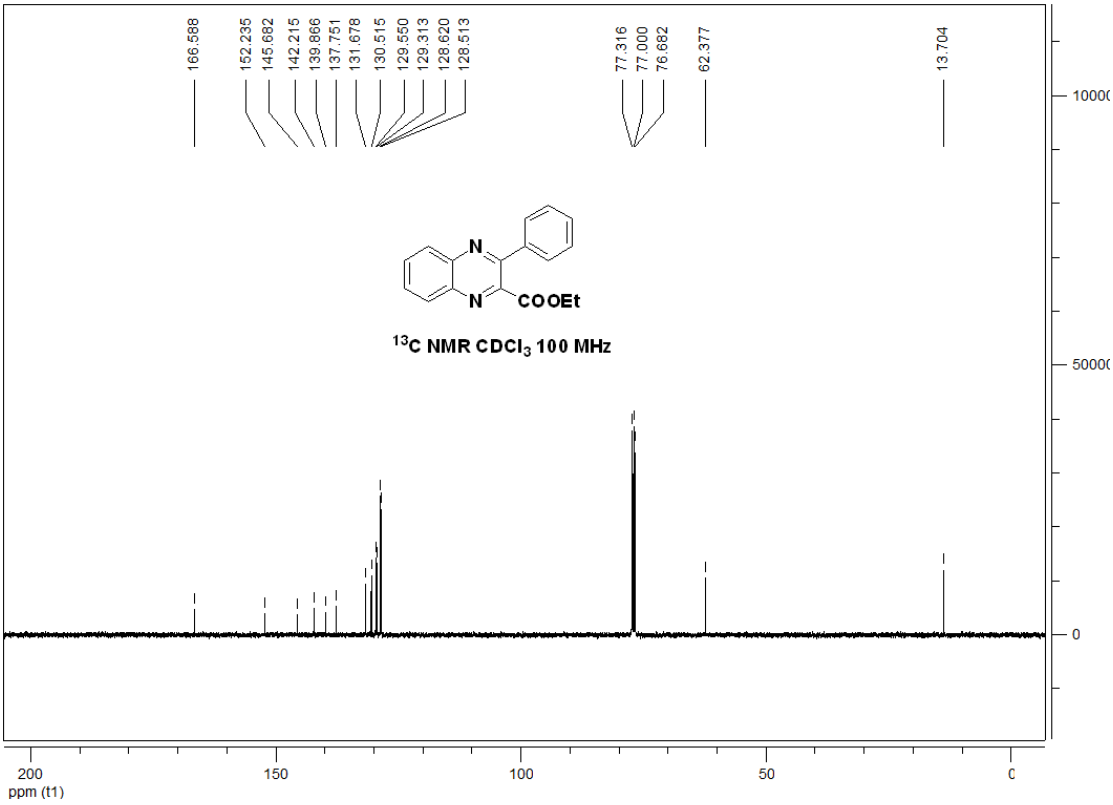
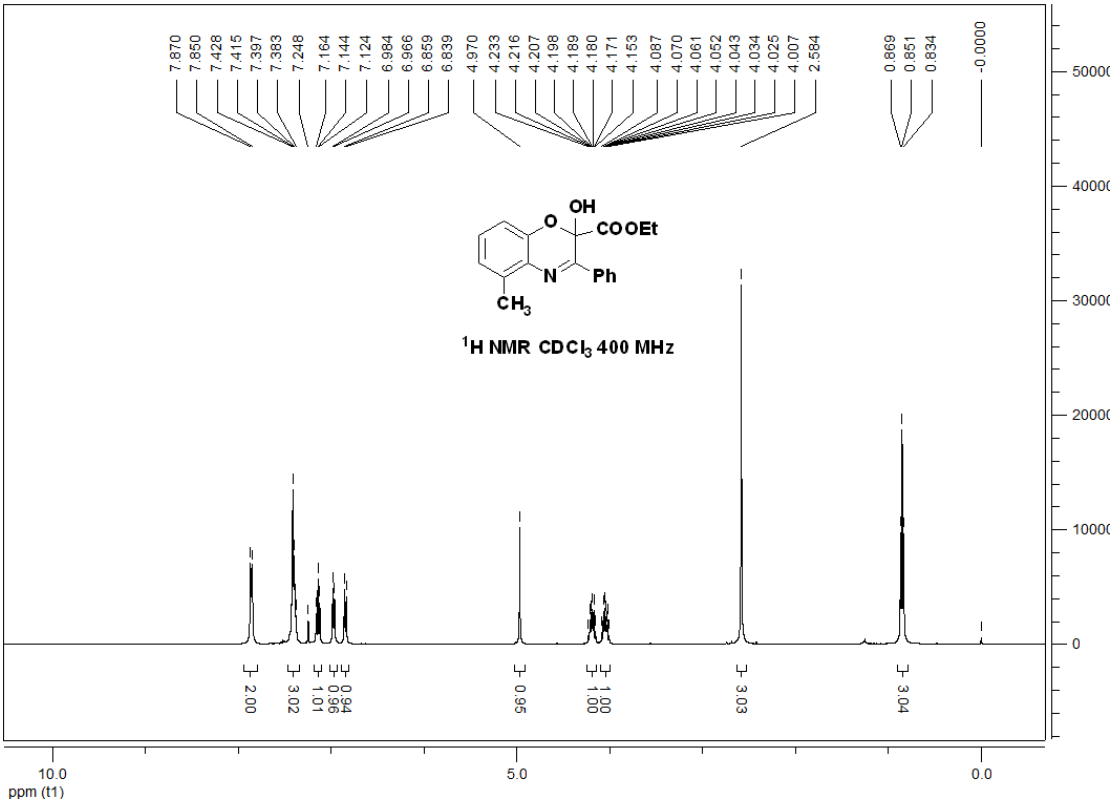
Mode: Positive
Scans: 1
Date: 16-OCT-2014
Time: 15:48:07
Scale: 28.6765
270.0740 [M+Na]+
274.2747
271.0785
275.2793
278.1028
280.1182
288.0839
290.0560
Mass/Charge

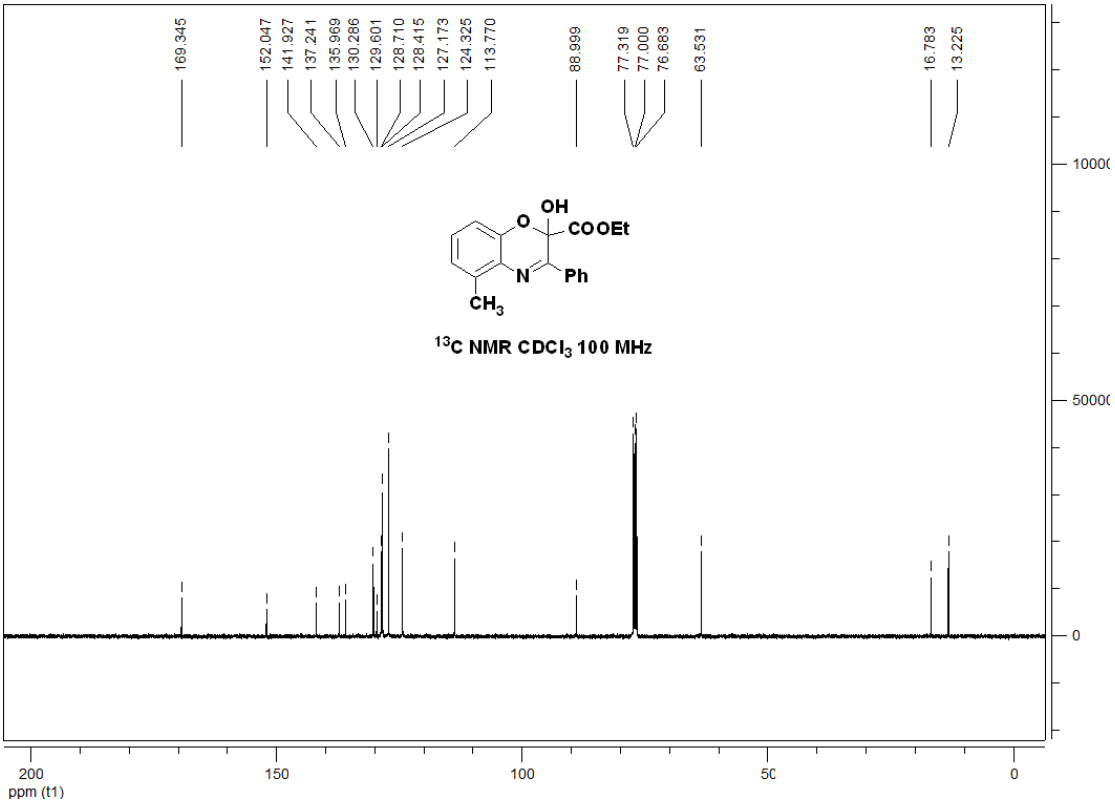
Ethyl 3-phenylquinoxaline-2-carboxylate (3a)

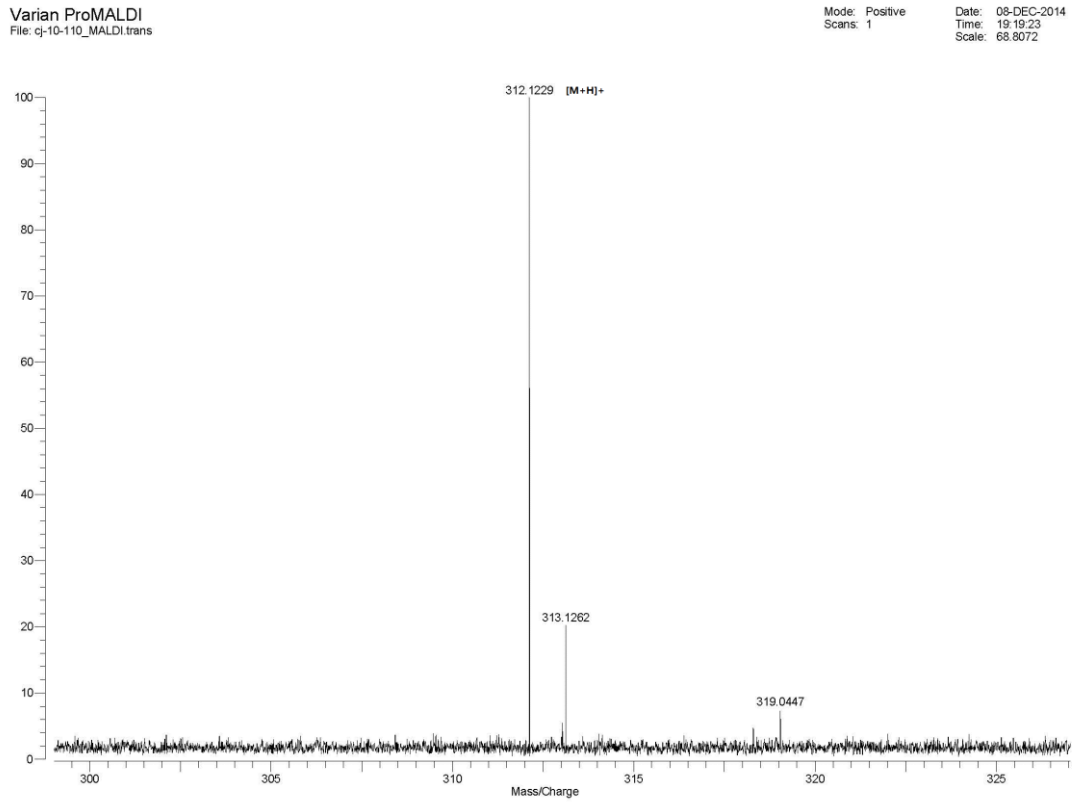


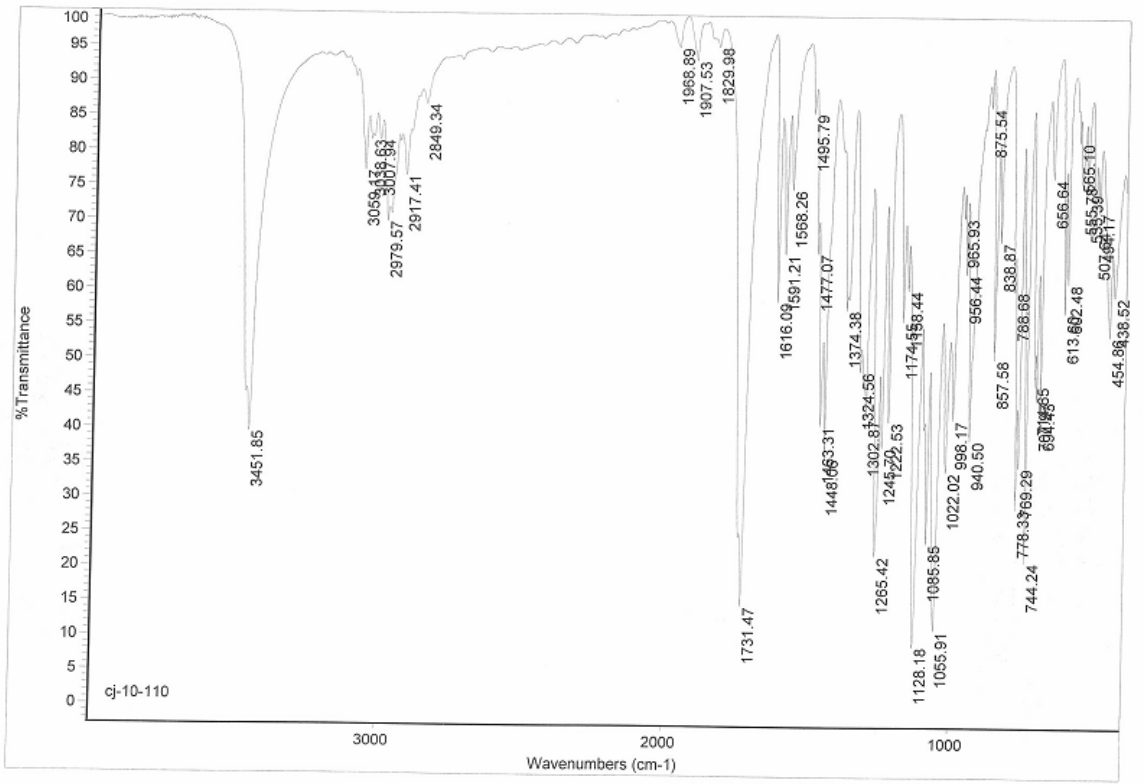


Ethyl 2-hydroxy-5-methyl-3-phenyl-2*H*-benzo[*b*][1,4]oxazine-2-carboxylate (3b)

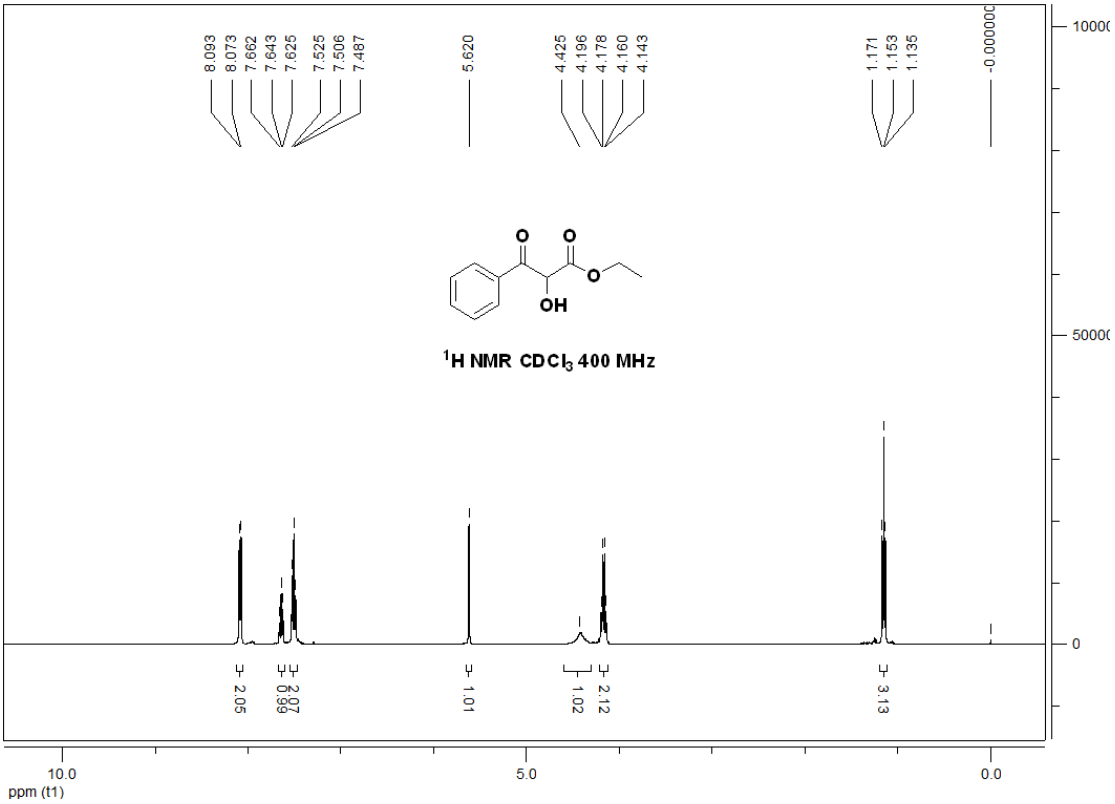


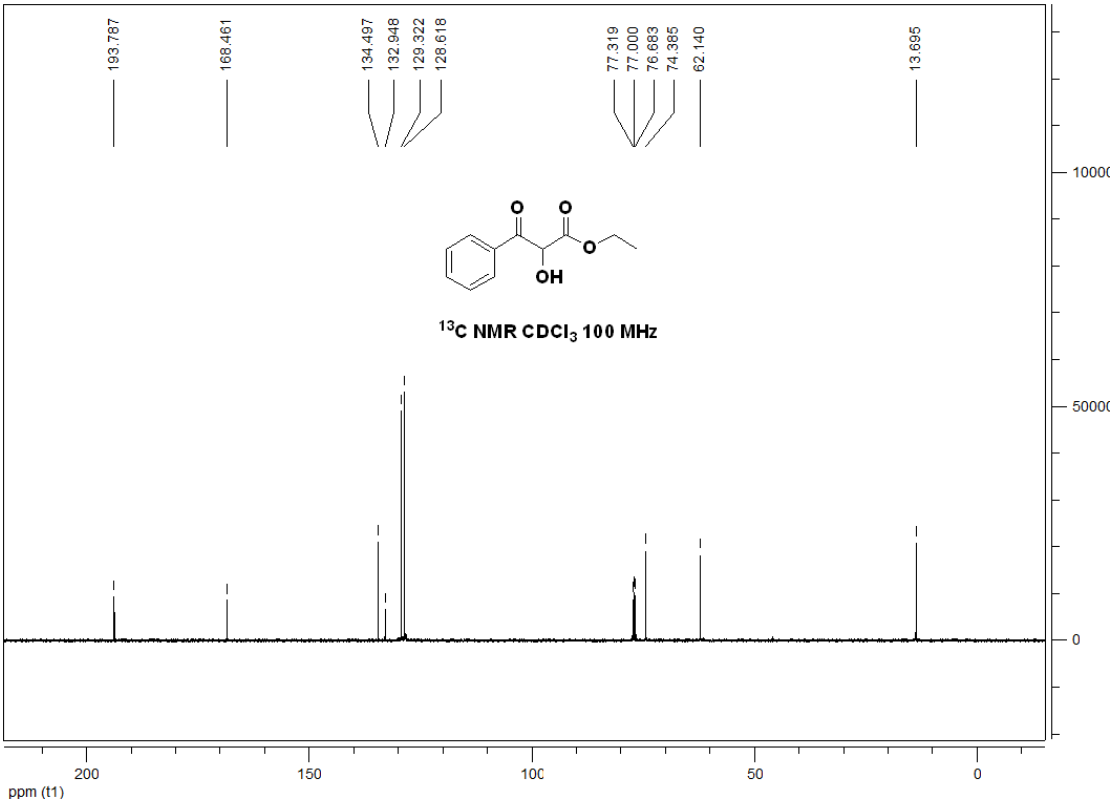



Varian ProMALDI
File: cj-10-110_MALDI.trans
Mode: Positive
Scans: 1
Date: 08-DEC-2014
Time: 19:19:23
Scale: 68.8072
312.1229 [M+H]+
313.1262
319.0447
Mass/Charge


%Transmittance
cj-10-110
Wavenumbers (cm-1)
3451.85
2979.57
2917.41
2849.34
1968.89
1907.53
1829.98
1731.47
1616.09
1591.21
1568.26
1495.79
1477.07
1374.38
1324.56
1265.42
1128.18
1085.85
1055.91
1022.02
998.17
940.50
875.54
857.58
838.87
788.68
744.24
656.64
613.60
454.86

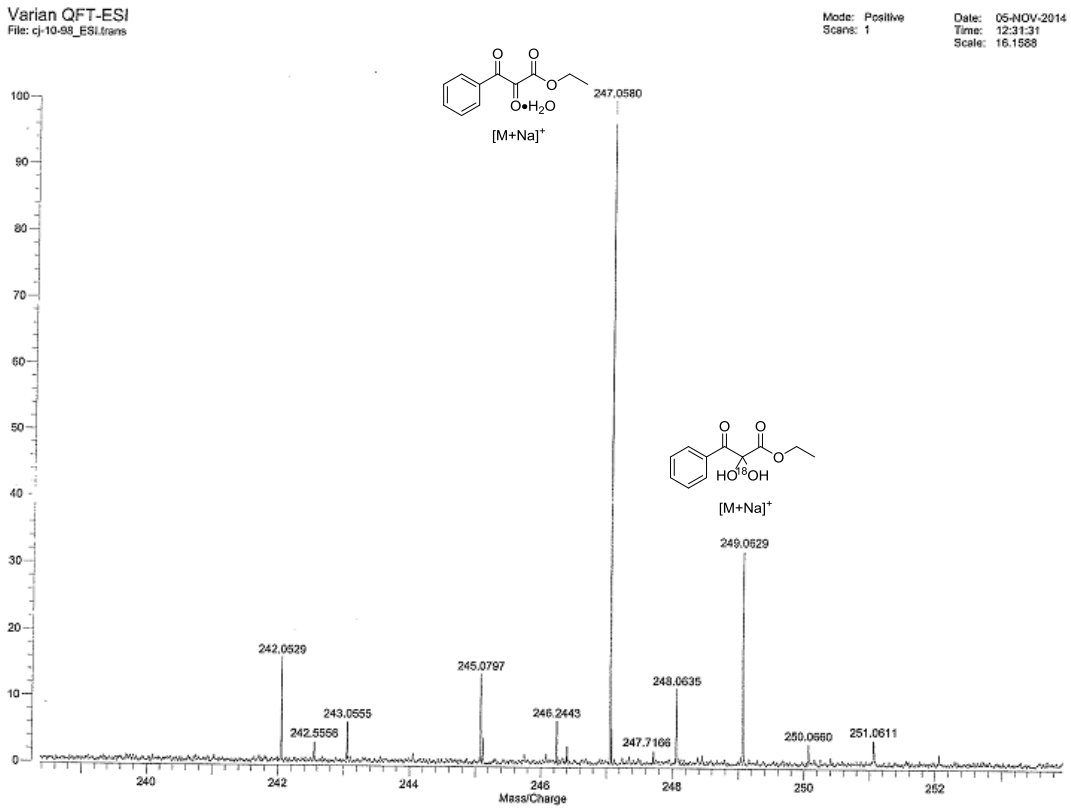
Ethyl 2-hydroxy-3-oxo-3-phenylpropanoate (4a)





Tracking experiment using ^{18}O-labeled PhIO:

The peak 247.0580 indicates ^{16}O-**2a**, and the peak 249.0629 indicates ^{18}O-**2a**.



Tracking experiment using ^{18}O-labeled H_2O as reaction additive:

The peak 247.0576 indicates ^{16}O-**2a**. The peak 249.0661 indicates ^{18}O-labeled-**2a**.