Electronic supplementary information

Efficient Synthesis of Organic Thioacetates in Water

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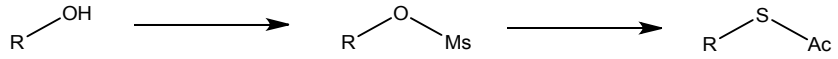
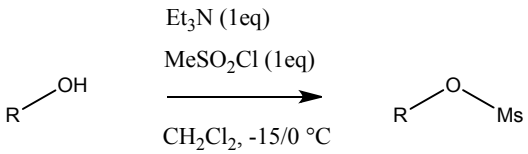
1. Total reaction scheme



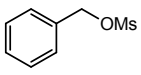
2. General procedure for the synthesis of organic mesylates



All the mesylates were synthetized starting from alcohol following the same procedures reported in literature. [1,2,3] The reactions were almost quantitative after work up and the freshly prepared product were used directly for the next step. All GC-MS and NMR spectra were comparable to those reported in literature, otherwise the other products were characterized by GC-MS and NMR spectroscopy.

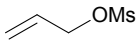
In a one neck round bottom flask, 4.6 mmol of the corresponding alcohol were dissolved in 20 ml of CH_2Cl_2 and the mixture cooled down at 0 °C. 4.6 mmol of triethylamine were added under stirring. After that, 4.6 mmol of methane sulfonyl chloride were added dropwise under stirring. The mixture was stirred open flask for one hour and half. The reaction was quenched with 2 ml of HCl 1N and the solution washed with $NaHCO_3$ saturated aqueous solution. The organic phase was extracted and washed three times with water. The organic phase was dried over Na_2SO_4. The solvent was removed under reduced pressure using a rotary evaporator to obtain a pink liquid. GC-MS Yield (>90%). A high vacuum pump was used to remove any residual solvent and minor impurities, after that the isolate yield was calculated in the usual way as percentage yield from the ratio between the actual yield and the theoretical yield. The theoretical yield was obtained considering the organic alcohol as the limiting reactant. The actual yield was obtained weighing the product after the mentioned work up.

3. General, Experimental and Analytical Information

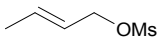


Benzyl mesylate (1a) , in accordance to general procedure, was prepared from benzyl alcohol (0.5g, 4.6 mmol) , triethylamine (1eq, 4.6mmol) and methane sulfonyl chloride (1 eq, 4.6 mmol), dissolved in 20 mL of CH_2Cl_2 , under stirring at -15 °C. After 1 hour and half the reaction mixture was quenched with 2ml of HCl 1N and the solution washed with $NaHCO_3$ saturated aqueous solution. The organic phase was washed three times with water and dried over Na_2SO_4. The solvent was removed under vacuum to afford a pink yellow

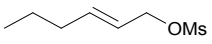
liquid in a quantitative yield. GC-MS spectra comparable to those reported in literature. NMR spectra comparable to those reported in literature.[4]



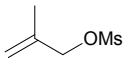
Allyl mesylate (2a) , in accordance to general procedure, was prepared from allyl alcohol (0.5g, 8.6 mmol) , triethylamine (1eq, 8.6 mmol) and methane sulfonyl chloride (1 eq, 8.6 mmol), dissolved in 20 mL of CH_2Cl_2 , under stirring at -15 °C. After 1 hour and half the reaction mixture was quenched with 2ml of HCl 1N and the solution washed with $NaHCO_3$ saturated aqueous solution. The organic phase was washed three times with water and dried over Na_2SO_4. The solvent was removed under vacuum to afford a pink yellow liquid in quantitative yield. ^{1}H-NMR: (300 MHz, $CDCl_3$, 25°C) δ = 3.02 (3H, s, CH_3), 4.72 (2H, d, ^3J = 6Hz, CH_2O), 5.38 (1H, dd, ^3J=10.3Hz, ^4J= 0.9Hz, CH_2=), 5.46 (1H, dd, ^3J=17.1Hz, ^4J= 1.3Hz,CH_2=), 5.97(1H, ddt, ^3J=17Hz, ^3J=10.4Hz, ^3J=6Hz, CH=). ^{13}C-NMR : (75 MHz, $CDCl_3$, 25°C) δ = 38.04 (CH_3-S), 70.38 (CH2-O), 120.79 (CH_2=), 130.48 (CH=CH_2).



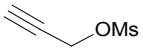
Trans-2-butenyl mesylate (3a) , in accordance to general procedure, was prepared from crotyl alcohol (0.5g, 6.9 mmol) , triethylamine (1eq, 6.9mmol) and methane sulfonyl chloride (1 eq, 6.9 mmol), dissolved in 20 mL of CH_2Cl_2 , under stirring at -15 °C. After 1 hour and half the reaction mixture was quenched with 2ml of HCl 1N and the solution washed with $NaHCO_3$ saturated aqueous solution. The organic phase was washed three times with water and dried over Na_2SO_4. The solvent was removed under vacuum to afford a pink yellow liquid in quantitative yield. GC-MS(ESI): m/z (%) , 150 (M^+, 0.4), 80 (11), 79 (15), 71 (42), 65 (11), 55 (100), 43 (27). ^{1}H-NMR: (300 MHz, $CDCl_3$, 25°C) δ = 1,77 (3H, d, ^3J=6.5Hz, CH_3CH), 3.00 (3H, s, CH_3S), 4.67 (2H, d, ^3J=6.9Hz, CH_2O), 5.58-5.68 (1H, m, CH=), 5.87-5.97(1H, m, CH=). ^{13}C-NMR : δ = 17.73 (CH_3-CH), 38.21 (CH_3S), 70.78 (CH_2S), 123.49 ($CHCH_2$), 134.57($CHCH_3$). NMR data comparable to those reported in literature.[5]



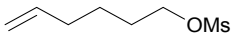
Trans-2-hexenyl mesylate (4a), in accordance to general procedure, was prepared from trans-hexenyl alcohol (0.5g, 4.9 mmol) , triethylamine (1eq, 6.9 mmol) and methane sulfonyl chloride (1 eq, 6.9 mmol), dissolved in 20 mL of CH_2Cl_2 , under stirring at -15 °C. After 1 hour and half the reaction mixture was quenched with 2ml of HCl 1N and the solution washed with $NaHCO_3$ saturated aqueous solution. The organic phase was washed three times with water and dried over Na_2SO_4. The solvent was removed under vacuum to afford a pink yellow liquid in quantitative yield. GC-MS(ESI): m/z (%) , 149 (0.4), 82 (59), 79 (27), 67 (100), 57 (31), 55 (43), 41 (55). ^{1}H-NMR: (300 MHz, $CDCl_3$, 25°C): δ = 0.90 (3H, t, ^3J=7.3Hz, CH_3CH_2), 1.42 (2H, sextuplet, ^3J=7.4Hz, CH_2CH_3), 2.06 (2H, q, ^3J=7.2Hz, CH_2CH), 2.99 (3H, s, CH_3S),4.67 (2H, d, ^3J=6.8Hz, CH_2S), 5.58-5.63(1H, m, CH=), 5.88-5.93(1H, m, CH=). ^{13}C-NMR : (75 MHz, $CDCl_3$, 25°C) δ = 13.58 (CH_3-CH_2), 21.82 (CH_2CH_3), 34.21, 38.24, 70.93 (CH_2S), 122.27($CHCH_2S$), 139.58($CHCH_2CH_2$).



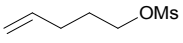
2-methylallyl mesylate (5a), in accordance to general procedure, was prepared from 2-methylallyl alcohol (0.5g, 6.9 mmol) , triethylamine (1eq, 6.9mmol) and methane sulfonyl chloride (1 eq, 6.9 mmol), dissolved in 20 mL of CH_2Cl_2 , under stirring at -15 °C. After 1 hour and half the reaction mixture was quenched with 2ml of HCl 1N and the solution washed with $NaHCO_3$ saturated aqueous solution. The organic phase was washed three times with water and dried over Na_2SO_4. The solvent was removed under vacuum to afford a pink yellow liquid in quantitative yield. GC-MS(ESI): m/z (%) , 150 (M^+, 10), 80 (31), 72 (21), 71 (100), 65 (23), 55 (69), 43 (54). ^{1}H-NMR: (300 MHz, $CDCl_3$, 25°C): δ = 1.81 (3H, s, CH_3C), 3.01 (3H, s, CH_3S), 4.61 (2H, s, CH_2), 5.09 (2H, d, ^2J=19.1Hz, CH_2=). ^{13}C-NMR : (75 MHz, $CDCl_3$, 25°C) δ = 19.09 (CH_3-C), 37.87 (CH_3-S), 73.37 (CH_2O), 116.08 (CH_2=), 138.17 (C=).



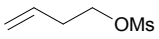
Prop-2-yn-1-ol mesylate (6b) , in accordance to general procedure, was prepared from propargyl alcohol (0.5g, 8.9 mmol) , triethylamine (1eq, 6.9mmol) and methane sulfonyl chloride (1 eq, 6.9 mmol), dissolved in 20 mL of CH_2Cl_2 , under stirring at -15 °C. After 1 hour and half the reaction mixture was quenched with 2ml of HCl 1N and the solution washed with $NaHCO_3$ saturated aqueous solution. The organic phase was washed three times with water and dried over Na_2SO_4. The solvent was removed under vacuum to afford a pink yellow liquid in quantitative yield. GC-MS spectra comparable to those reported in literature.[6] NMR spectra comparable to those reported in literature.[7]



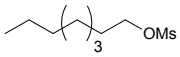
5-hexenyl mesylate (1c) , in accordance to general procedure, was prepared from 5-hexen-1-ol (0.5g, 4.9 mmol) , triethylamine (1eq, 4.9mmol) and methane sulfonyl chloride (1 eq, 4.9 mmol), dissolved in 20 mL of CH_2Cl_2 , under stirring at -15 °C. After 1 hour and half the reaction mixture was quenched with 2ml of HCl 1N and the solution washed with $NaHCO_3$ saturated aqueous solution. The organic phase was washed three times with water and dried over Na_2SO_4. The solvent was removed under vacuum to afford a pink yellow liquid in quantitative yield. GC-MS(ESI): m/z (%) , 150 (0.4), 82 (31), 79 (29), 67 (100), 54 (96), 53 (4), 42 (4), 41 (38) . NMR comparable to those reported in literature.[8]



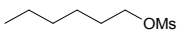
4-pentenyl mesylate (2c) , in accordance to general procedure, was prepared from 4-penten-1-ol (0.5g, 5.8 mmol) , triethylamine (1eq, 5.8mmol) and methane sulfonyl chloride (1 eq, 5.8 mmol), dissolved in 20 mL of CH_2Cl_2 , under stirring at -15 °C. After 1 hour and half the reaction mixture was quenched with 2ml of HCl 1N and the solution washed with $NaHCO_3$ saturated aqueous solution. The organic phase was washed three times with water and dried over Na_2SO_4. The solvent was removed under vacuum to afford a pink yellow liquid in quantitative yield. GC-MS(ESI): m/z (%) , 135 (0.4), 109 (0.8), 79 (31), 67 (100), 55 (13), 53 (19), 42 (8), 41 (23) . NMR comparable to those reported in literature.[9]



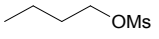
4-methanesulfonyloxy-1-butene (3c) , in accordance to general procedure, was prepared from homoalylic alcohol (0.5g, 5.8 mmol) , triethylamine (1eq, 5.8mmol) and methane sulfonyl chloride (1 eq, 5.8 mmol), dissolved in 20 mL of CH_2Cl_2 , under stirring at -15 °C. After 1 hour and half the reaction mixture was quenched with 2ml of HCl 1N and the solution washed with $NaHCO_3$ saturated aqueous solution. The organic phase was washed three times with water and dried over Na_2SO_4. The solvent was removed under vacuum to afford a pink yellow liquid in quantitative yield. GC-MS(ESI): m/z (%) , 149 (M-1, 0.4), 109 (52), 79 (95), 54 (100), 45 (9), 41 (52) . NMR comparable to those reported in literature.[10]



n-octyl mesylate (4c) , in accordance to general procedure, was prepared from n-octyl alcohol (0.5g, 5.8 mmol) , triethylamine (1eq, 5.8mmol) and methane sulfonyl chloride (1 eq, 5.8 mmol), dissolved in 20 mL of CH_2Cl_2 , under stirring at -15 °C. After 1 hour and half the reaction mixture was quenched with 2ml of HCl 1N and the solution washed with $NaHCO_3$ saturated aqueous solution. The organic phase was washed three times with water and dried over Na_2SO_4. The solvent was removed under vacuum to afford a pink yellow liquid in quantitative yield. GC-MS spectra comparable to those reported in literature.[102] NMR comparable to those reported in literature.[11]



n-hexyl mesylate (5c) , in accordance to general procedure, was prepared from n-hexyl alcohol (0.5g, 4.9 mmol) , triethylamine (1eq, 4.9mmol) and methane sulfonyl chloride (1 eq, 4.9 mmol), dissolved in 20 mL of CH_2Cl_2 , under stirring at -15 °C. After 1 hour and half the reaction mixture was quenched with 2ml of HCl 1N and the solution washed with $NaHCO_3$ saturated aqueous solution. The organic phase was washed three times with water and dried over Na_2SO_4. The solvent was removed under vacuum to afford a pink yellow liquid in quantitative yield. GC-MS spectra comparable to those reported in literature.[102]NMR comparable to those reported in literature.[12]



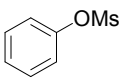
n-butyl mesylate (6c) , in accordance to general procedure, was prepared from n-butanol (0.5g, 6.7 mmol) , triethylamine (1eq, 6.7 mmol) and methane sulfonyl chloride (1 eq, 6.7 mmol), dissolved in 20 mL of CH_2Cl_2 , under stirring at -15 °C. After 1 hour and half the reaction mixture was quenched with 2ml of HCl 1N and the

solution washed with $NaHCO_3$ saturated aqueous solution. The organic phase was washed three times with water and dried over Na_2SO_4. The solvent was removed under vacuum to afford a pink yellow liquid in quantitative yield. GC-MS spectra comparable to those reported in literature.[6] NMR comparable to those reported in literature.[13]

Sec-butyl mesylate (1e) , in accordance to general procedure, was prepared from iso-butanol (0.5g, 3.7 mmol) , triethylamine (1eq, 3.7mmol) and methane sulfonyl chloride (1 eq, 3.7 mmol), dissolved in 20 mL of CH_2Cl_2 , under stirring at -15 °C. After 1 hour and half the reaction mixture was quenched with 2ml of HCl 1N and the solution washed with $NaHCO_3$ saturated aqueous solution. The organic phase was washed three times with water and dried over Na_2SO_4. The solvent was removed under vacuum to afford a pink yellow liquid in quantitative yield. GC-MS spectra comparable to those reported in literature.[6] NMR spectra comparable to those reported in literature.[14]

But-3-en-2-yl mesylate (2e) , in accordance to general procedure, was prepared from 2-hydroxy-3-butene (0.5g, 3.0 mmol) , triethylamine (1eq, 6.9mmol) and methane sulfonyl chloride (1 eq, 6.9 mmol), dissolved in 20 mL of CH_2Cl_2 , under stirring at -15 °C. After 1 hour and half the reaction mixture was quenched with 2ml of HCl 1N and the solution washed with $NaHCO_3$ saturated aqueous solution. The organic phase was washed three times with water and dried over Na_2SO_4. The solvent was removed under vacuum to afford a pink yellow liquid in quantitative yield. GC-MS(ESI): m/z (%) , 149 (M-1, 0.4), 135 (4), 79 (19), 72 (4), 71 (31), 57 (15), 55 (100), 51 (8), 43 (35). ^{1}H-NMR: (300 MHz, $CDCl_3$, 25°C): δ = 1.49 (3H, d, ^3J=6.4Hz, CH_3CH), 2.99 (3H, s, CH_3S), 5.14-5.23 (1H, m, $CHCH_3$), 5.14-5.43 (2H, m, CH_2=), 5.85-5.97(1H, m, CH=). ^{13}C-NMR : (75 MHz, $CDCl_3$, 25°C) δ = 21.35 (CH_3-CH), 38.99 (CH_3-S), 79.99 (CH-O), 118.35 (CH_2=), 136.34 (CH=).

***Tert*-butyl mesylate (3e) ,** in accordance to general procedure, was prepared from tert-butyl alcohol (0.5g, 6.7 mmol) , triethylamine (1eq, 6.7mmol) and methane sulfonyl chloride (1 eq, 6.97 mmol), dissolved in 20 mL of CH_2Cl_2 , under stirring at -15 °C. After 1 hour and half the reaction mixture was quenched with 2ml of HCl 1N and the solution washed with $NaHCO_3$ saturated aqueous solution. The organic phase was washed three times with water and dried over Na_2SO_4. The solvent was removed under vacuum to afford a pink yellow liquid in quantitative yield. NMR comparable to those reported in literature.[15]

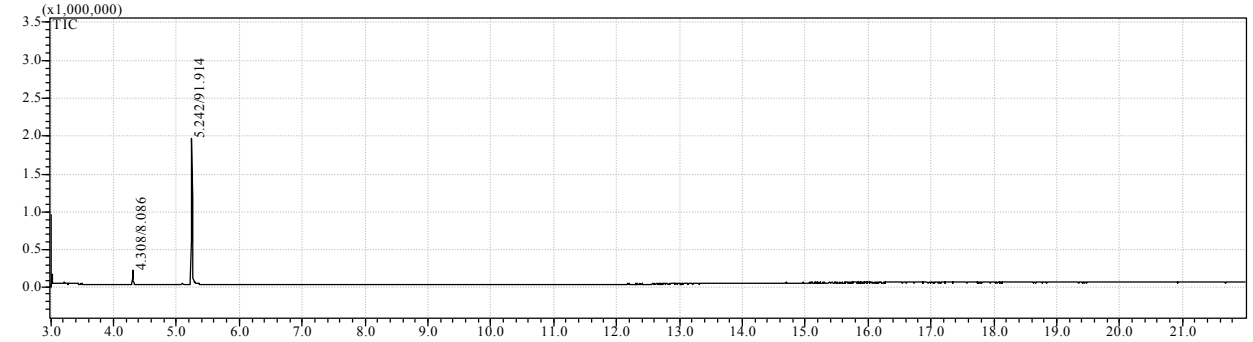


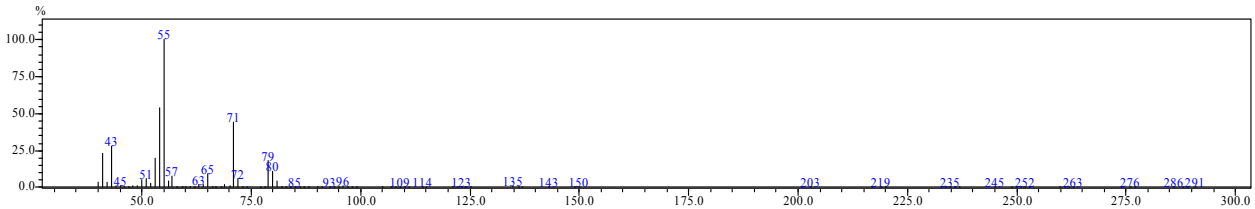
Phenyl mesylate (4e) , in accordance to general procedure, was prepared from phenol (0.5g, 5.3 mmol) , triethylamine (1eq, 5.3mmol) and methane sulfonyl chloride (1 eq, 5.3 mmol), dissolved in 20 mL of CH_2Cl_2 , under stirring at -15 °C. After 1 hour and half the reaction mixture was quenched with 2ml of HCl 1N and the

solution washed with $NaHCO_3$ saturated aqueous solution. The organic phase was washed three times with water and dried over Na_2SO_4. The solvent was removed under vacuum to afford a pink yellow liquid in quantitative yield. GC-MS spectra comparable to those reported in literature.[16] NMR comparable to those reported in literature.[17]

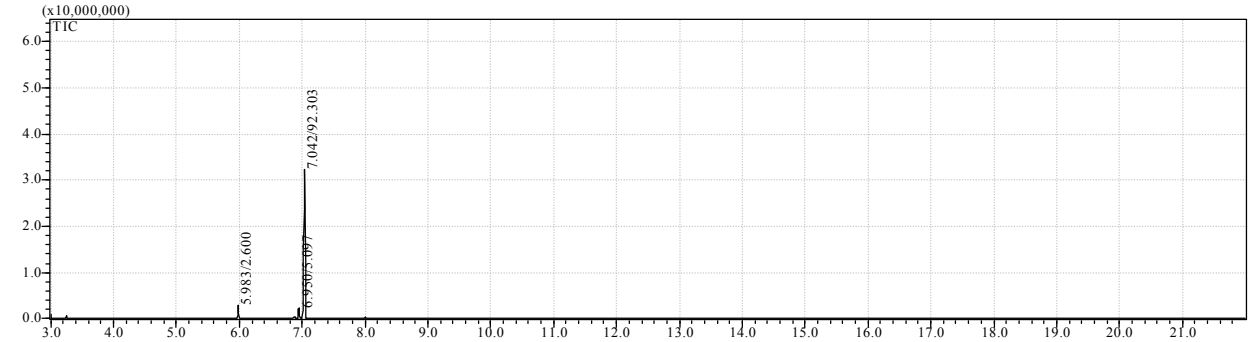
a. GC-MS SPECTRA

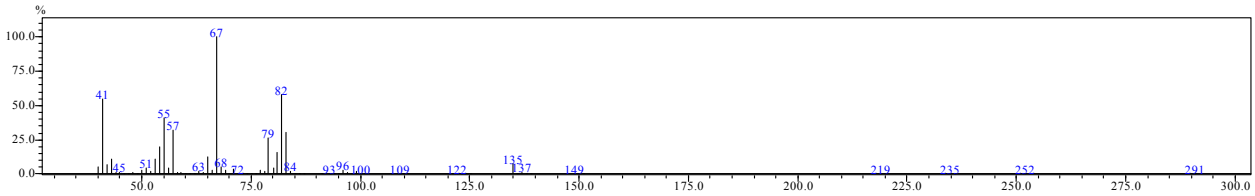
Trans-2-butenyl mesylate (3a)

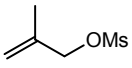


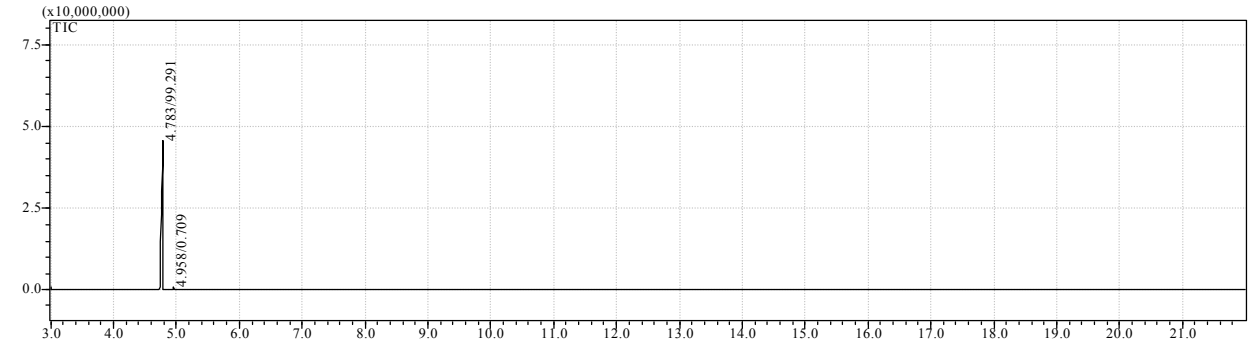


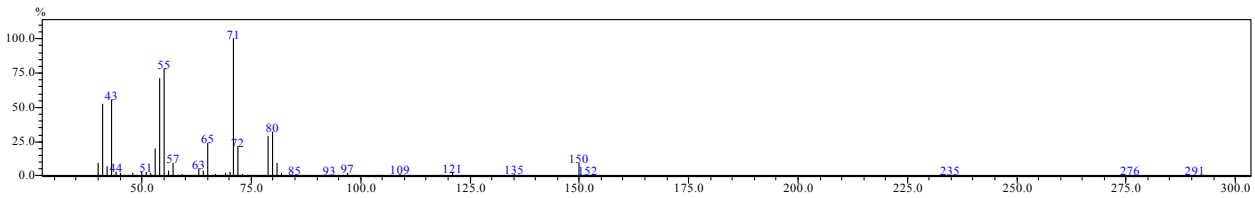
Trans-2-hexenyl mesylate (4a)

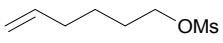


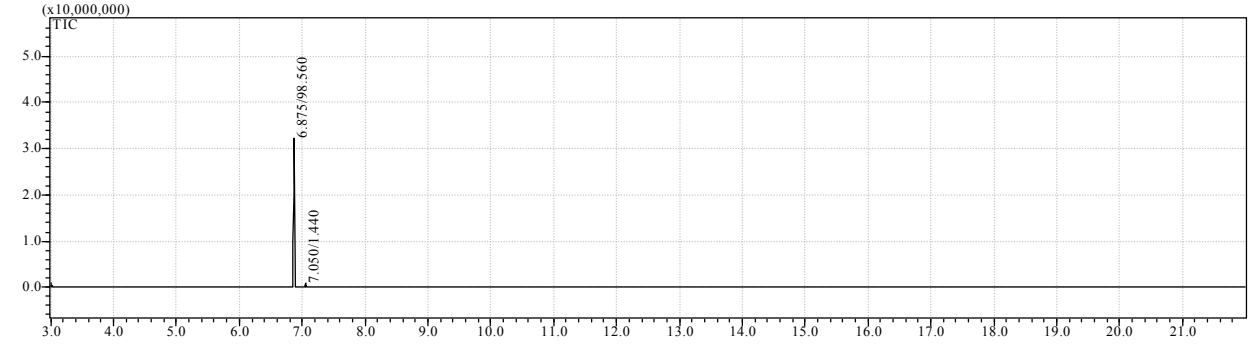


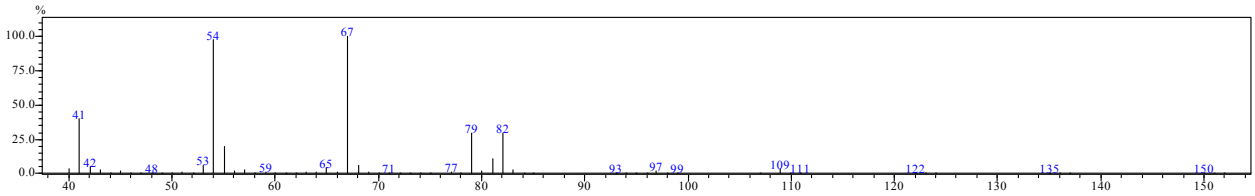
2-methylallyl mesylate (5a) 



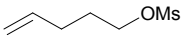


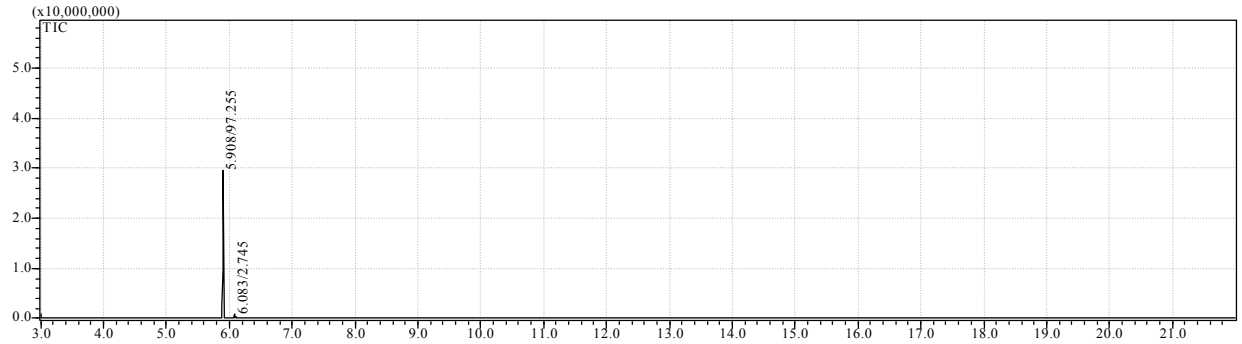
5-hexenyl mesylate (1c) 

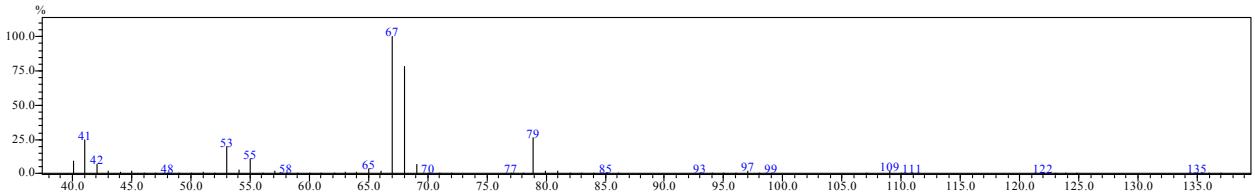




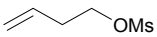
4-pentenyl mesylate (2c)

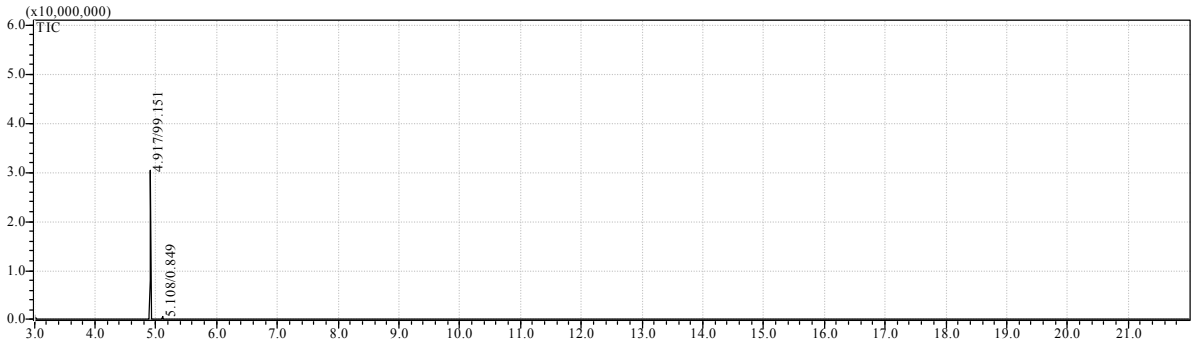


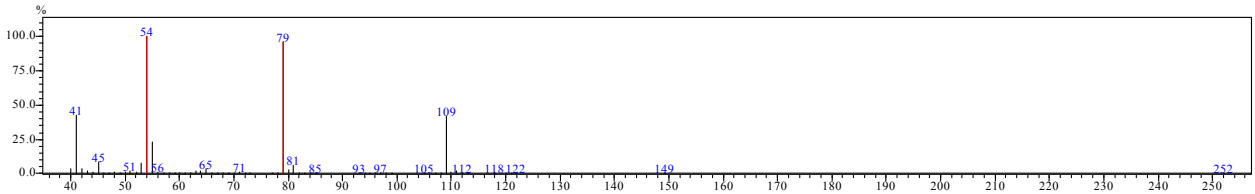


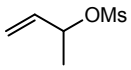


4-methanesulfonyloxy-1-butene (3c)

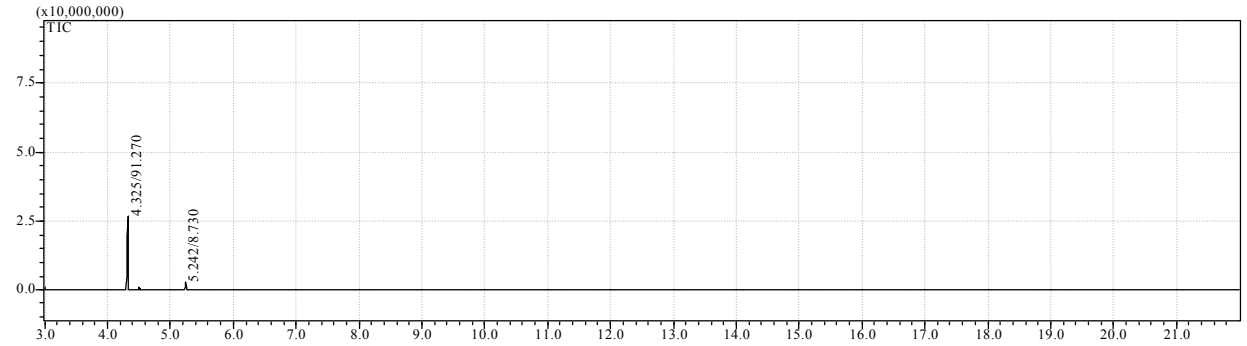


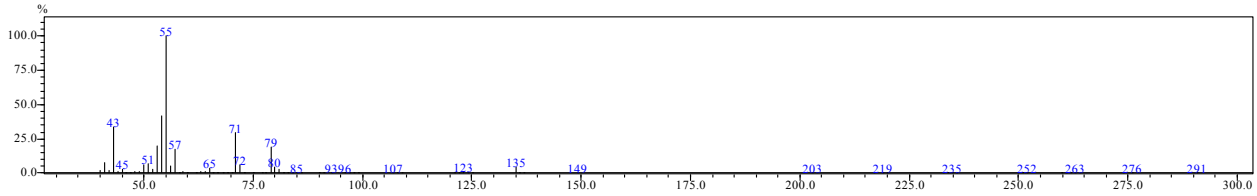




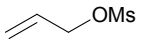


But-3-en-2-yl mesylate (2e)

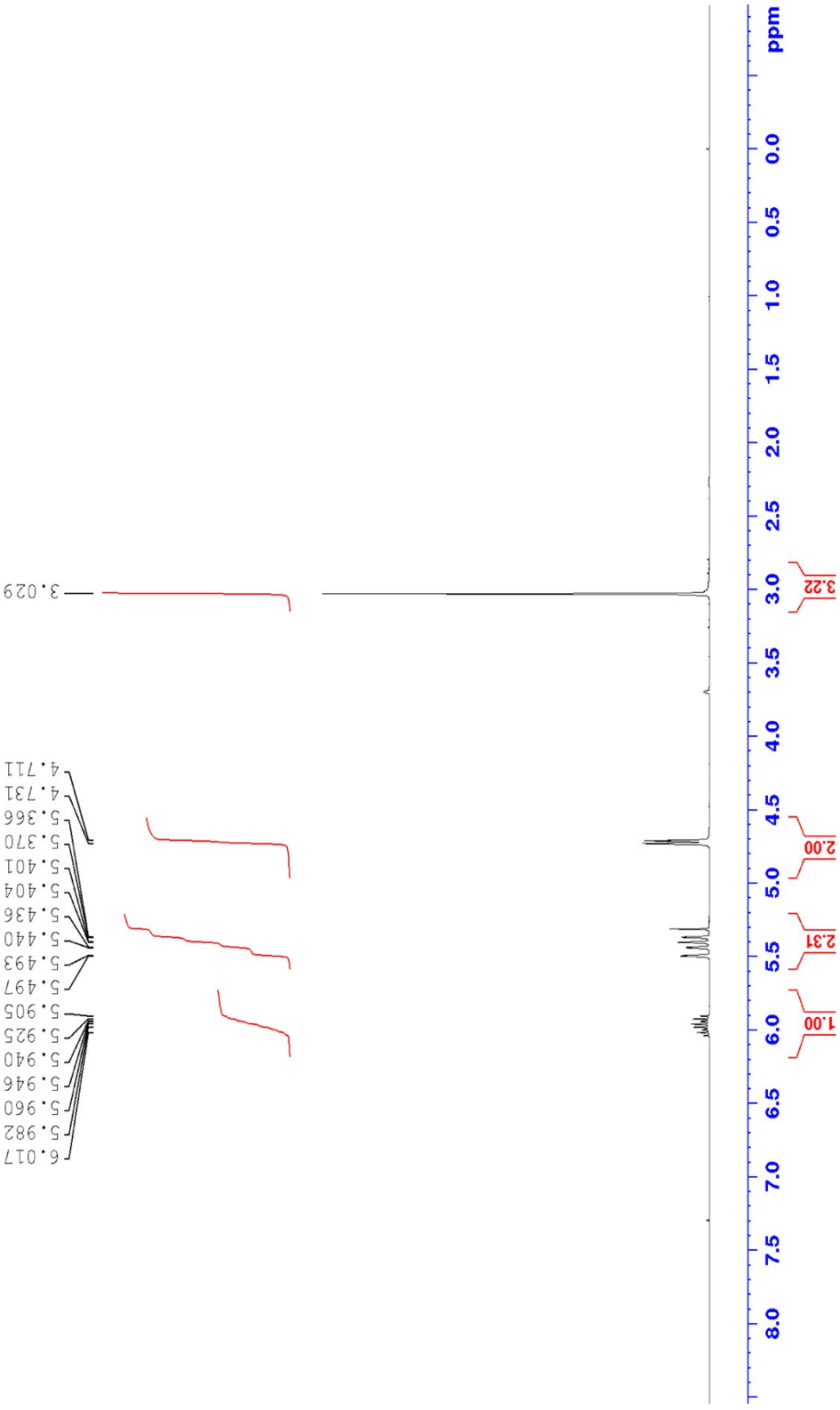




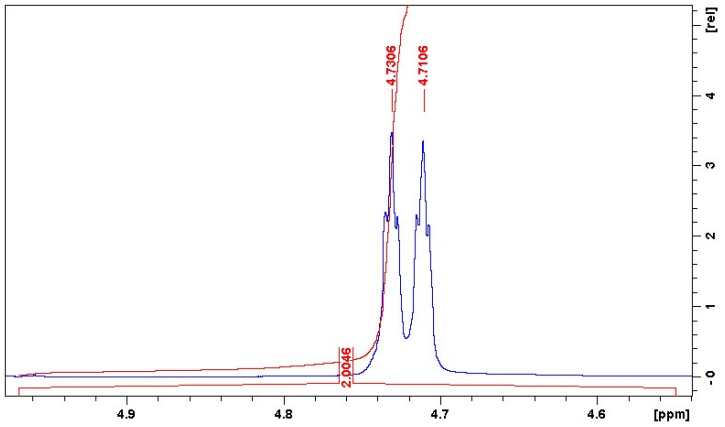
b. NMR SPECTRA

Allyl mesylate (2a) 

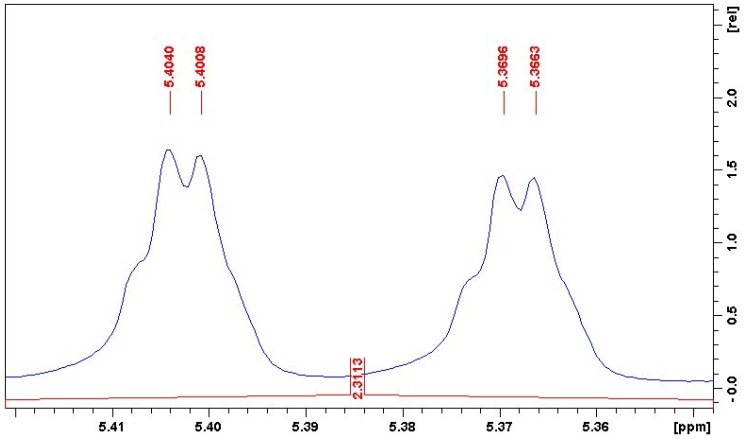
^{1}H-NMR


3.029
4.711
4.731
5.366
5.370
5.401
5.404
5.436
5.440
5.493
5.497
5.905
5.925
5.940
5.946
5.960
5.982
6.017
3.22
2.00
2.31
1.00
ppm
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0

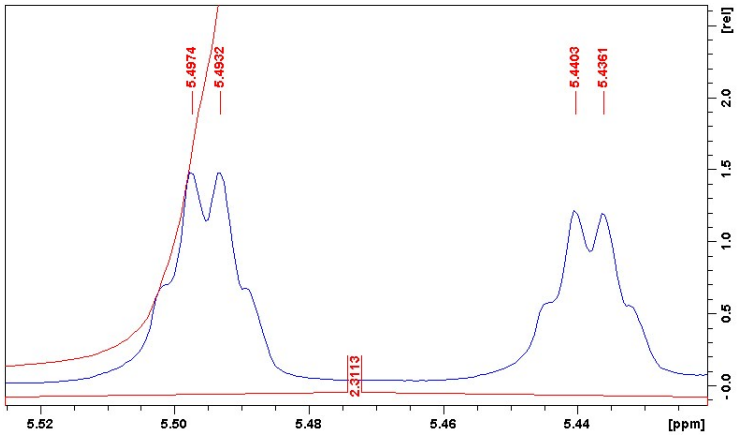
Doublet



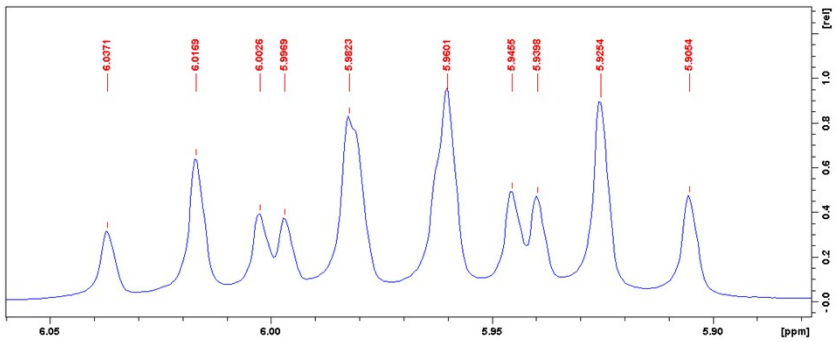
Doublet of doublets



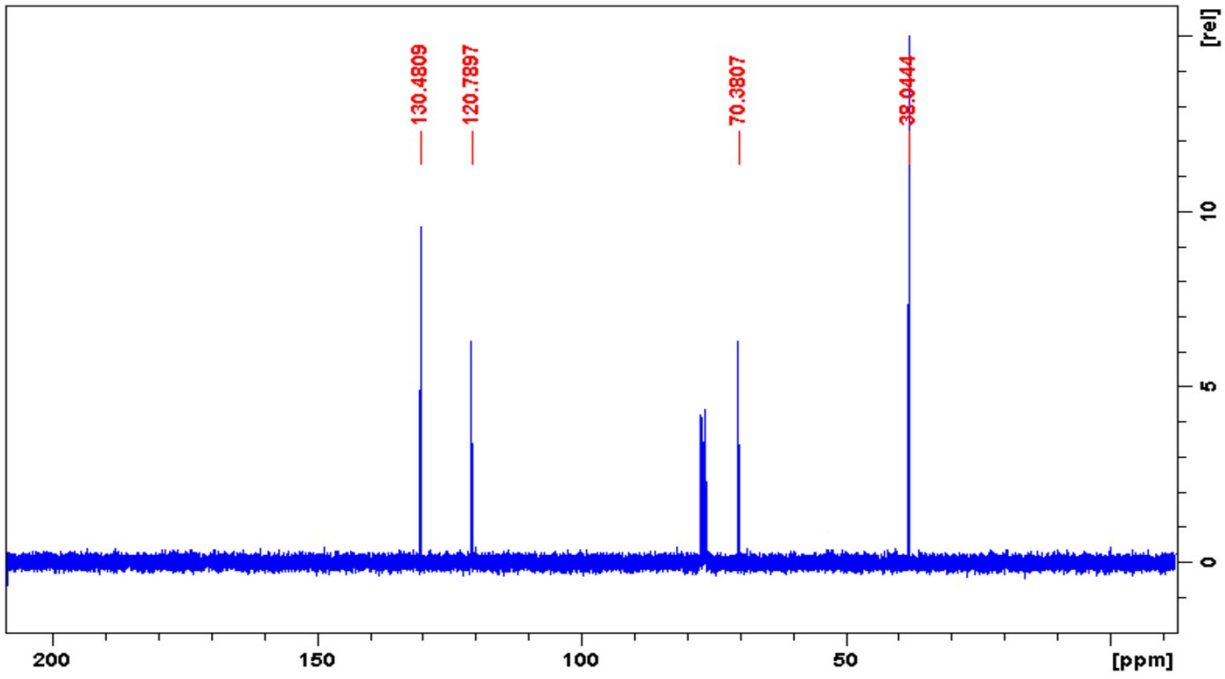
Doubet of doublets

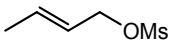


Doublet doublet of triplets

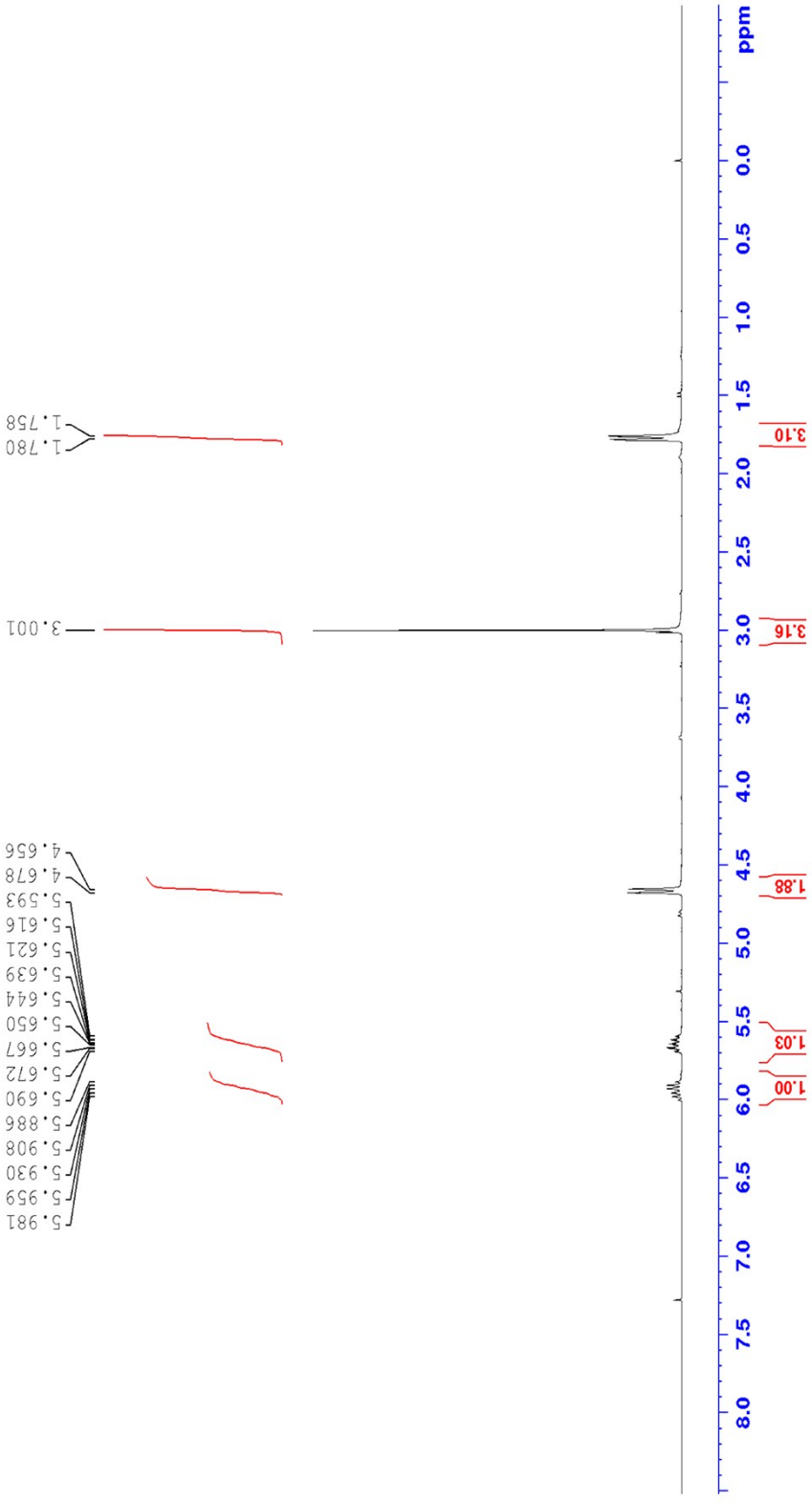

6.0371
6.0169
6.0026
5.9969
5.9823
5.9601
5.9455
5.9398
5.9254
5.9054
[rel]
1.0
0.8
0.6
0.4
0.2
-0.0
6.05
6.00
5.95
5.90
[ppm]

^{13}C-NMR

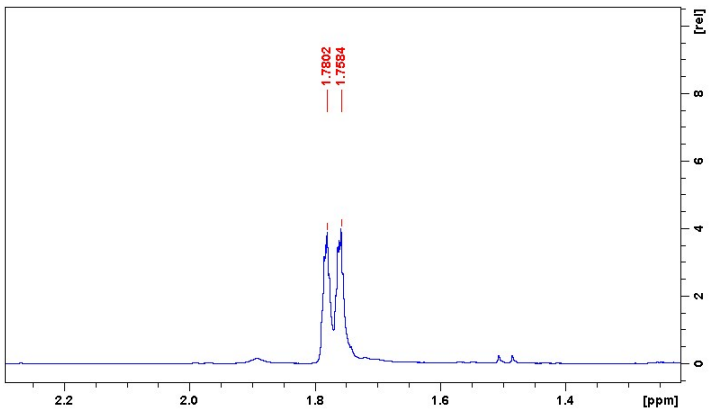

130.4809
120.7897
70.3807
38.0444
[rel]
10
5
0
200
150
100
50
[ppm]

Trans-2-butenyl mesylate (3a) 

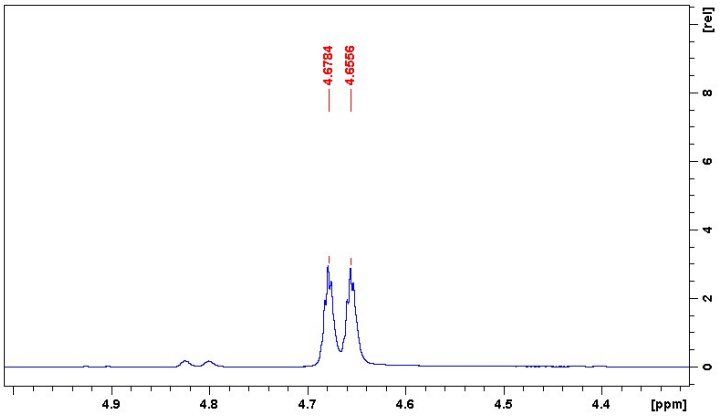
^{1}H-NMR



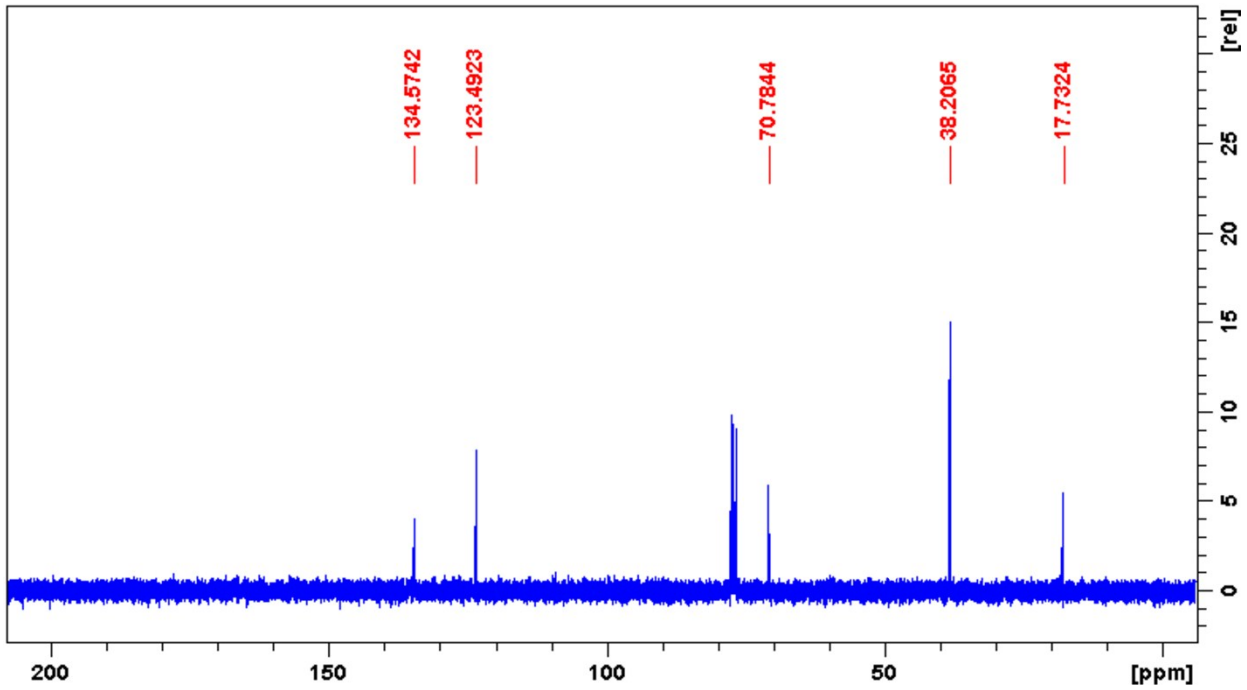
Doublet



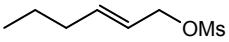
Doublet



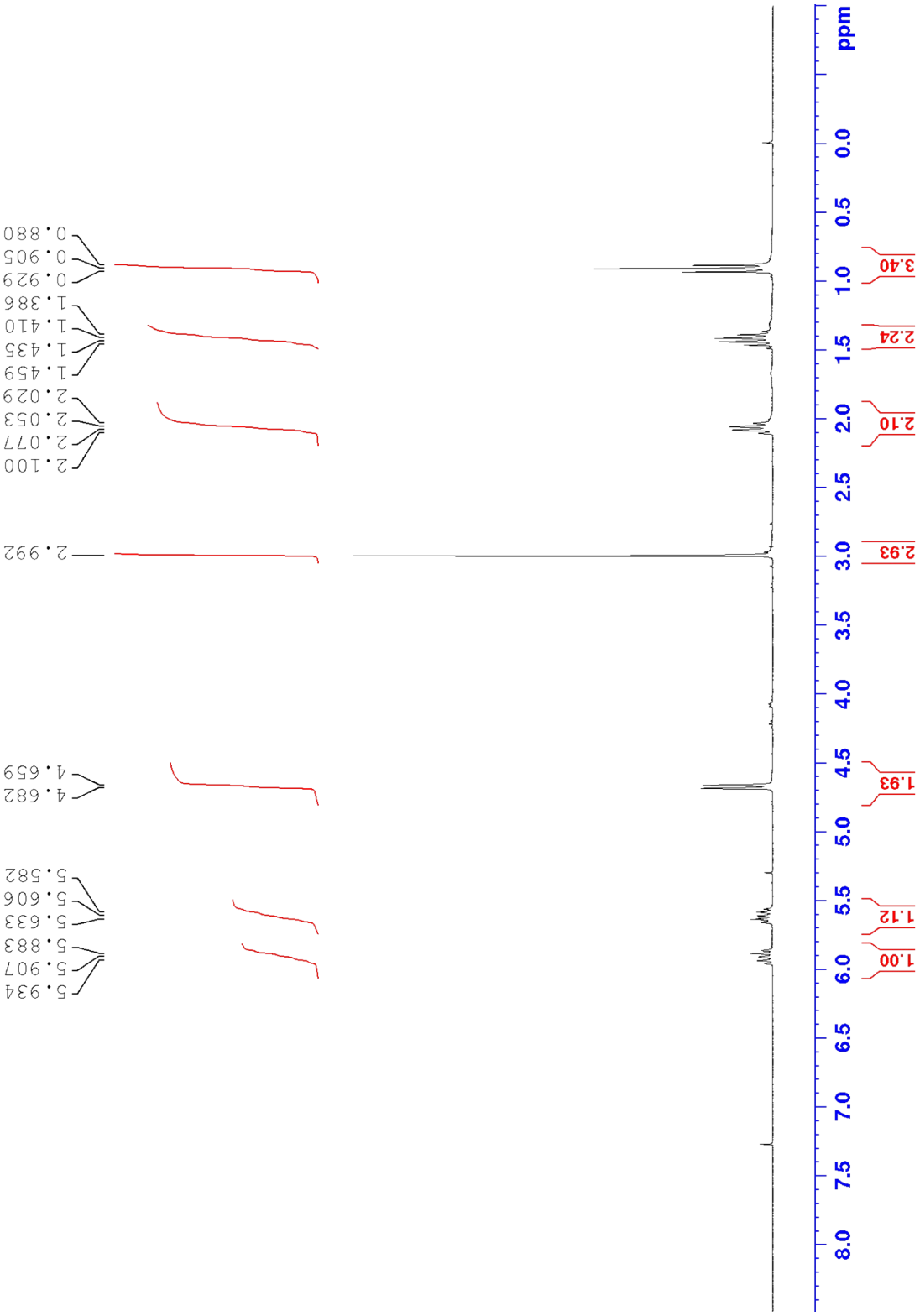
^{13}C-NMR


134.5742
123.4923
70.7844
38.2065
17.7324
200
150
100
50
[ppm]
0
5
10
15
20
25
[rel]

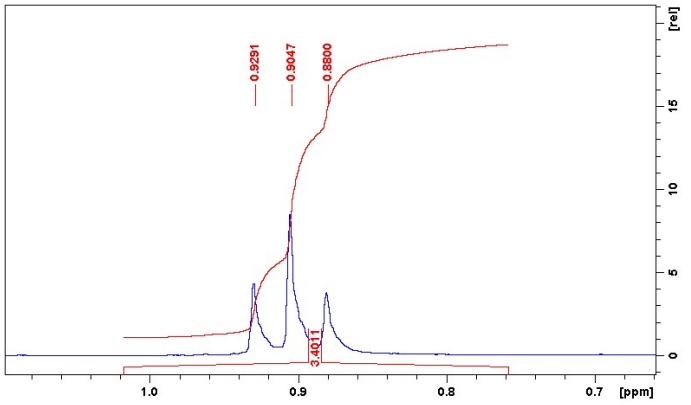
Trans-2-hexenyl mesylate (4a)



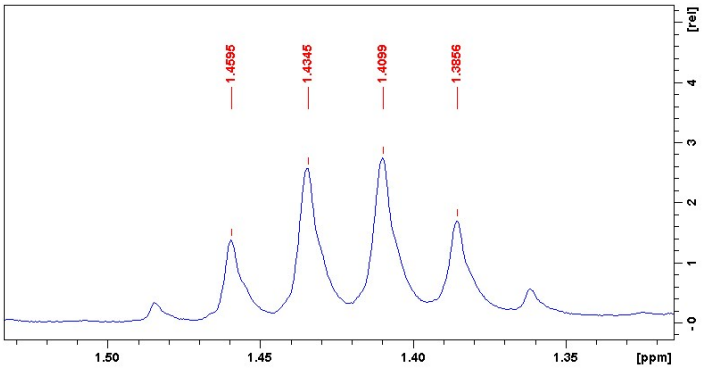
^{1}H-NMR


ppm
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
3.40
2.24
2.10
2.93
1.93
1.12
1.00
0.880
0.905
0.929
1.386
1.410
1.435
1.459
2.029
2.053
2.077
2.100
2.992
4.659
4.682
5.582
5.606
5.633
5.883
5.907
5.934

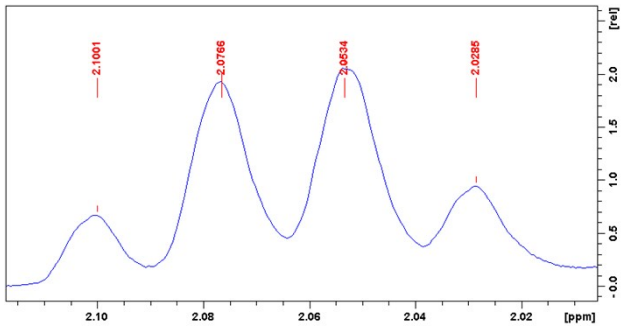
Triplet



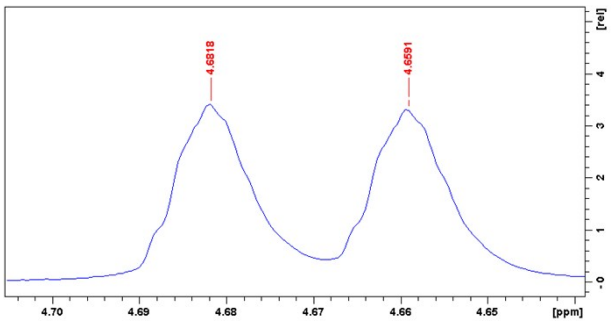
Sextet



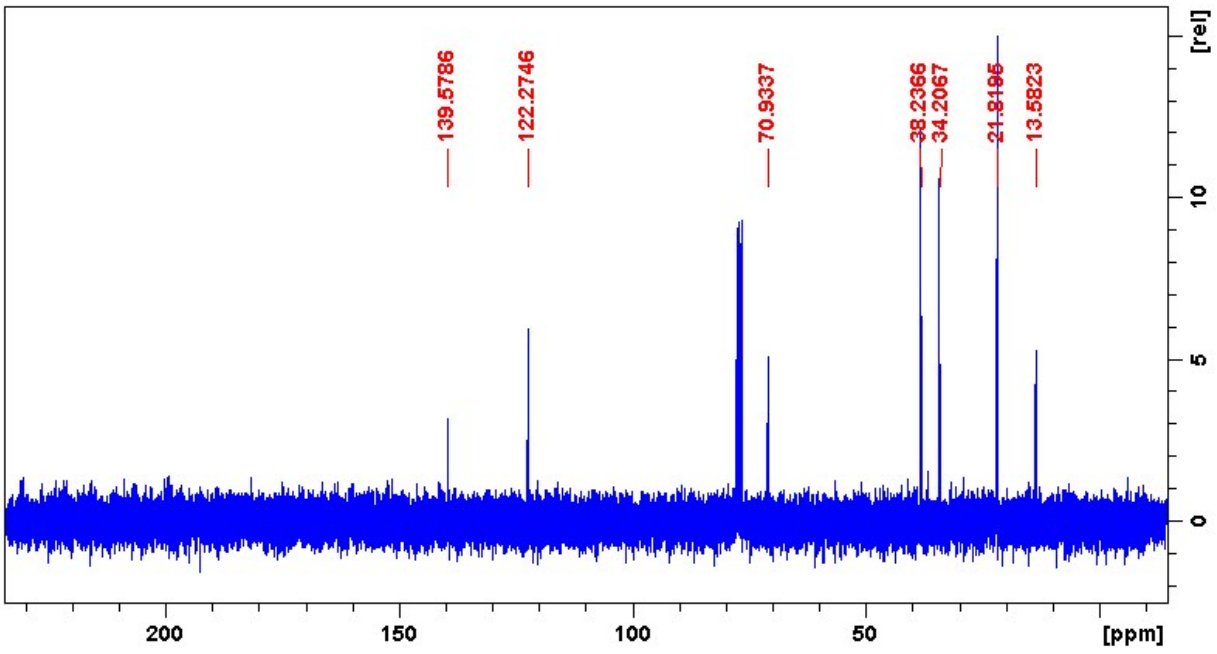
Doublet of triplets

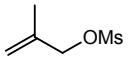


Doublet

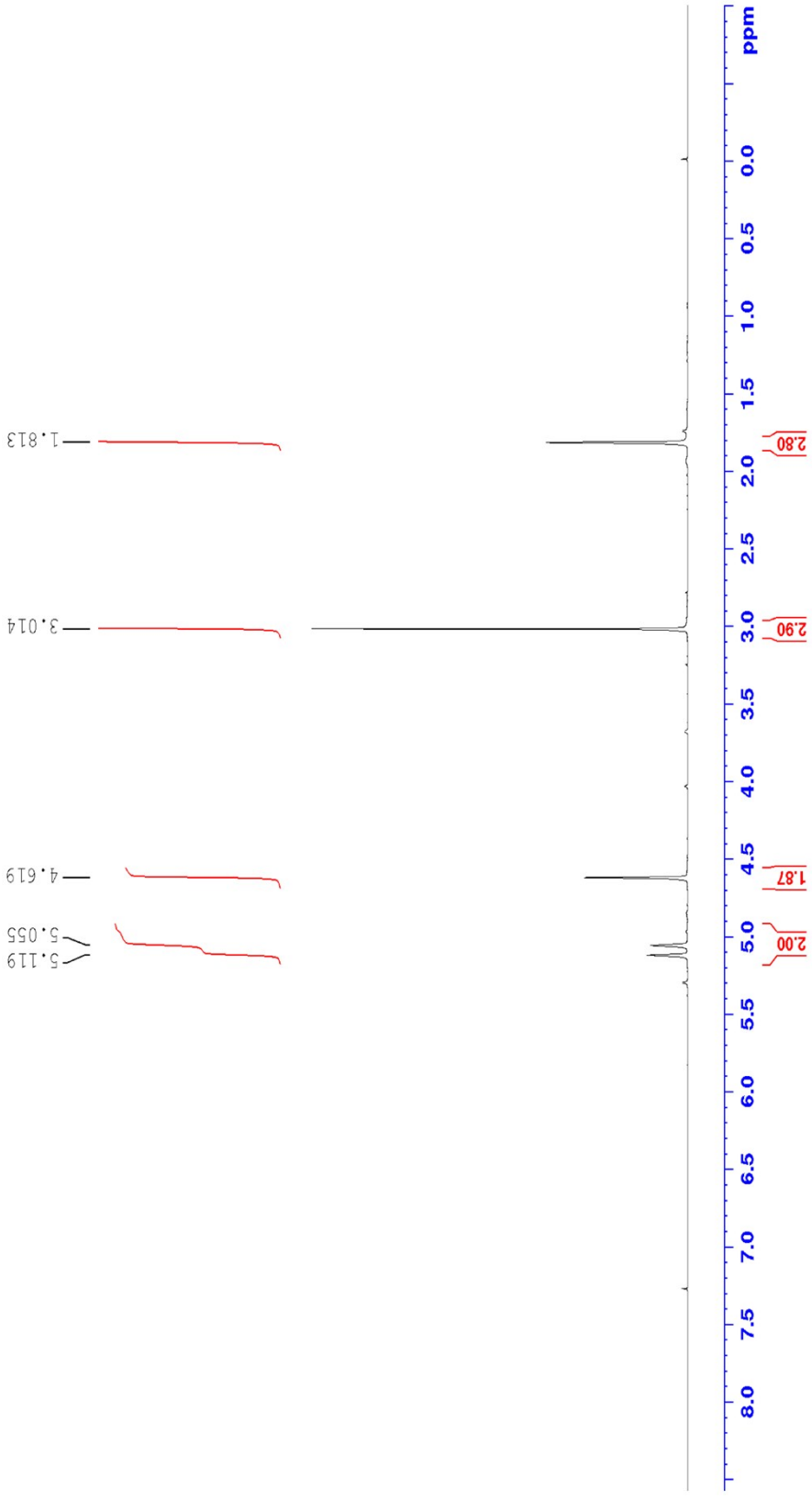

4.6818
4.6591
[rel]
4.70
4.69
4.68
4.67
4.66
4.65
[ppm]

^{13}C-NMR

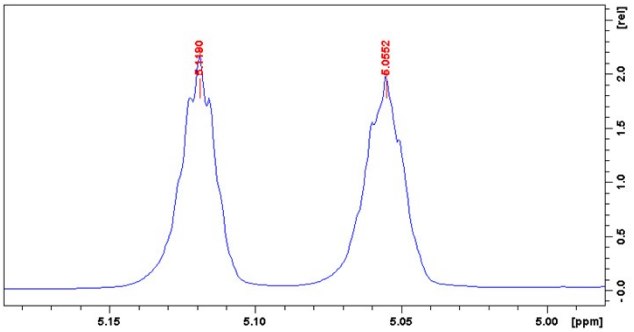

139.5786
122.2746
70.9337
38.2366
34.2067
21.8195
13.5823
[rel]
200
150
100
50
[ppm]

2-methylallyl mesylate (5a) 

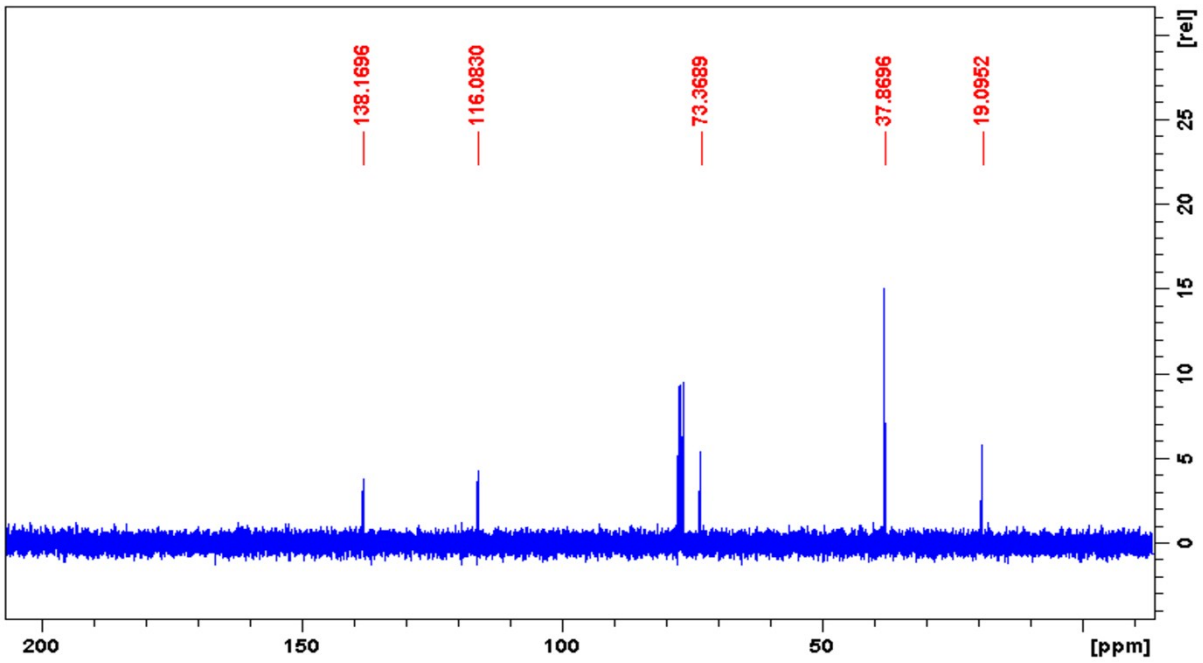
^{1}H-NMR

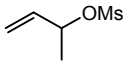


Doublet

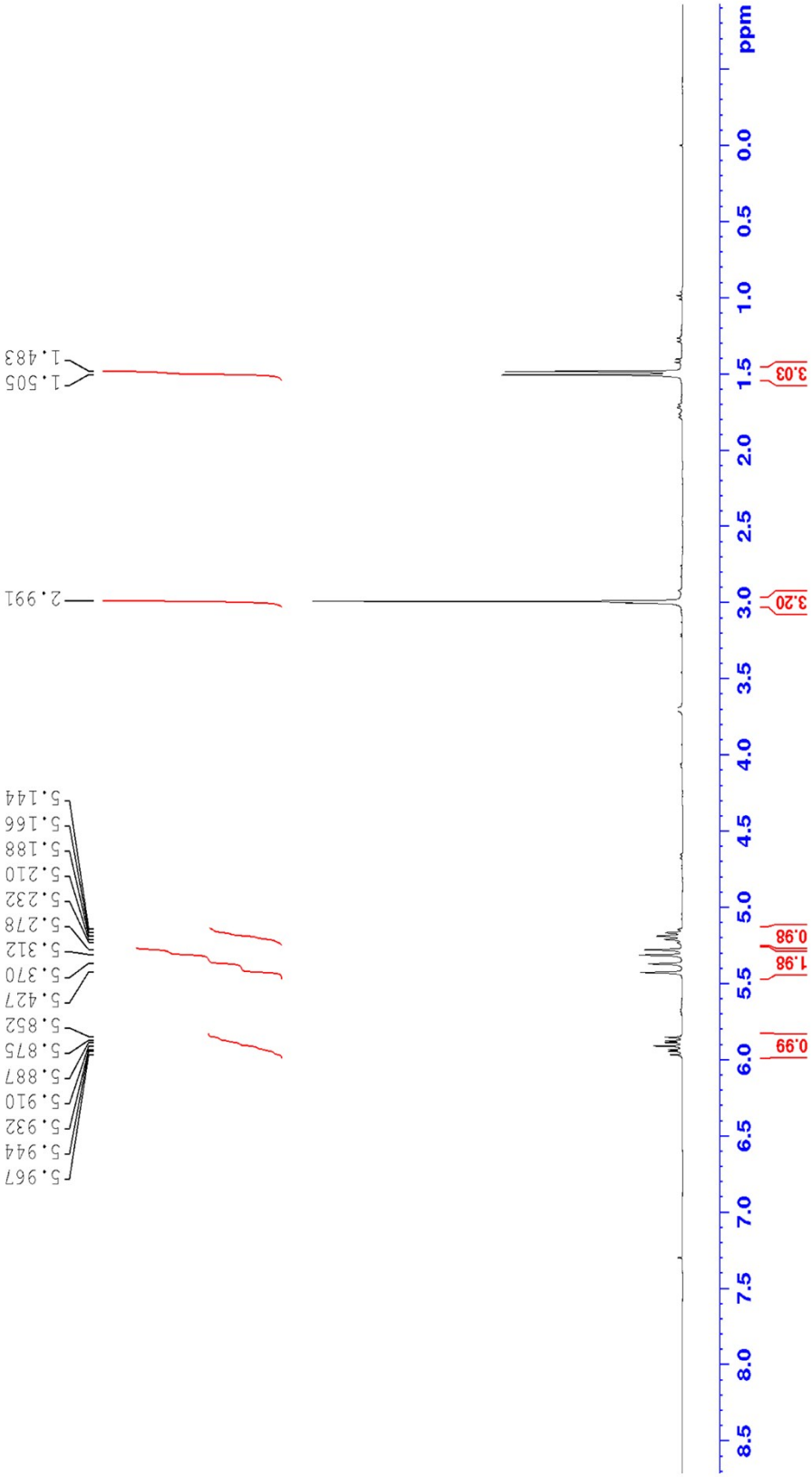


^{13}C-NMR

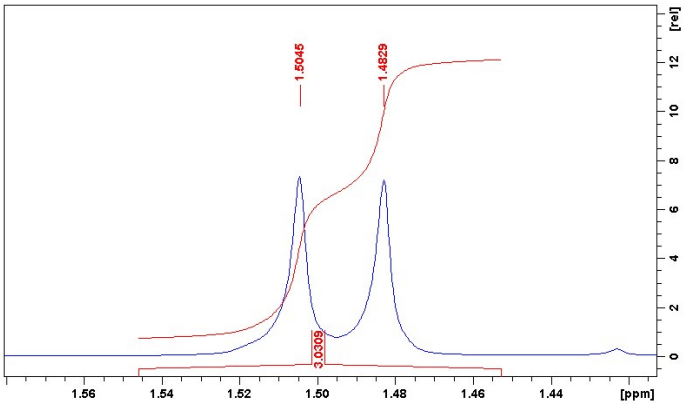


But-3-en-2-yl mesylate (2e) 

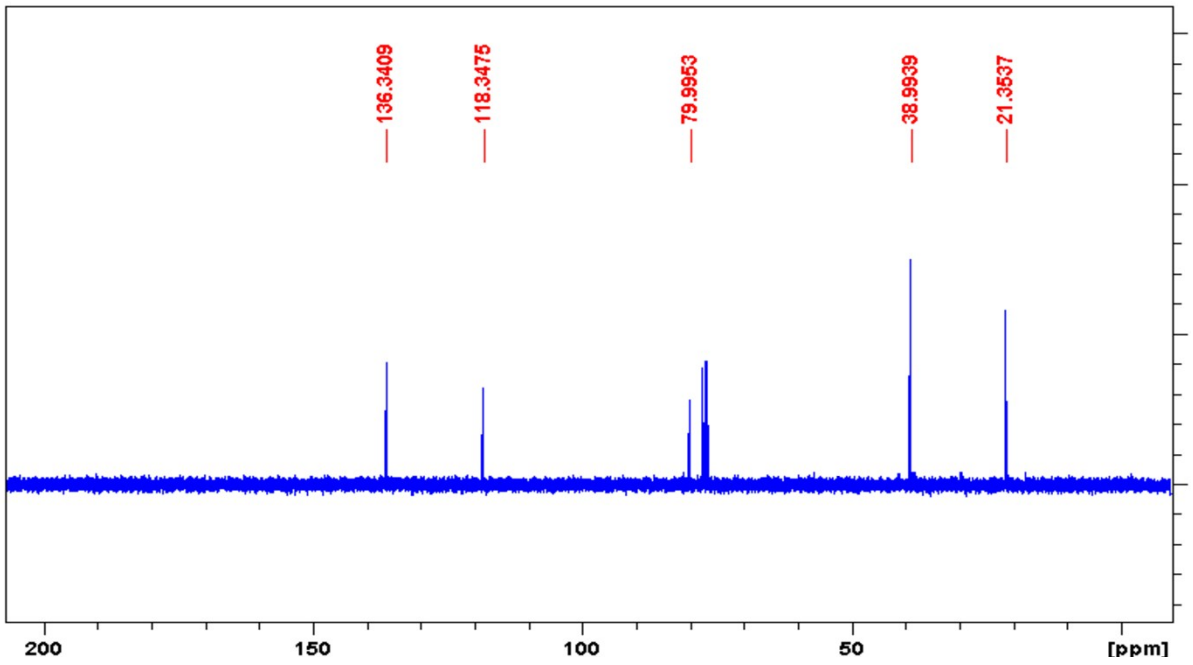
^{1}H-NMR


ppm
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
3.03
3.20
0.98
1.98
0.99
1.483
1.505
2.991
5.144
5.166
5.188
5.210
5.232
5.278
5.312
5.370
5.427
5.852
5.875
5.887
5.910
5.932
5.944
5.967

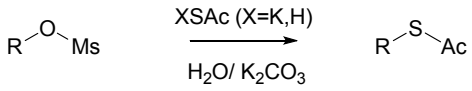
Doublet



^{13}C-NMR



4. General reaction scheme for the synthesis of organic thioacetates



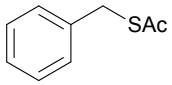
5. General procedure for the synthesis of benzyl thioacetate (1b), allylic thioacetates (2b-5b) and propargyl thioacetate (6b)

2.7 mmol of freshly prepared mesylate were put in a one neck round bottom flask. In the same flask 20 ml of 0.4 M aqueous potassium carbonate solution were added. 1.5 eq of potassium thioacetate or acetic acid were added dropwise to the solution under stirring. The mixture was stirred at 40 °C with a reflux condenser for almost 2 hours. The organic phase was extracted with a small portion of diethyl ether and washed three times with water. The organic phase was dried over Na_2SO_4 and the solvent removed under reduced pressure using a rotary evaporator. The product was recovered as a yellow liquid (GC-MS yield was determined). A high vacuum pump was used to remove any residual solvent and minor impurities, after that the isolated yield was calculated in the usual way as percentage yield from the ratio between the actual yield and the theoretical yield. The theoretical yield was obtained considering the organic mesylate as the limiting reactant. The actual yield was obtained weighing the product after the mentioned work up.

6. General procedure for the synthesis of primary thioacetates *(1d-6d)*

2.8 mmol of freshly prepared mesylate were put in a one neck round bottom flask. In the same flask 20 ml of 0.4 M aqueous potassium carbonate solution were added. 1.5 eq of potassium thioacetate or acetic acid were added to the solution under stirring. The mixture was stirred at 80 °C under reflux for almost 2 hours. The organic phase was extracted with a small portion of diethyl ether and washed three times with water. The organic phase was dried over Na_2SO_4 and the solvent removed under reduced pressure using a rotary evaporator. The product was recovered as a yellow liquid (GC-MS yield was determined). A high vacuum pump was used to remove any residual solvent and minor impurities, after that the isolated yield was calculated in the usual way as percentage yield from the ratio between the actual yield and the theoretical yield. The theoretical yield was obtained considering the organic mesylate as the limiting reactant. The actual yield was obtained weighing the product after the mentioned work up.

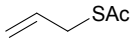
7. General, Experimental and Analytical Information



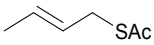
Benzyl thioacetate (1b) was prepared in accordance to the general procedure.

Benzyl mesylate (1a) (0.5g, 2.7 mmol) was put in a one neck round bottom flask. In the same flask 20 ml of 0.4 M aqueous potassium carbonate solution were added. 1.5 eq of potassium thioacetate or acetic acid were added dropwise to the solution under stirring. The mixture was stirred at 40 °C with a reflux condenser for almost 2 hours. The organic phase was extracted with a small portion of diethyl ether and washed three times with water. The organic phase was dried over Na_2SO_4 and the solvent removed under vacuum. The product was recovered as a yellow liquid in 94 % yield (GC-MS). Isolated yield after work up: 89%. GC-MS spectra comparable to those reported in literature.[18] ^{1}H-NMR: (300 MHz, $CDCl_3$, 25°C) δ = 2.33 (s, CH_3), 4.11

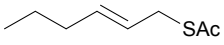
(s, CH_2), 7.20-7.36 (m, 5H). ^{13}C-NMR: (75 MHz, $CDCl_3$, 25°C) δ = 30.29(CH_3), 33.48(CH_2S), 126.99(CH), 127.28(CH), 128.56(CH), 128.64(CH), 128.82(CH), 137.61(CH), 195.10(C=O).



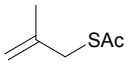
Allyl thioacetate (2b) was prepared in accordance to the general procedure.
Allyl mesylate (2a) (0.37g, 2.7 mmol) was put in a one neck round bottom flask. In the same flask 20 ml of 0.4 M aqueous potassium carbonate solution were added. 1.5 eq of potassium thioacetate or acetic acid were added dropwise to the solution under stirring. The mixture was stirred at 40 °C with a reflux condenser for almost 2 hours. After the usual work up the product was recovered as a yellow liquid obtained in 96 % yield (GC-MS) . Isolated yield after work up: 92%. ^{1}H-NMR: (300 MHz, $CDCl_3$, 25°C) δ = 2.33 (3H, s, CH_3), 3.53 (2H, d, ^3J = 7Hz, CH_2S), 5.08-5.26 (2H, m, CH=CH_2), 5.73-5.93 (1H, m, CH=CH_2). ^{13}C-NMR : (75 MHz, $CDCl_3$, 25°C) δ = 30.48 (CH_3), 32.01 (CH_2S), 117.88 (CH=CH_2), 133.07 (CH=CH_2), 195.12 (C=O).



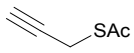
***trans*-2-butenyl thioacetate (3b)** was prepared in accordance to the general procedure.
trans-2-butenyl mesylate (3a) (0.40g, 2.7 mmol) was put in a one neck round bottom flask. In the same flask 20 ml of 0.4 M aqueous potassium carbonate solution were added. 1.5 eq of potassium thioacetate or acetic acid were added dropwise to the solution under stirring. The mixture was stirred at 40 °C with a reflux condenser for almost 2 hours. After the usual work up the product was recovered as a yellow liquid obtained in 97% yield (GC-MS) . Isolated yield after work up: 91%. GC-MS(ESI): m/z (%) , 149 (M^+, 31), 88 (35), 55 (73), 53 (15), 43 (100). ^{1}H-NMR: (300 MHz, $CDCl_3$, 25°C) δ = 1.65-1.68 (3H, m, CH_3), 2.32 (3H, s, CH_3), 3.48 (2H, bd, ^3J =7Hz, CH_2), 5.37-5.48 (1H, m, CH=CH), 5.57-5.72 (1H, m, CH=CH) . ^{13}C-NMR : (75 MHz, $CDCl_3$, 25°C) δ = 17.53 (CH_3-CH), 30.29(CH_3-C=0), 31.25 (CH_2-S), 125.63 (CH=CH), 129.03 (CH=CH), 195.04 (C=0).



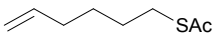
***trans*-2-hexenyl thioacetate (4b)** was prepared in accordance to the general procedure.
t*rans*-2-hexenyl mesylate (4a) (0.48g, 2.7 mmol) was put in a one neck round bottom flask. In the same flask 20 ml of 0.4 M aqueous potassium carbonate solution were added. 1.5 eq of potassium thioacetate or acetic acid were added dropwise to the solution under stirring. The mixture was stirred at 40 °C with a reflux condenser for almost 2 hours. After the usual work up the product was recovered as a yellow liquid obtained in 93% yield (GC-MS) . Isolated yield after work up: 89%. GC-MS(ESI): m/z (%) , 158 (M^+, 0.4), 116 (19), 82 (81), 73 (9), 67 (30), 55 (67), 43 (100), 41 (38) . ^{1}H-NMR: (300 MHz, $CDCl_3$, 25°C) δ = 0.87 (3H, t, ^3J=7.2Hz, CH_3), 1.37 (2H,sextuplet, ^3J=7.3Hz, CH_2-CH_3), 1.98 (2H,dt , ^3J =7Hz, ^{3}J=7Hz, CH_2-CH=), 2.32 (3H, s, CH3), 3.49 (2H, bd, ^3J=7Hz, CH_2-S), 5.36-5.46 (1H, m, CH=CH), 5.60-5.69 (1H, m, CH=CH). ^{13}C-NMR : (75 MHz, $CDCl_3$, 25°C) δ = 13.55 (CH_3-CH_2), 22.21(CH2-CH_3), 30.45, 31.48, 34.28, 124.55(CH=CH), 134.54(CH=CH), 195.41(C=0).



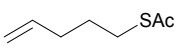
2-methylallyl thioacetate (5b) was prepared in accordance to the general procedure.
2-methylallyl mesylate (5a) (0.40g, 2.7 mmol) was put in a one neck round bottom flask. In the same flask 20 ml of 0.4 M aqueous potassium carbonate solution were added. 1.5 eq of potassium thioacetate or acetic acid were added dropwise to the solution under stirring. The mixture was stirred at 40 °C with a reflux condenser for almost 2 hours. After the usual work up the product was recovered as a yellow liquid obtained in 97% yield (GC-MS) . Isolated yield after work up: 93%. GC-MS(ESI): m/z (%) , 130 (M^+, 0.4), 88 (90), 55 (25), 45 (11), 43 (100) . ^{1}H-NMR: (300 MHz, $CDCl_3$, 25°C) δ = 1.75-1.76 (3H, m, CH_3-C), 2.35 (3H,s, CH_3-C=O), 3.54 (2H,s , CH_2-S), 4.89 (2H, dt, ^2J=34.1Hz, ^4J=0.9Hz, CH_2=C). ^{13}C-NMR : (75 MHz, $CDCl_3$, 25°C) δ = 21.15(CH_3-C), 30.40 (CH_3-C=O), 35.98(CH_2-S), 113.97(CH_2=C), 140.72 (C=CH_2), 195.14(C=O).



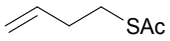
S-Prop-2-ynyl-thioacetate (6b) was prepared in accordance to the general procedure.
S-prop-2-ynyl mesylate (6a) (0.36g, 2.7 mmol) was put in a one neck round bottom flask. In the same flask 20 ml of 0.4 M aqueous potassium carbonate solution were added. 1.5 eq of potassium thioacetate or acetic acid were added dropwise to the solution under stirring. The mixture was stirred at 40 °C with a reflux condenser for almost 2 hours. After the usual work up the product was recovered as a yellow liquid obtained in 94% yield (GC-MS). Isolated yield after work up: 90%. GC-MS comparable to those reported in literature.[19]
^{1}H-NMR: (300 MHz, $CDCl_3$, 25°C): δ = 2.15 (1H, t, ^4J=2.7Hz, CH≡C), 2.36 (3H,s, CH_3-C=O), 3.64 (2H, d, ^4J=2.7Hz, CH_2).
^{13}C-NMR : δ = 17.45(CH_2), 30.11(CH_3-C=O), 70.86 (C≡C), 78.75 (C≡C), 193.70 (C=O).



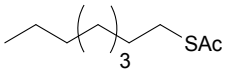
5-Acetylsulfanyl-hex-1-ene (1d) was prepared in accordance to the general procedure.
5-hexenyl mesylate (1c) (0.50g, 2.8 mmol) was put in a one neck round bottom flask. In the same flask 20 ml of 0.4 M aqueous potassium carbonate solution were added. 1.5 eq of potassium thioacetate or acetic acid were added dropwise to the solution under stirring. The mixture was stirred at 80 °C under reflux for almost 2 hours. After the usual work up the product was recovered as a yellow liquid obtained in 96% yield (GC-MS) . Isolated yield after work up: 92%. GC-MS(ESI): m/z (%) , 158 (M^+, 0.4), 115 (69), 100 (11), 87 (6), 81 (11), 67 (8), 54 (4), 43 (100), 41 (11) . ^{1}H-NMR: (300 MHz, $CDCl_3$, 25°C): δ = 1.43-1.50 (2H, m, CH_2), 1.54-1.61 (2H,m, CH_2), 2.06 (2H, m, CH_2-CH_2-CH_2), 2.33 (3H, s, CH_3), 2.87 (2H,t, ^3J=7Hz,CH_2S), 4.94-5.04 (2H, m, CH_2=C), 5.74-5.83 (1H, m, CH=C). ^{13}C-NMR : δ = 27.99, 28.9, 28.9 30.60, 33.18, 114.75 (CH_2=), 138.33(CH=), 195.88 (C=O).



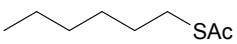
S-Pent-4-en-1-yl ethanethioate (2d) was prepared in accordance to the general procedure.
4-pentenyl mesylate (2c) (0.45g, 2.8 mmol) was put in a one neck round bottom flask. In the same flask 20 ml of 0.4 M aqueous potassium carbonate solution were added. 1.5 eq of potassium thioacetate or acetic acid were added dropwise to the solution under stirring. The mixture was stirred at 80 °C under reflux for almost 2 hours. After the usual work up the product was recovered as a yellow liquid obtained in 97% yield (GC-MS) . Isolated yield after work up: 93%. GC-MS: m/z (%) , 144 (M^+, 0.4), 101 (96), 86 (8), 67 (29), 59 (9), 55 (6), 47 (4), 43 (100) . ^{1}H-NMR: (300 MHz, $CDCl_3$, 25°C): δ = 1.66 (2H, quintet, ^{3}J=7.3Hz, CH_2-CH_2-CH_2), 2.08-2.13 (2H,m, CH_2), 2.31 (3H, s, CH_3-C=O), 2.87 (2H, t, ^{3}J=7.3Hz, CH_2S), 4.96-5.05 (2H, m, CH_2=CH), 5.70-5.81 (1H, m, CH=CH_2). ^{13}C-NMR : (75 MHz, $CDCl_3$, 25°C) δ = 28.49, 30.59, 32.73, 115.39(CH_2=CH), 137.39 (CH=CH_2), 208.89(C=O). NMR spectra comparable to those reported in literature.[20,21]



Thioacetic acid S-but-3-enyl ester (3d) was prepared in accordance to the general procedure.
4-butenyl mesylate (3c) (0.42g, 2.8 mmol) was put in a one neck round bottom flask. In the same flask 20 ml of 0.4 M aqueous potassium carbonate solution were added. 1.5 eq of potassium thioacetate or acetic acid were added dropwise to the solution under stirring. The mixture was stirred at 80 °C under reflux for almost 2 hours. After the usual work up the product was recovered as a yellow liquid obtained in 96% yield (GC-MS) . Isolated yield after work up: 93%. GC-MS(ESI): m/z (%) , 130 (M^+, 0.4), 88 (23), 87 (15), 71 (19), 54 (10), 45 (6), 43 (100) . ^{1}H-NMR: (300 MHz, $CDCl_3$, 25°C): δ = 2.29-2.35 (2H, m, CH_2-CH), 2.33 (3H,s, CH_3), 2.94 (2H, t, ^{3}J=7.3Hz, CH_2-CH_2), 5.02-5.11 (2H, m, CH_2=), 5.71-5.85 (1H,m). ^{13}C-NMR : (75 MHz, $CDCl_3$, 25°C) δ = 28.26(CH_2), 30.50 (CH_3),33.50 (CH_2), 116.34(CH_2), 135.99 (CH), 195.46(C=O).

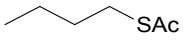


S-Octyl thioacetate (4d) was prepared in accordance to the general procedure.
Octane mesylate (4c) (0.58g, 2.8 mmol) was put in a one neck round bottom flask. In the same flask 20 ml of 0.4 M aqueous potassium carbonate solution were added. 1.5 eq of potassium thioacetate or acetic acid were added dropwise to the solution under stirring. The mixture was stirred at 80 °C under reflux for almost 2 hours. After the usual work up the product was recovered as a yellow liquid obtained in 95% yield (GC-MS) . Isolated yield after work up: 90%. GC-MS comparable to those reported in literature.[22] NMR spectra comparable to those reported in literature.[23]



S-Hexyl thioacetate (5d) was prepared in accordance to the general procedure.

Hexane mesylate (5c) (0.50g, 2.8 mmol) was put in a one neck round bottom flask. In the same flask 20 ml of 0.4 M aqueous potassium carbonate solution were added. 1.5 eq of potassium thioacetate or acetic acid were added dropwise to the solution under stirring. The mixture was stirred at 80 °C under reflux for almost 2 hours. After the usual work up the product was recovered as a yellow liquid obtained in 96% yield (GC-MS) . Isolated yield after work up: 92%. ^{1}H-NMR: (300 MHz, $CDCl_3$, 25°C): δ = 0.86-0.92 (3H, m, CH_3CH_2), 1.25-1.38 (6H,m, $CH_2CH_2CH_2$), 1.51-1.61 (2H, quintet, ^{3}J=7.4Hz, CH_2), 2.32 (3H, s, CH_3), 2.86 (2H,t, ^{3}J=7.2Hz,CH_2S). ^{13}C-NMR : (75 MHz, $CDCl_3$, 25°C) δ = 13.94, 22.47, 28.46, 29.15, 29.45, 31.28, 32.78, 208.89 (C=O).

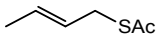


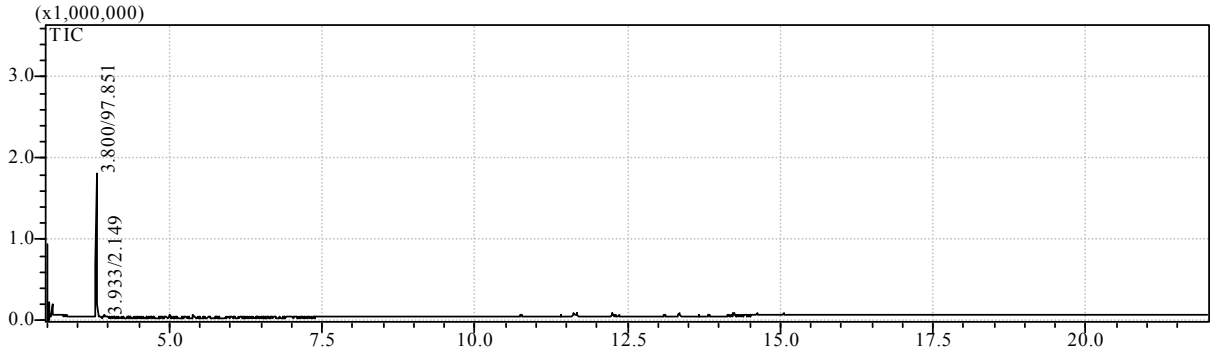
S-Butyl thioacetate (6d) was prepared in accordance to the general procedure.

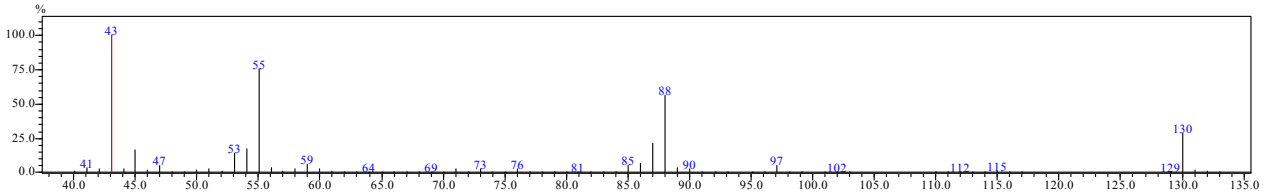
Butane mesylate (6c) (0.42g, 2.8 mmol) was put in a one neck round bottom flask. In the same flask 20 ml of 0.4 M aqueous potassium carbonate solution were added. 1.5 eq of potassium thioacetate or acetic acid were added dropwise to the solution under stirring. The mixture was stirred at 80 °C under reflux for almost 2 hours. After the usual work up the product was recovered as a yellow liquid obtained in 97% yield (GC-MS) . Isolated yield after work up: 95%. GC-MS comparable to those reported in literature.[24] ^{1}H-NMR: (300 MHz, $CDCl_3$, 25°C): δ = 0.90 (3H, t, ^{2}J=7.5, CH3CH2), 1.37 (2H,sext, ^{2}J=7.4Hz, $CH_3CH_2CH_2$), 1.49-1.59 (2H, quintet, ^{3}J=7.5Hz, CH_2-CH_2-CH_2), 2.31 (3H, s, CH_3C=O), 2.86 (2H,t, ^{3}J=7.4Hz,CH_2S). ^{13}C-NMR : (75 MHz, $CDCl_3$, 25°C) δ = 13.53, 21.91, 28.83, 31.56, 195.91 (C=O). NMR spectra comparable to those reported in literature.[25]

a. GC-MS SPECTRA

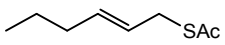
***trans*-2-butenyl thioacetate (3b)**

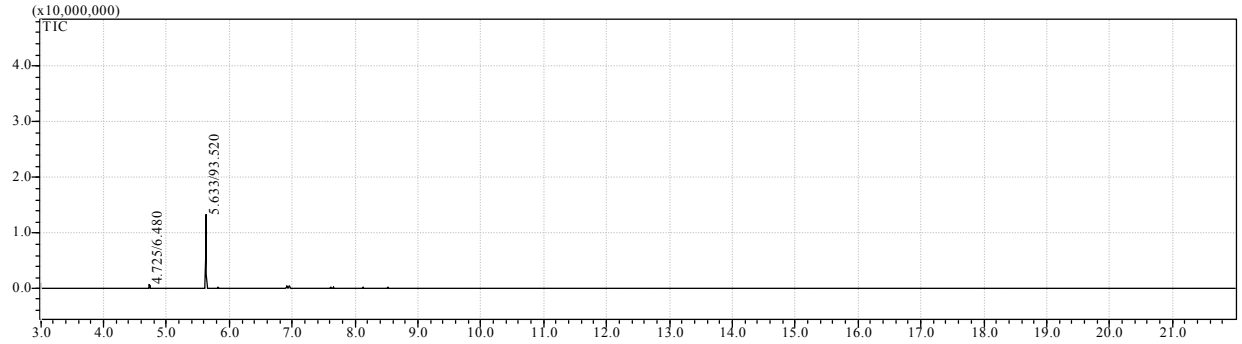


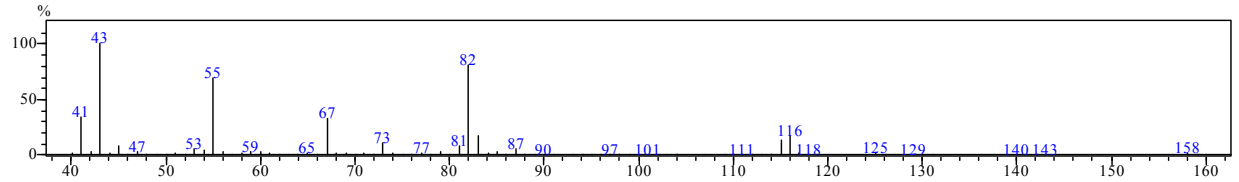




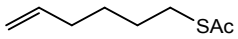
***trans*-2-hexenyl thioacetate (4b)**

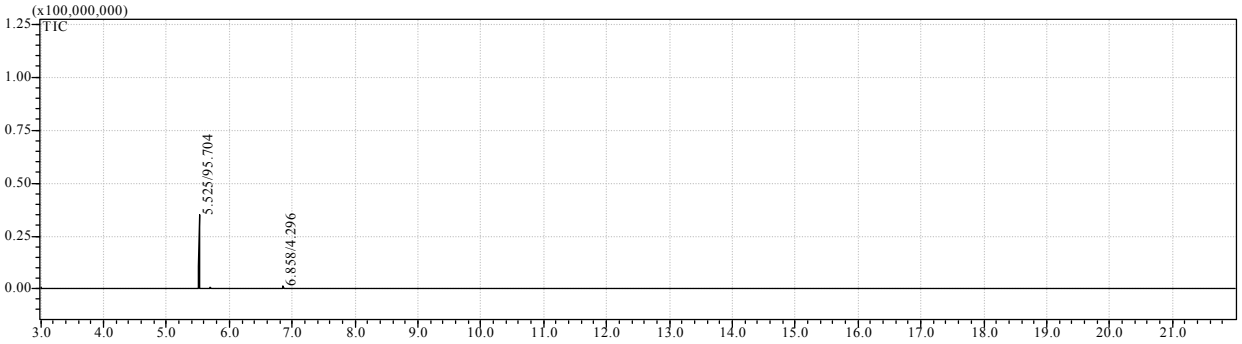


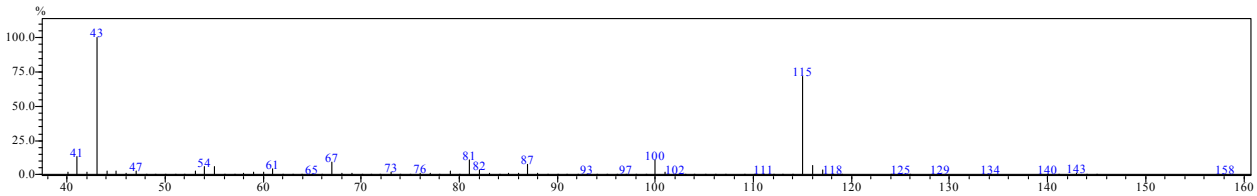




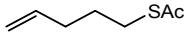
5-Acetylsulfanyl-hex-1-ene (1d)

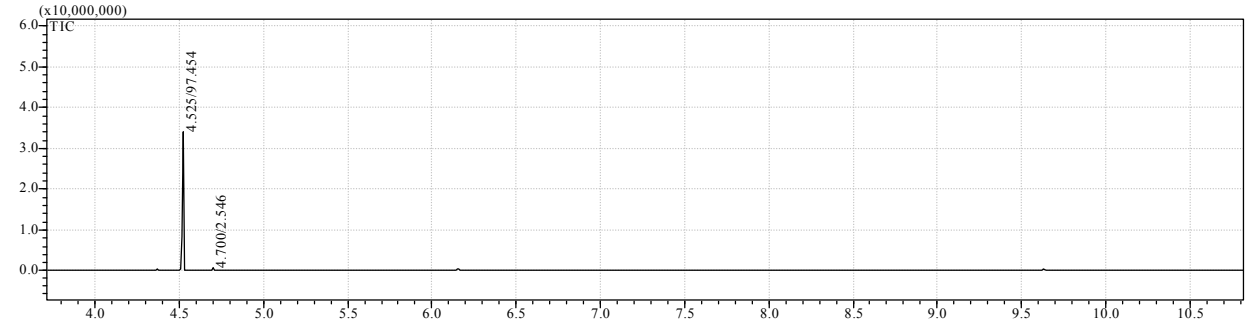


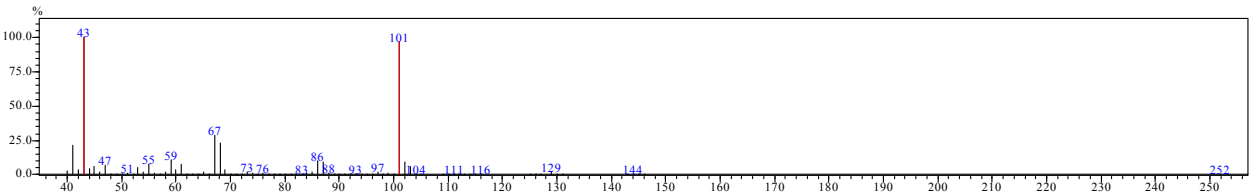




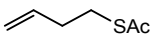
S-Pent-4-en-1-yl ethanethioate (2d)

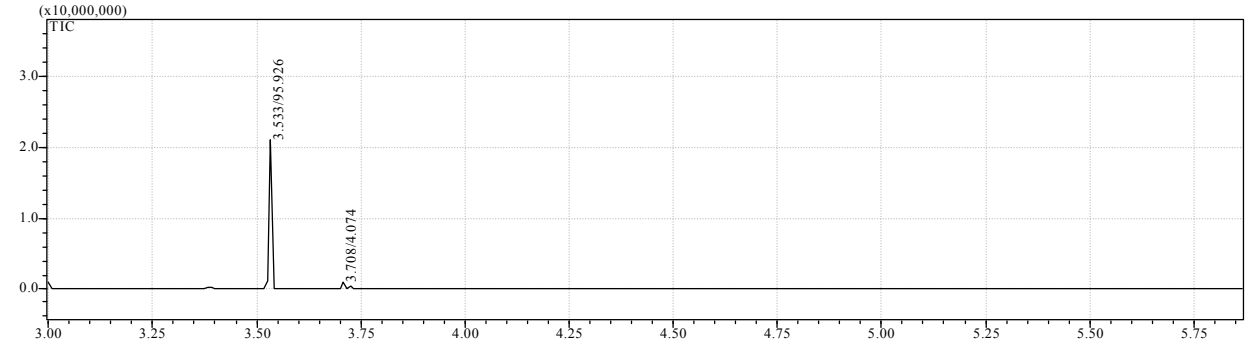


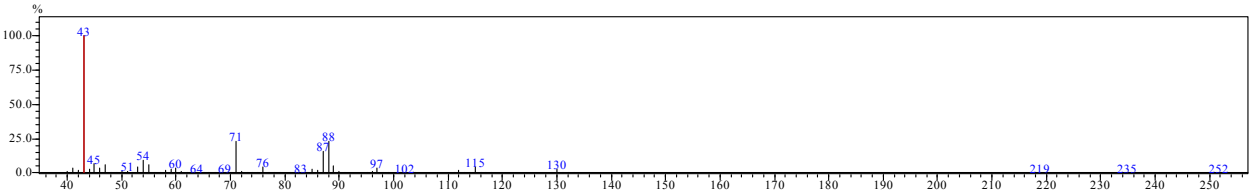




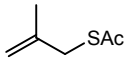
Thioacetic acid S-but-3-enyl ester (3d)

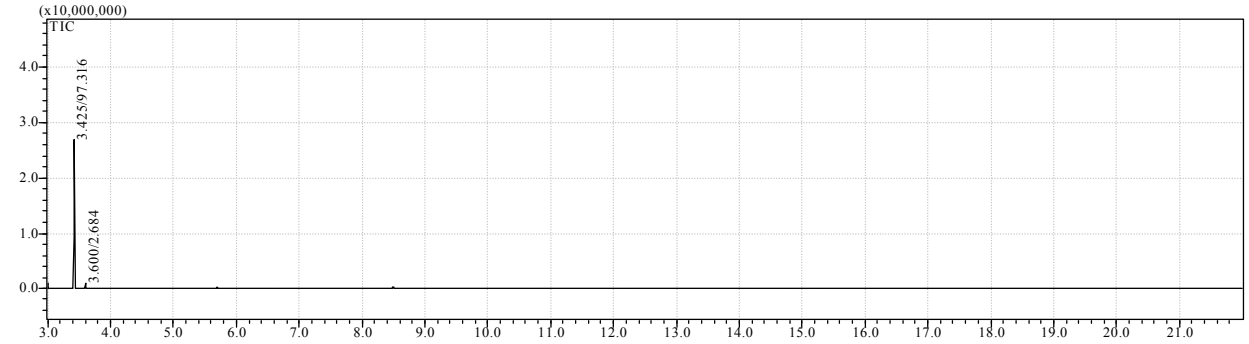


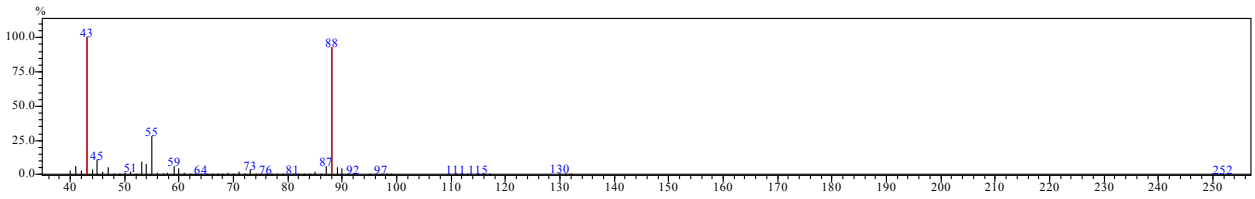




2-methylallyl thioacetate (5b)

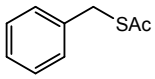




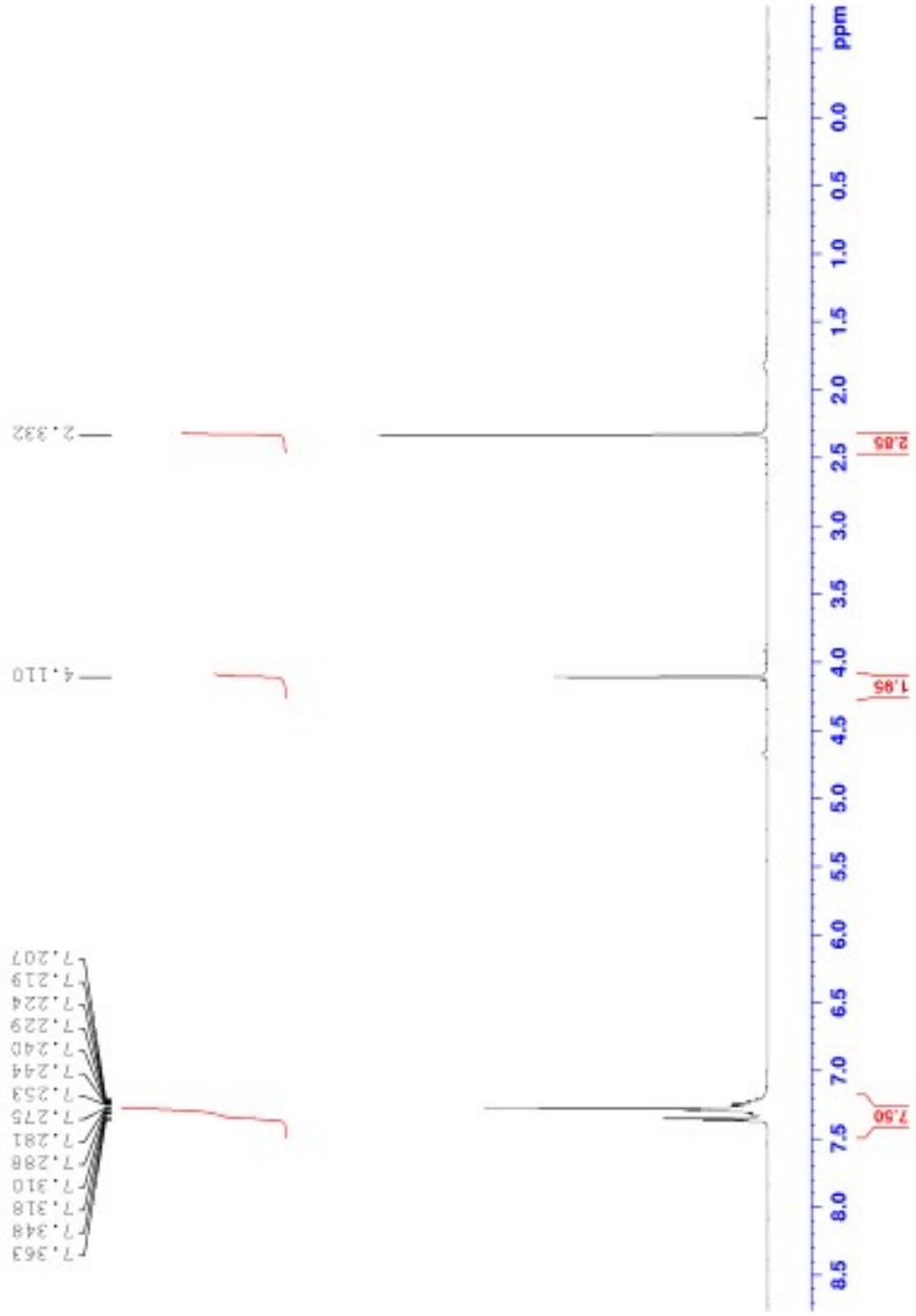


b. NMR SPECTRA

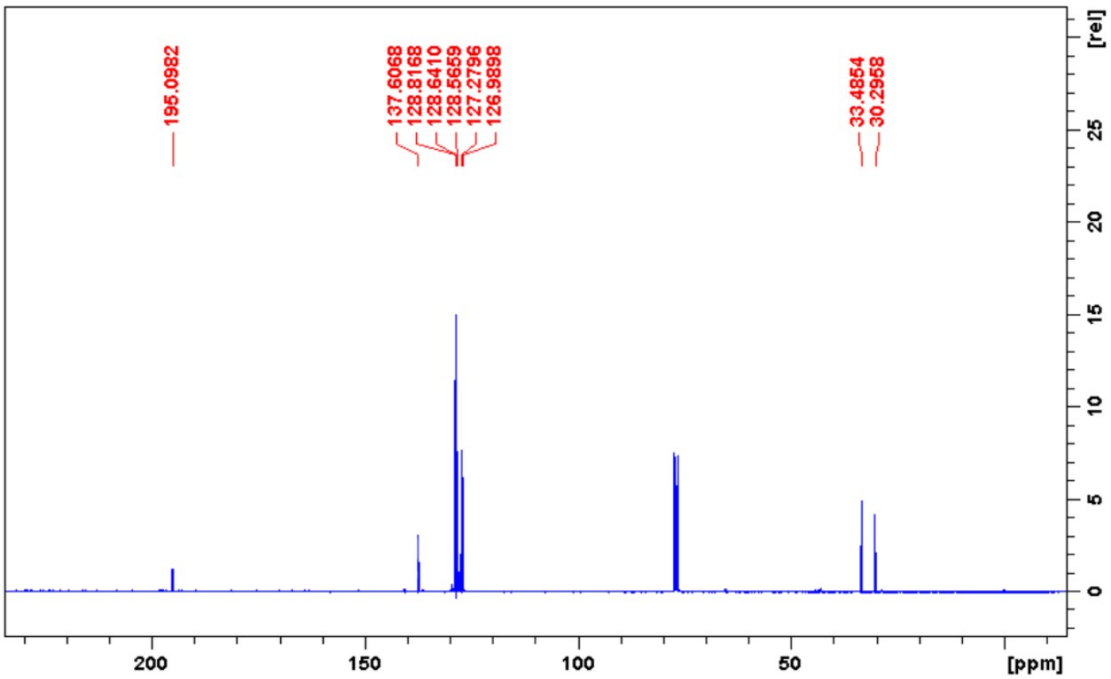
Benzyl thioacetate (1b)

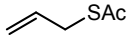


^{1}H-NMR

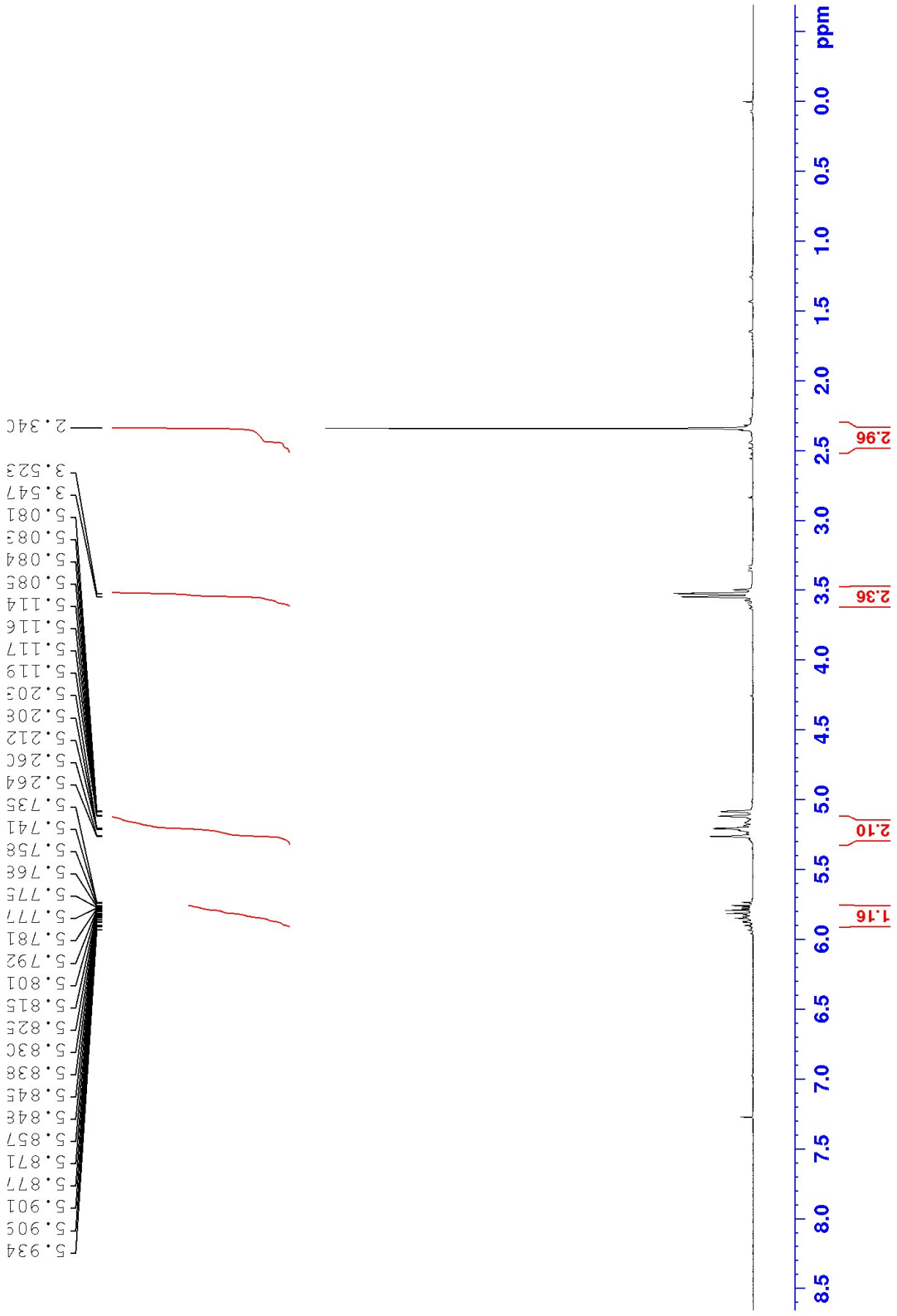


^{13}C-NMR



Allyl thioacetate (2b) 

^{1}H-NMR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4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
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3.547
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5.083
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5.085
5.114
5.116
5.117
5.119
5.203
5.208
5.212
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5.264
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5.741
5.758
5.768
5.775
5.777
5.781
5.792
5.801
5.815
5.825
5.830
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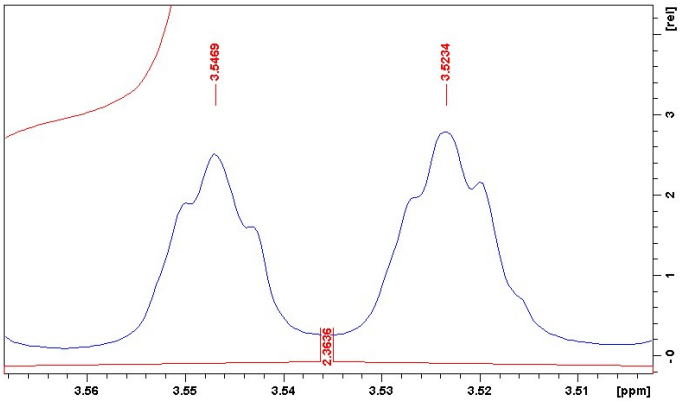
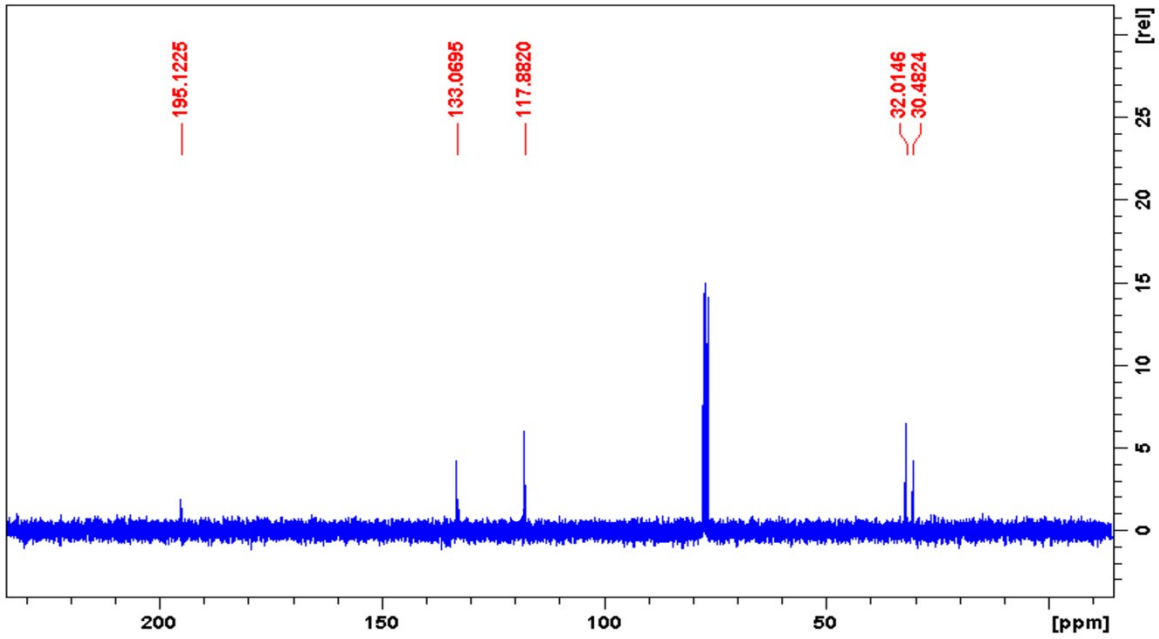
5.845
5.848
5.857
5.871
5.877
5.901
5.905
5.934

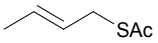
Doublet



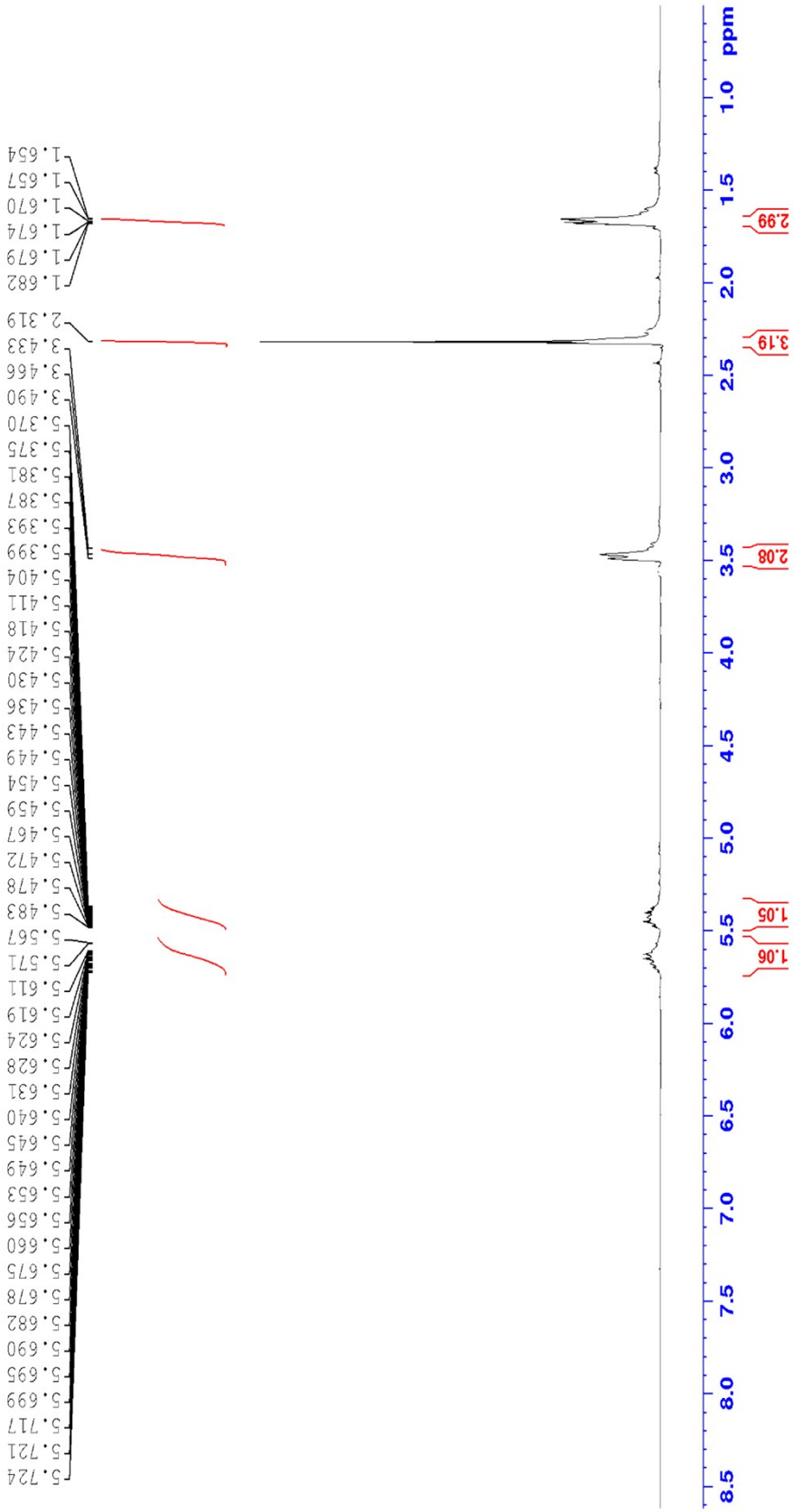
^{13}C-NMR



***trans*-2-butenyl thioacetate (3b)**



^{1}H-NMR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1.682
2.319
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3.490
5.370
5.375
5.381
5.387
5.393
5.399
5.404
5.411
5.418
5.424
5.430
5.436
5.443
5.449
5.454
5.459
5.467
5.472
5.478
5.483
5.567
5.571
5.611
5.619
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5.628
5.631
5.640
5.645
5.649
5.653
5.656
5.660
5.675
5.678
5.682
5.690
5.695
5.699
5.717
5.721
5.724
ppm
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1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
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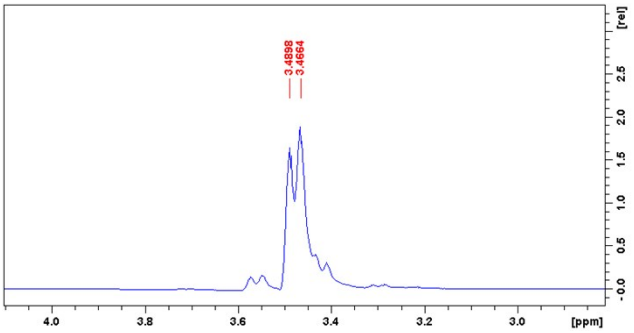
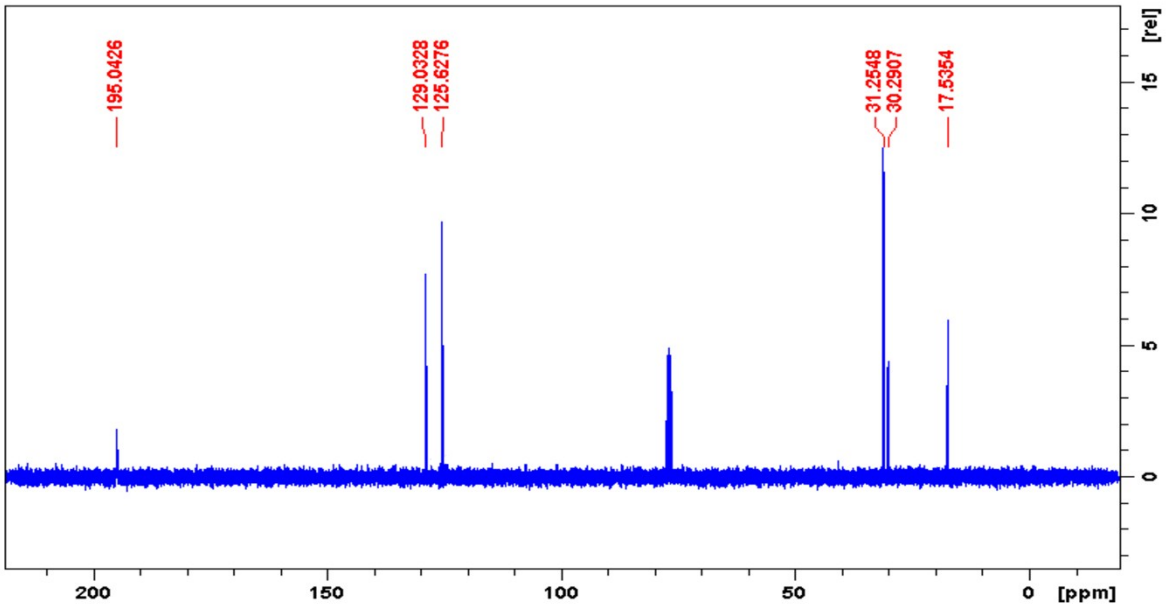
3.19
2.08
1.05
1.06

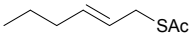
Doublet



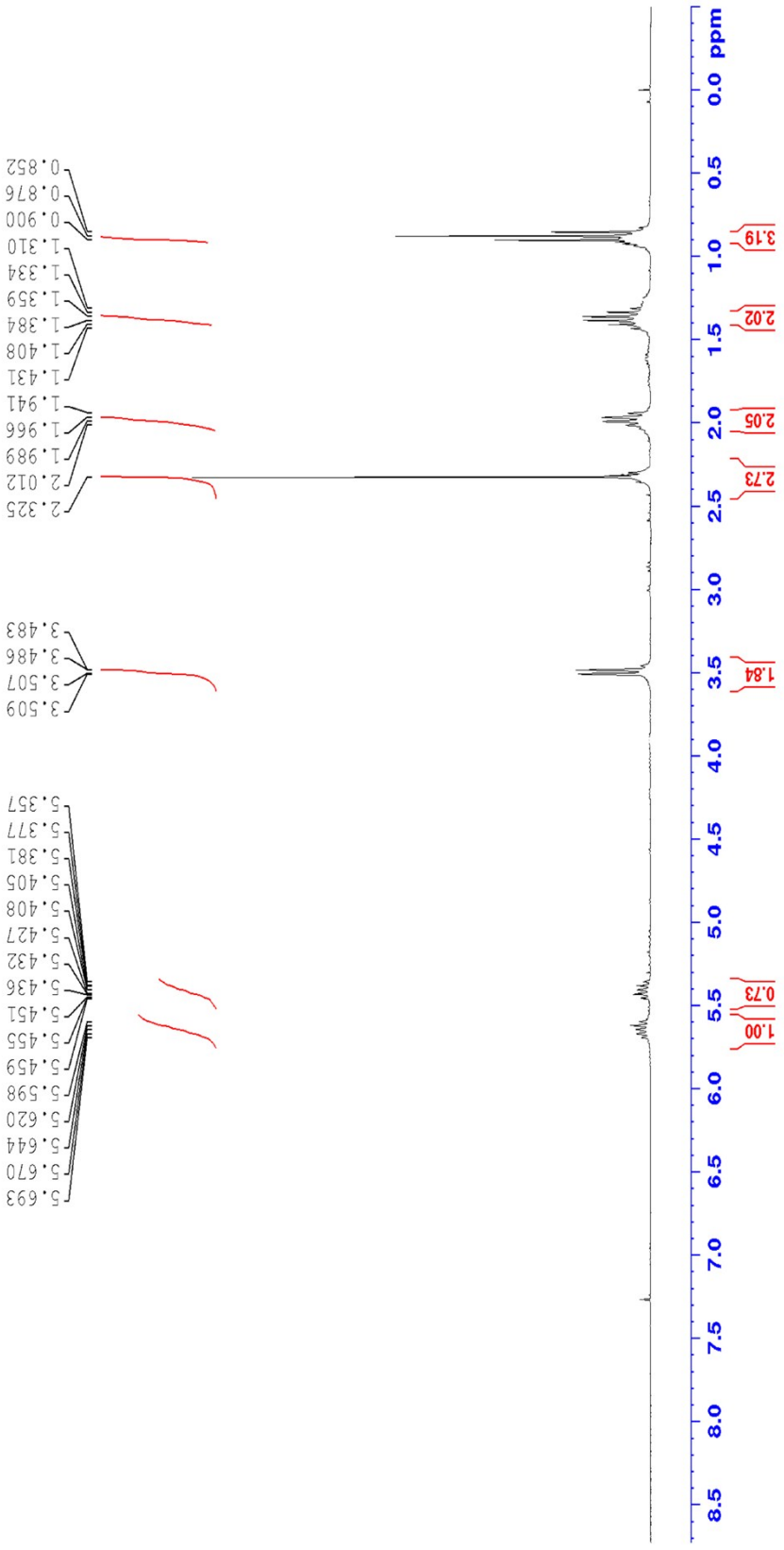
^{13}C-NMR



***trans*-2-hexenyl thioacetate (4b)**



^{1}H-NMR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1.408
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1.966
1.989
2.012
2.325
3.483
3.486
3.507
3.509
5.357
5.377
5.381
5.405
5.408
5.427
5.432
5.436
5.451
5.455
5.459
5.598
5.620
5.644
5.670
5.693
3.19
2.02
2.05
2.73
1.84
0.73
1.00
ppm
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0.5
1.0
1.5
2.0
2.5
3.0
3.5
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5.0
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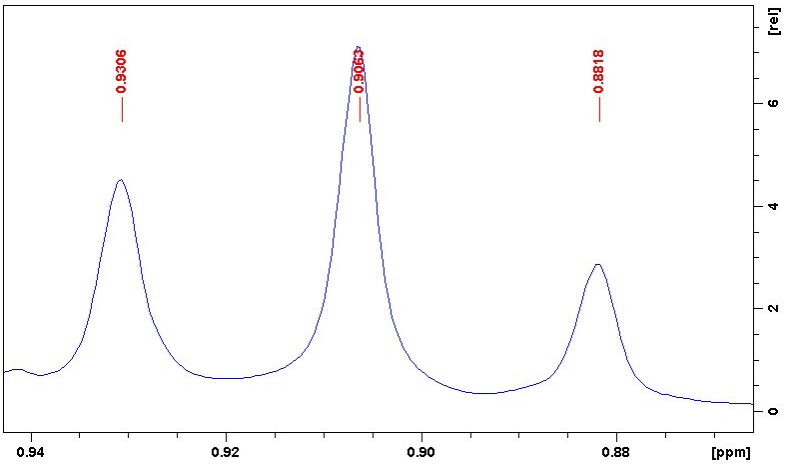
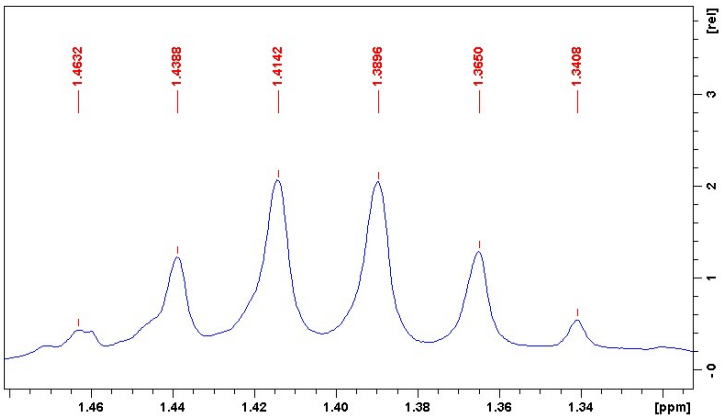
6.0
6.5
7.0
7.5
8.0
8.5

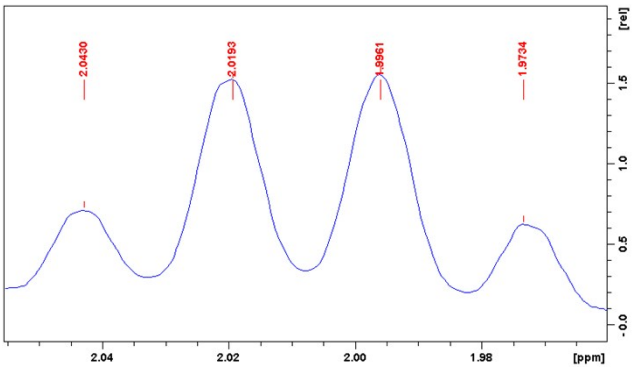
Triplet



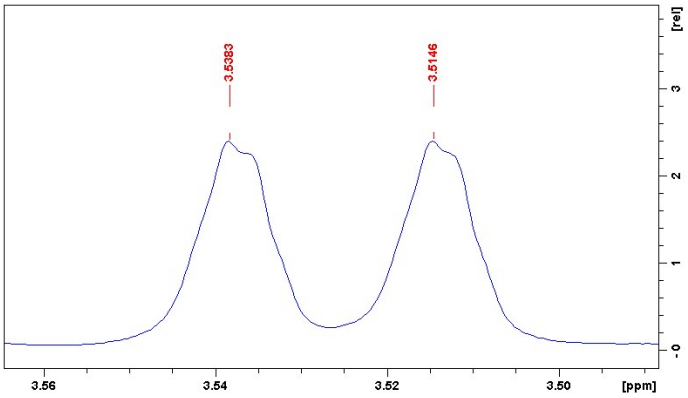
Sextet



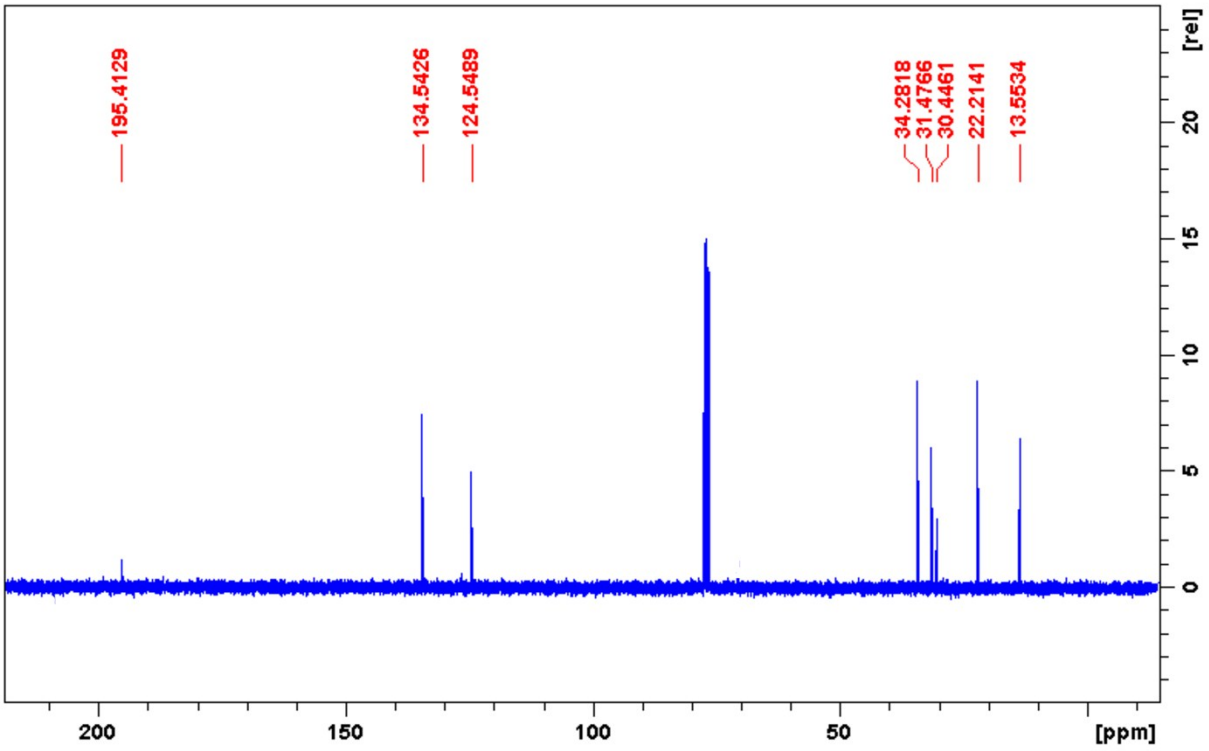
Doublet of triplets



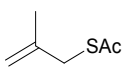
Doublet



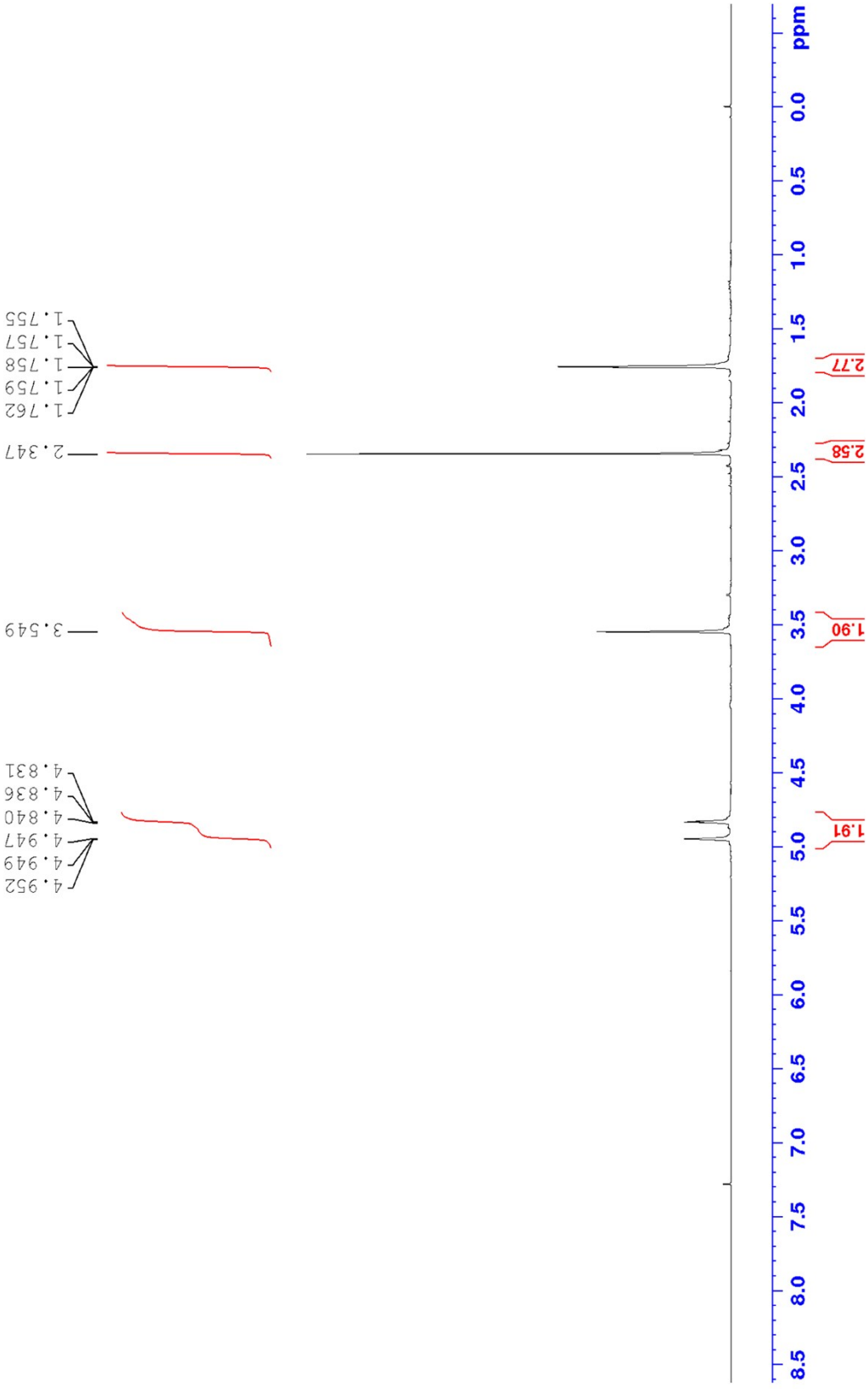
^{13}C-NMR



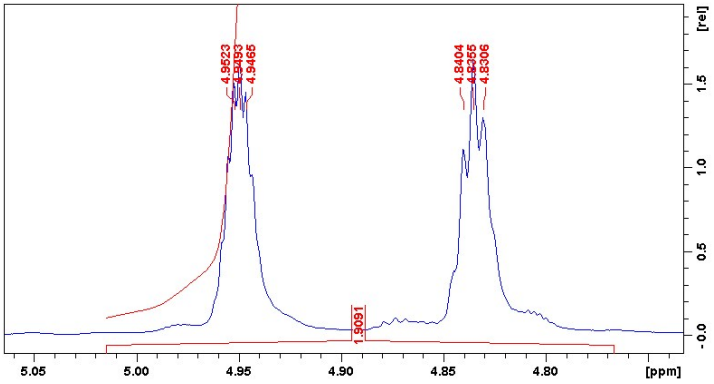
2-methylallyl thioacetate (5b)



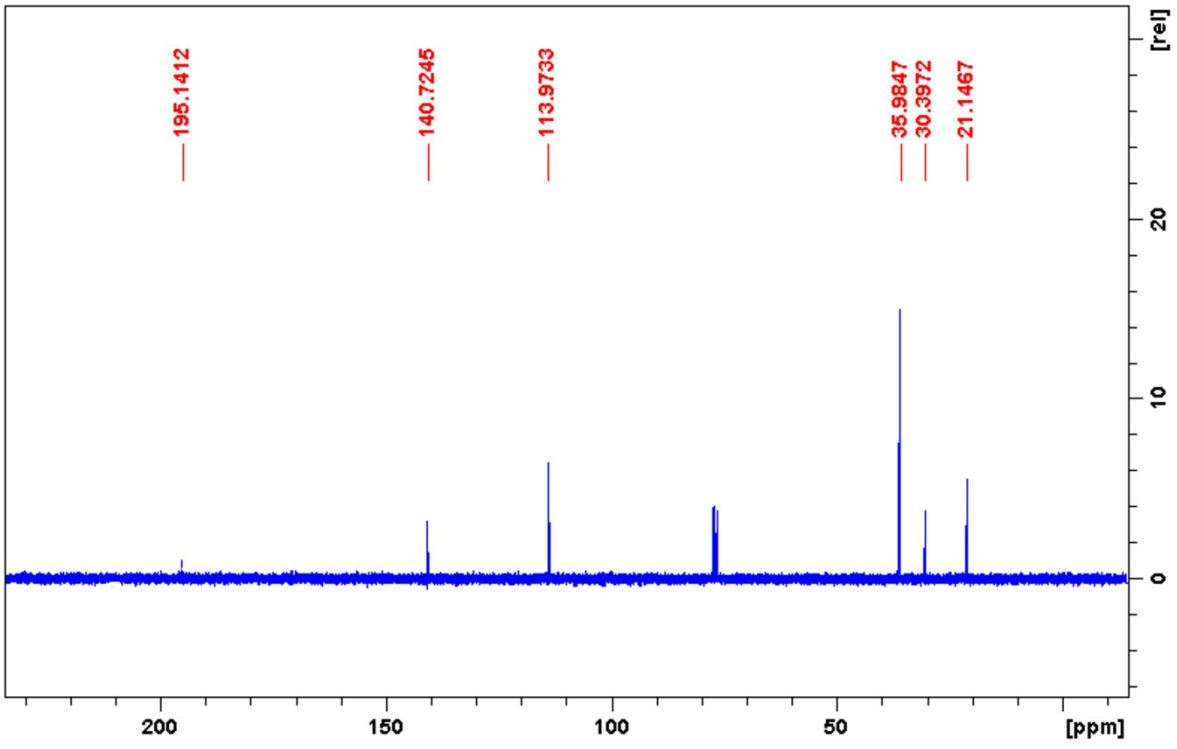
^{1}H-NMR



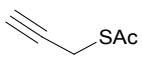
Doublet of triplets



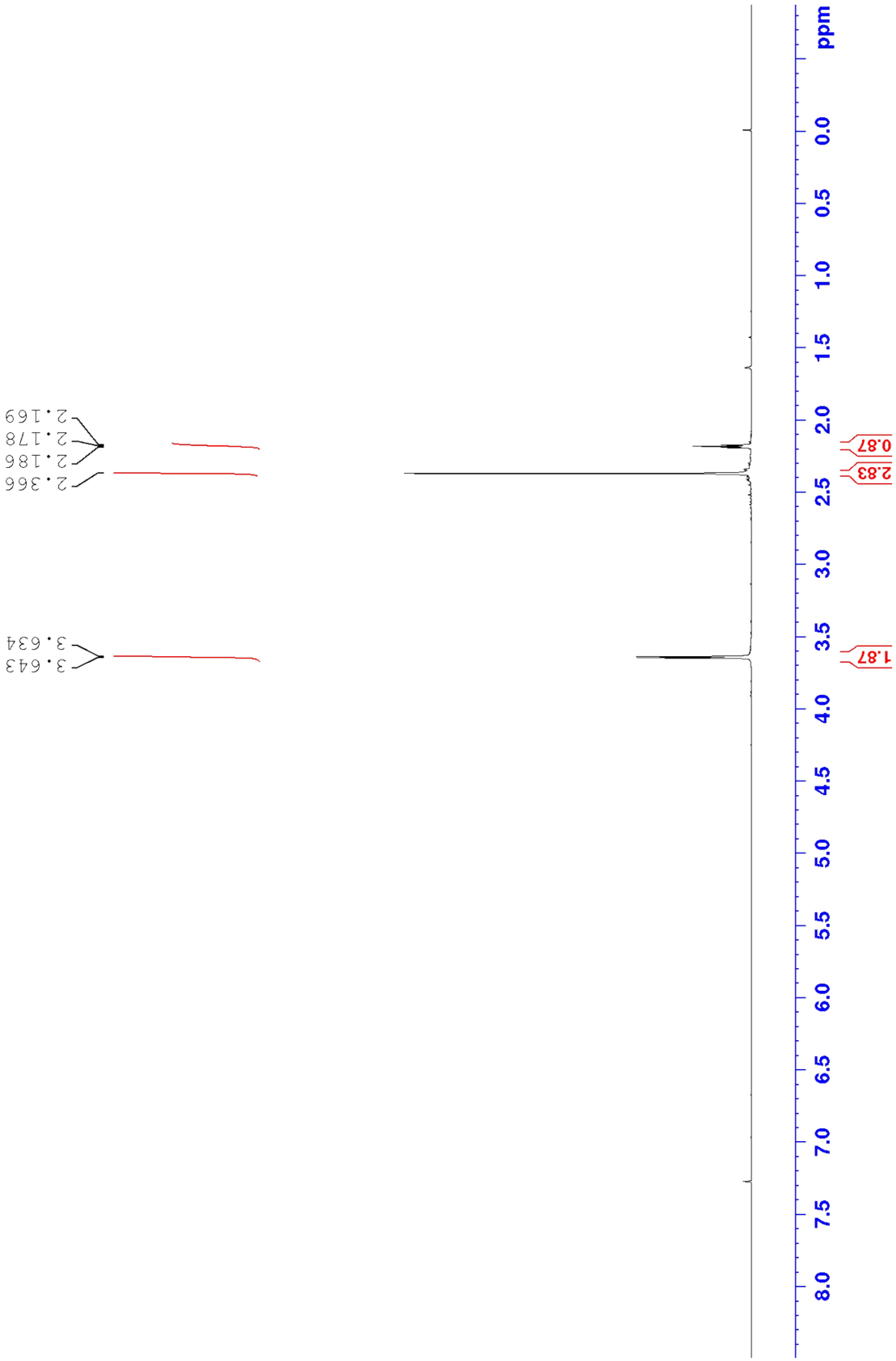
^{13}C-NMR



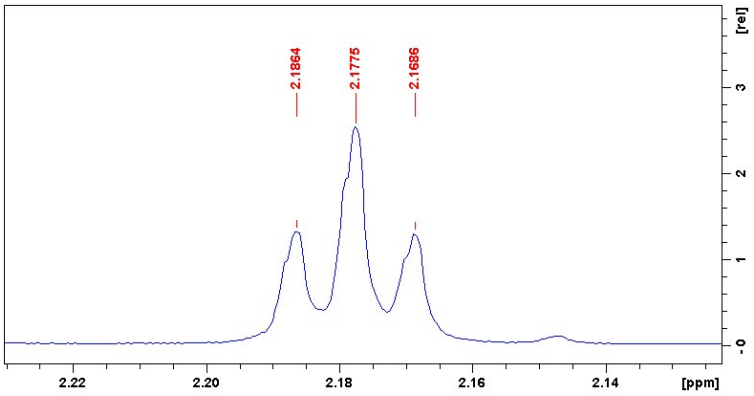
S-Prop-2-ynyl-thioacetate (6b)



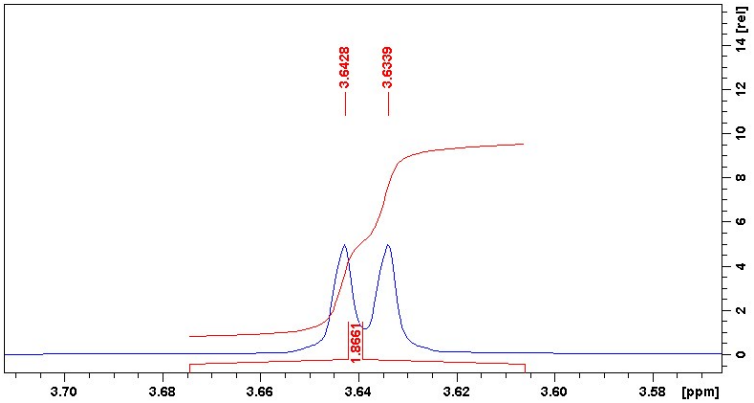
^{1}H-NMR



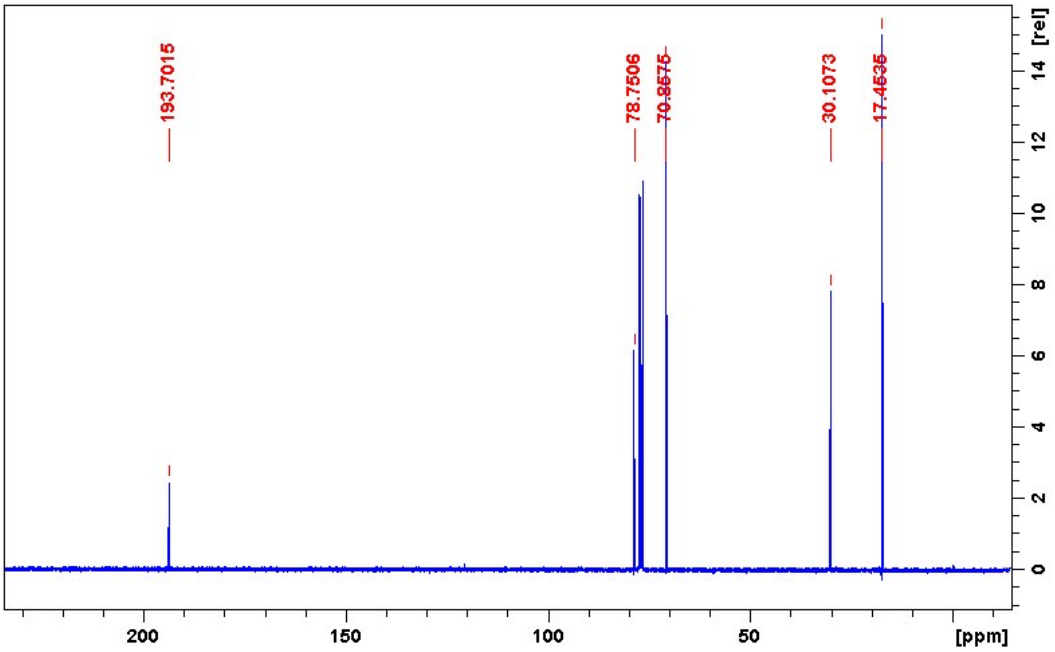
Triplet



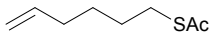
Doublet



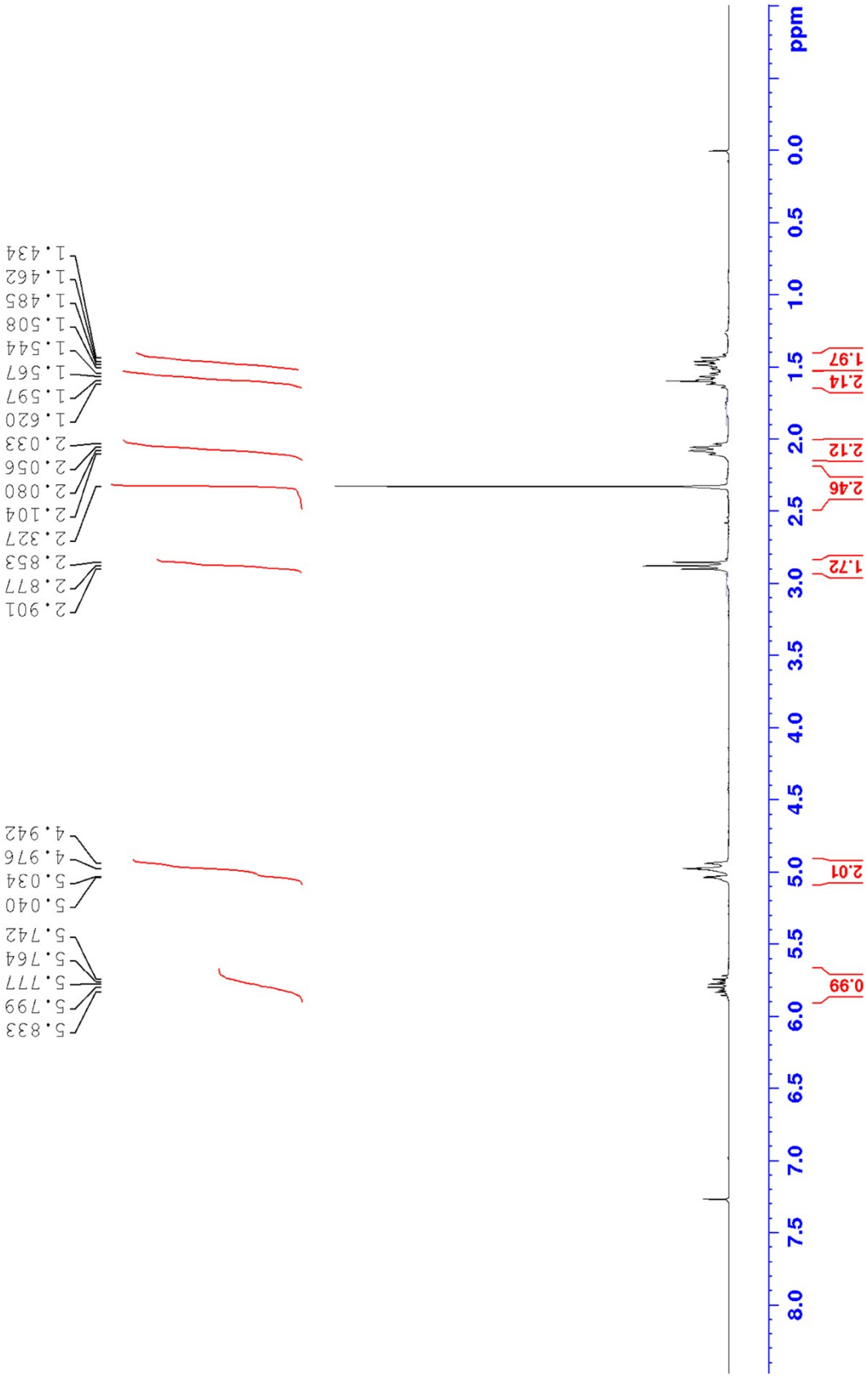
^{13}C-NMR



5-Acetylsulfanyl-hex-1-ene (1d)



^{1}H-NMR


ppm
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1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
0.99
2.01
1.72
2.46
2.12
2.14
1.97
1.434
1.462
1.485
1.508
1.544
1.567
1.597
1.620
2.033
2.056
2.080
2.104
2.327
2.853
2.877
2.901
4.942
4.976
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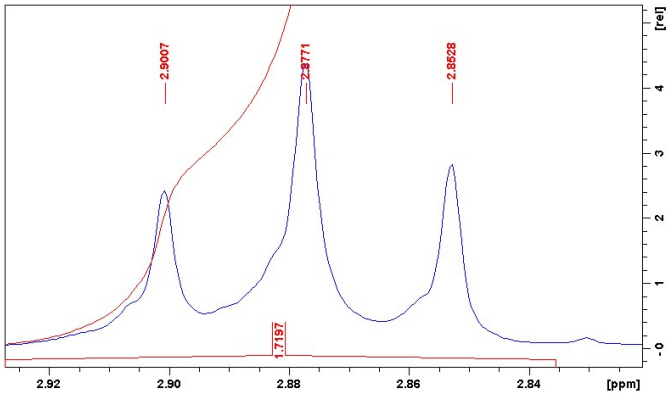
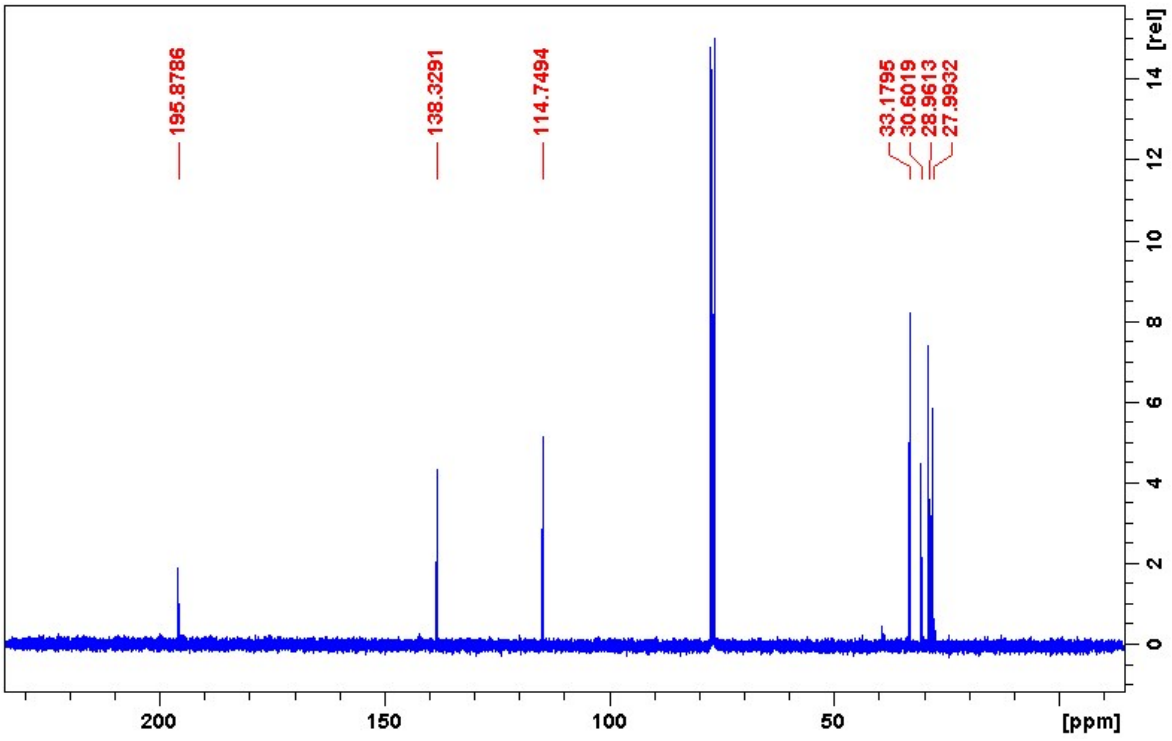
5.040
5.742
5.764
5.777
5.799
5.833

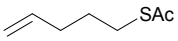
Triplet



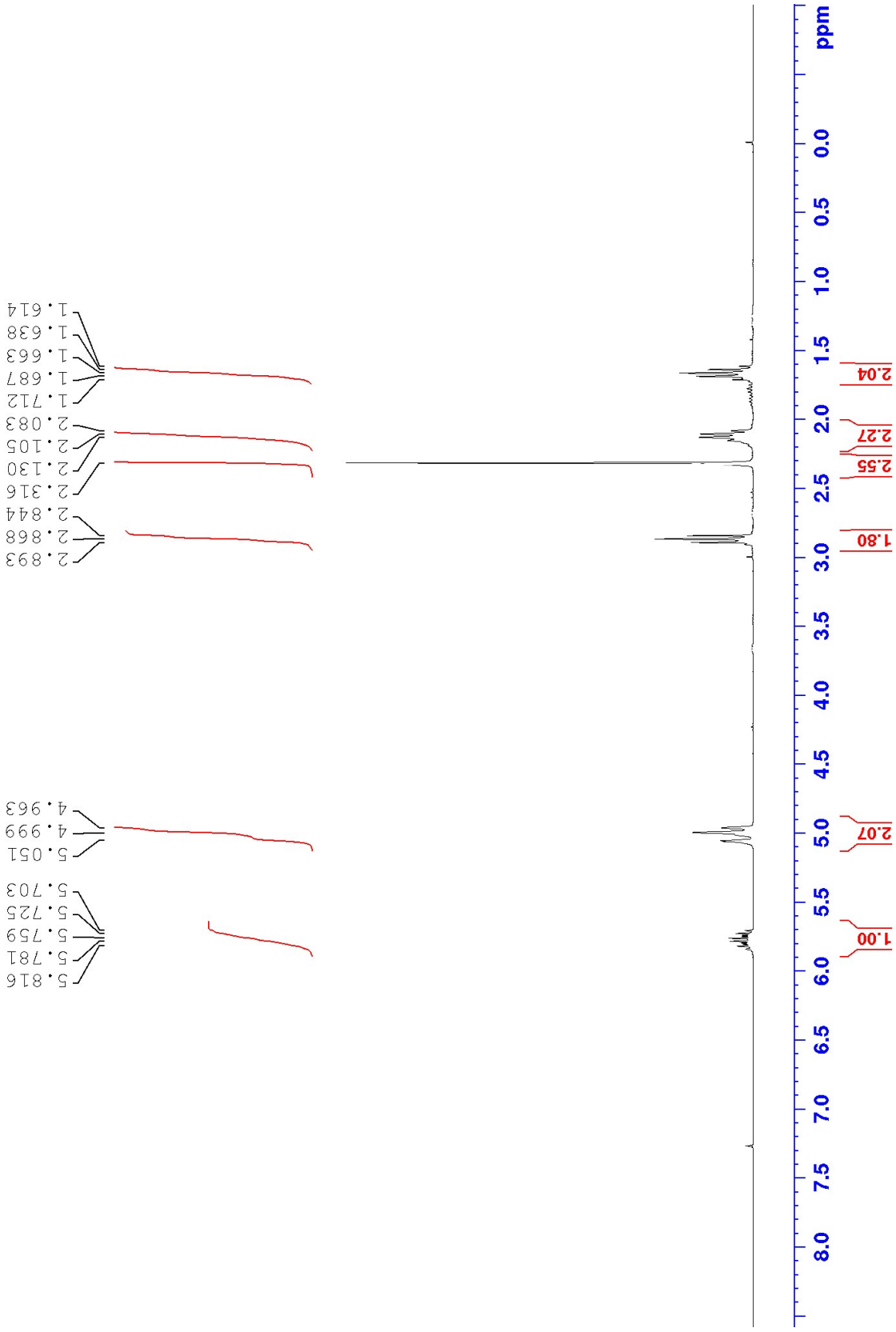
^{13}C-NMR



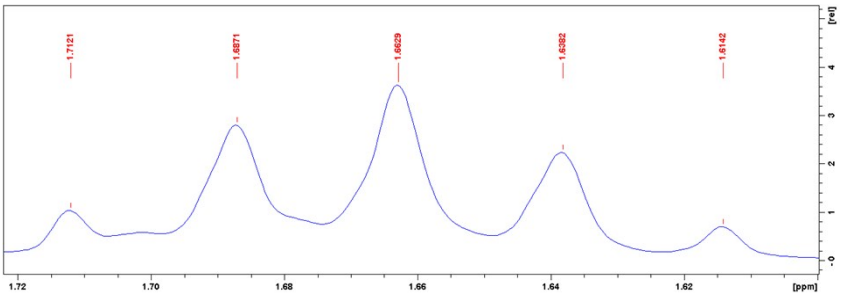
S-Pent-4-en-1-yl ethanethioate (2d)



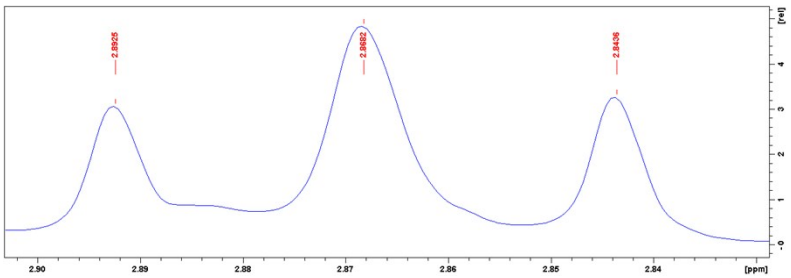
^{1}H-NMR



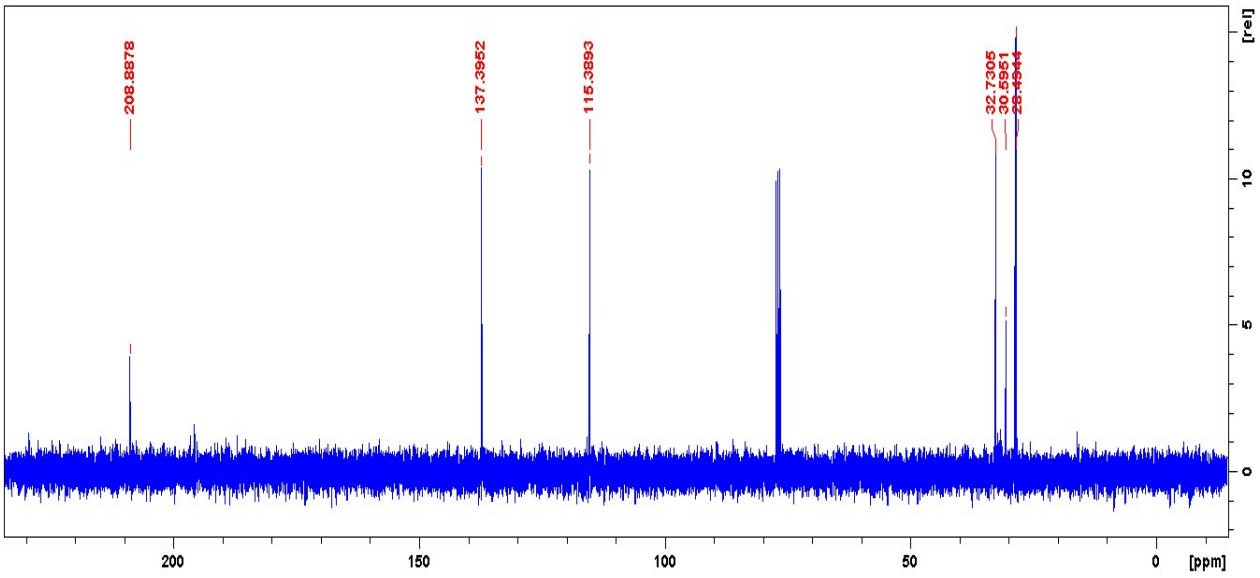
Quintet

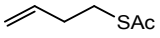


Triplet

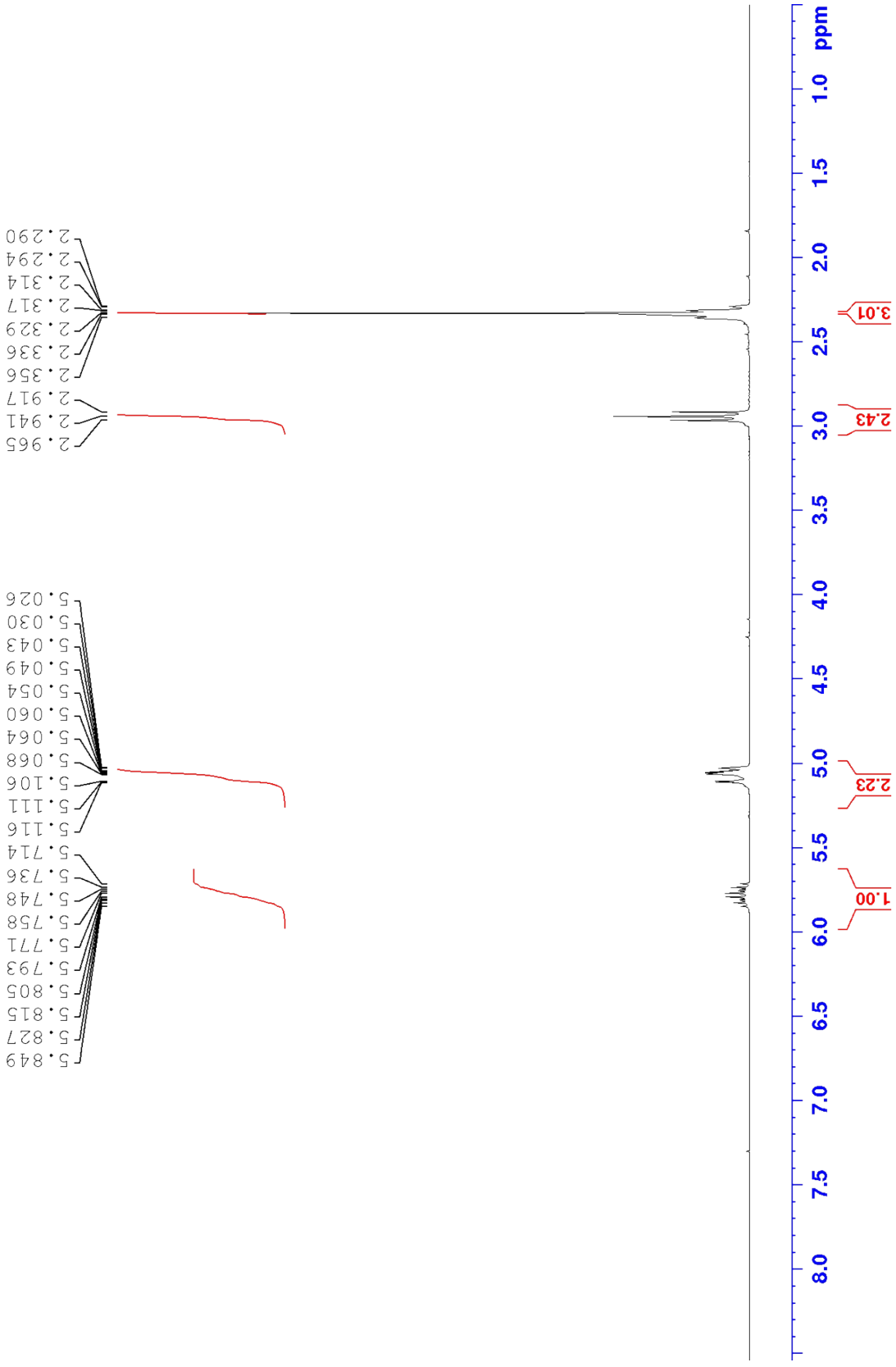


^{13}C-NMR



Thioacetic acid S-but-3-enyl ester (3d) 

^{1}H-NMR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2.314
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2.329
2.336
2.356
2.917
2.941
2.965
5.026
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5.043
5.049
5.054
5.060
5.064
5.068
5.106
5.111
5.116
5.714
5.736
5.748
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5.815
5.827
5.849
3.01
2.43
2.23
1.00
ppm
8.0
7.5
7.0
6.5
6.0
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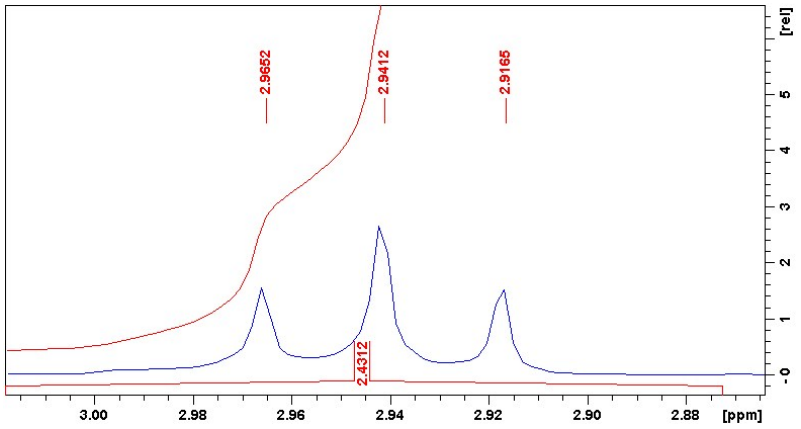
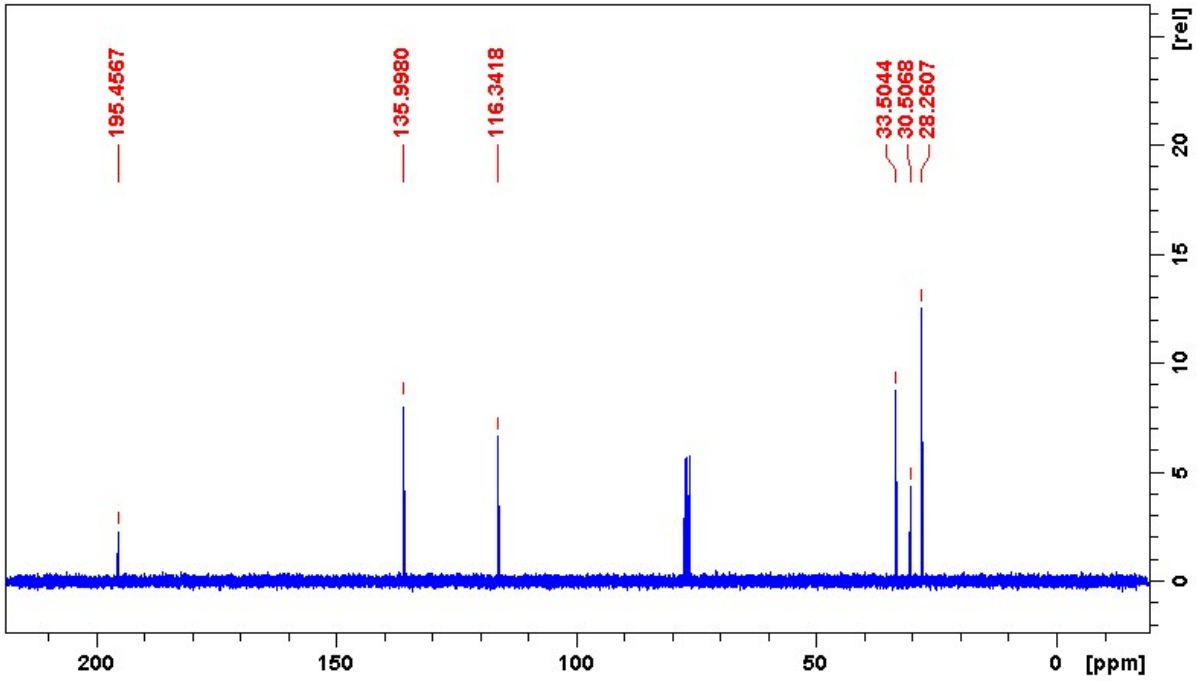
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0

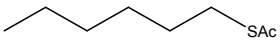
Triplet



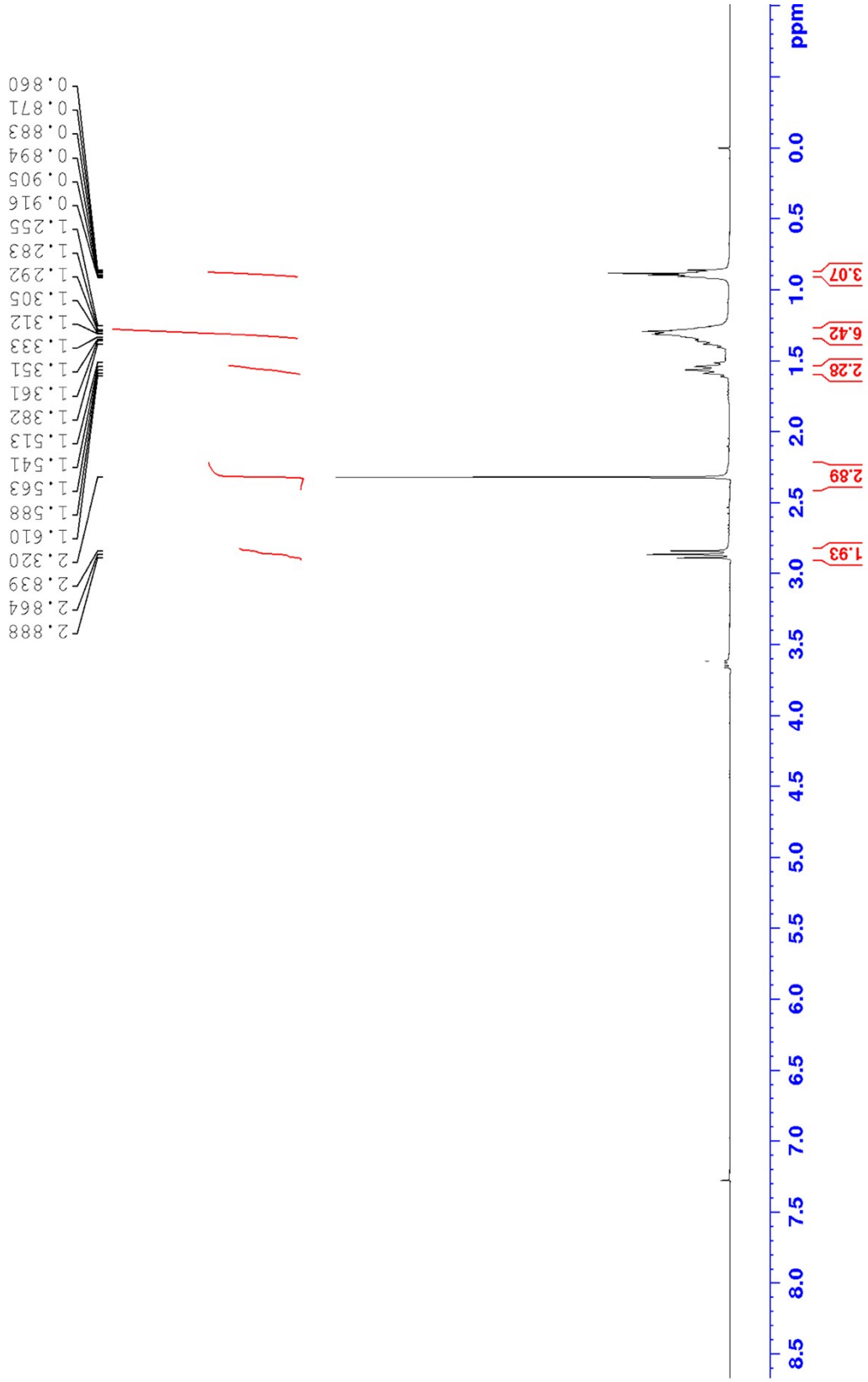
^{13}C-NMR



S-Hexyl thioacetate (5d)



^{1}H-NMR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0.905
0.916
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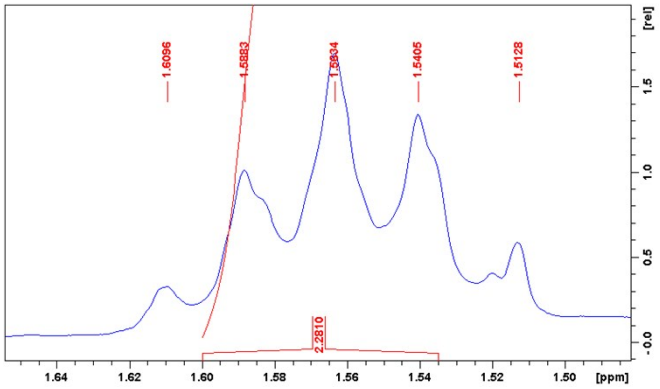
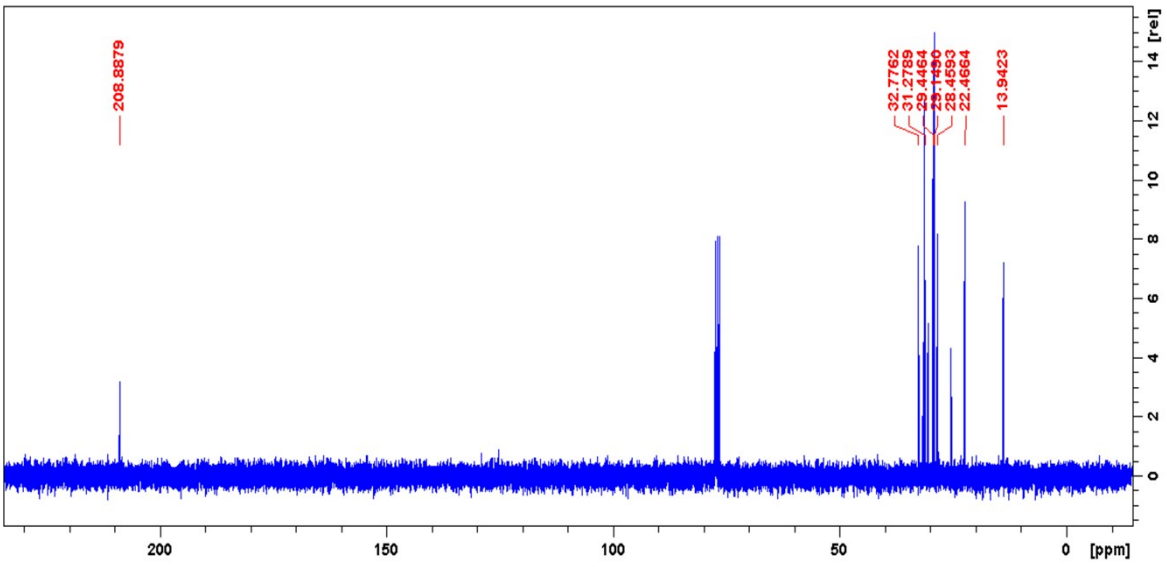
1.283
1.292
1.305
1.312
1.333
1.351
1.361
1.382
1.513
1.541
1.563
1.588
1.610
2.320
2.839
2.864
2.888
3.07
6.42
2.28
2.89
1.93
ppm
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5

Quintet

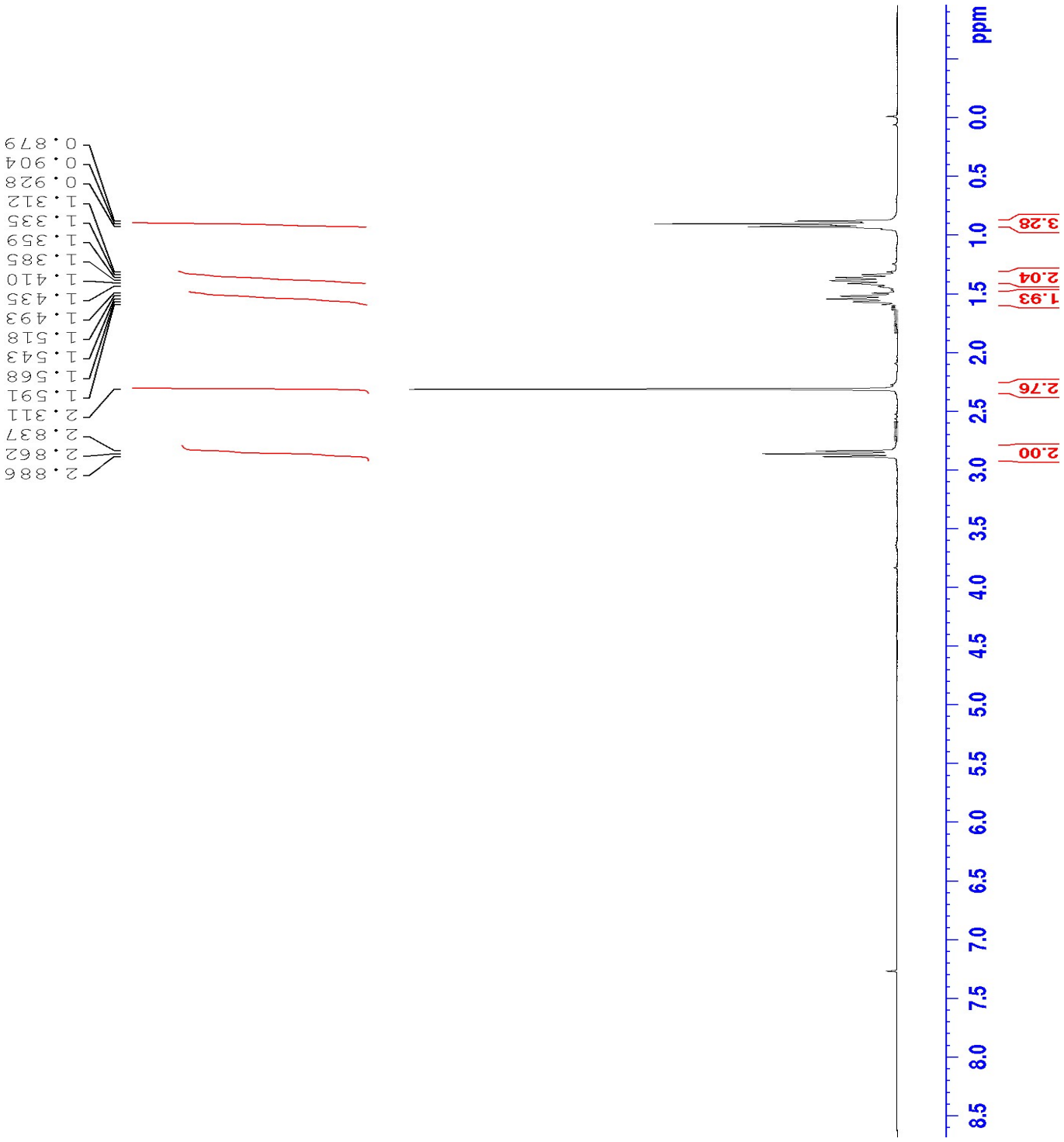

1.6096
1.5883
1.5834
1.5405
1.5128
2.2810
[rel]
1.5
1.0
0.5
-0.0
1.64
1.62
1.60
1.58
1.56
1.54
1.52
1.50
[ppm]

^{13}C-NMR

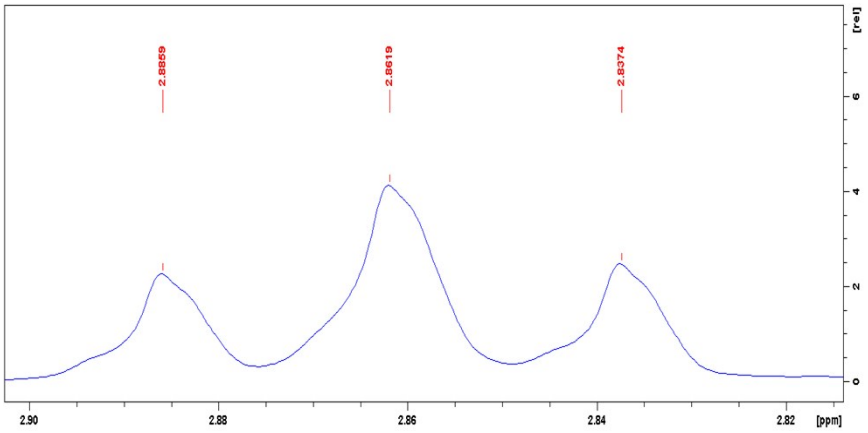

208.8879
32.7762
31.2789
29.4464
23.1430
28.4593
22.4664
13.9423
[rel]
14
12
10
8
6
4
2
0
200
150
100
50
0
[ppm]

S-Butyl thioacetate (6d)

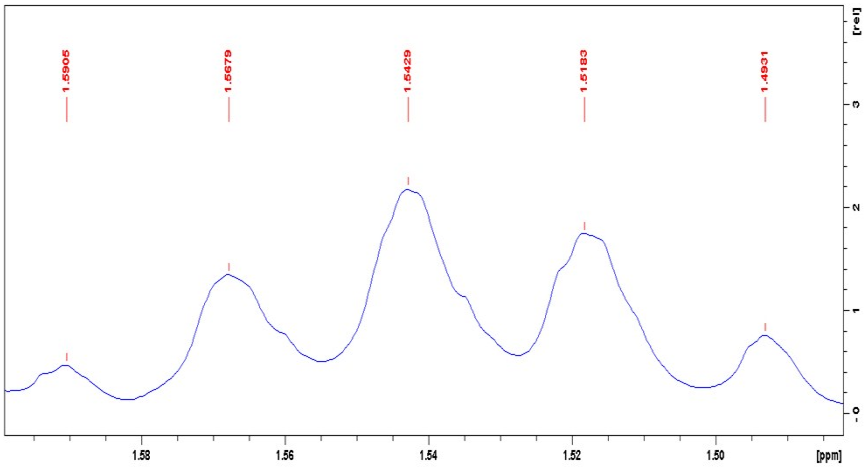
^{1}H-NMR



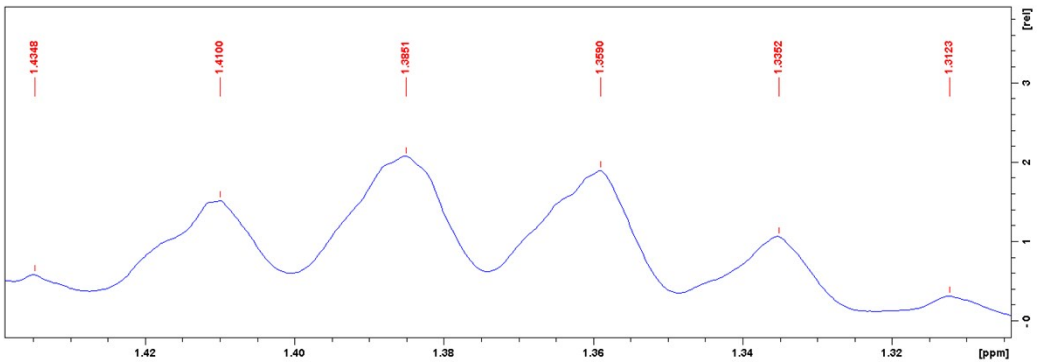
Triplet



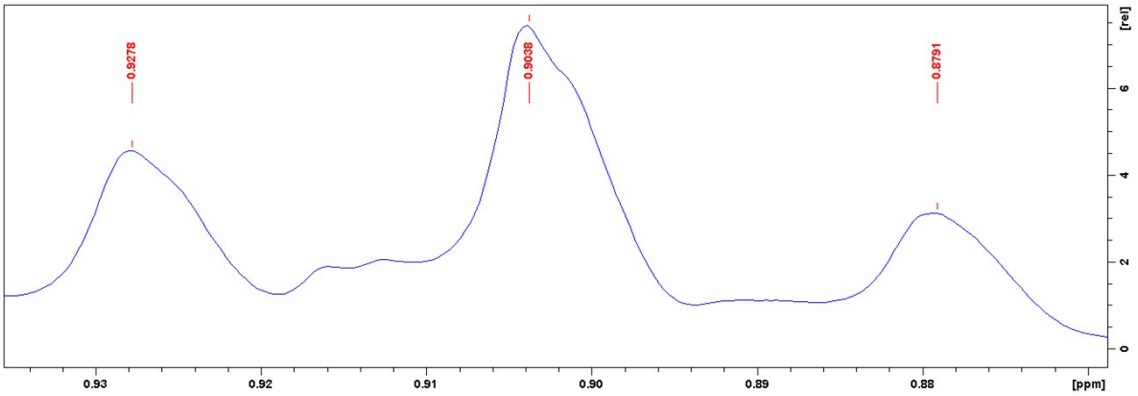
Quintet



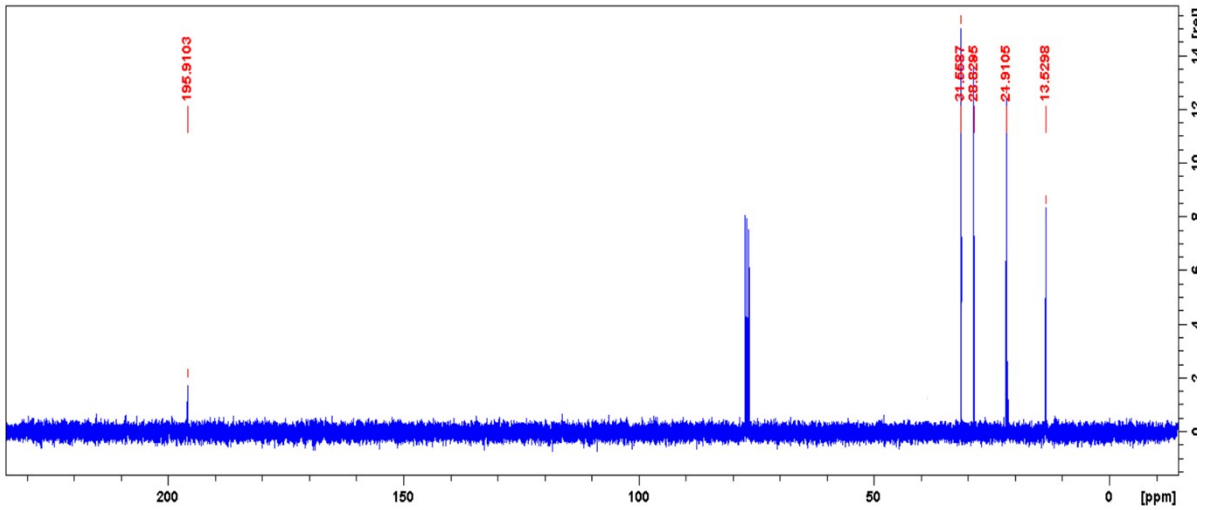
Sextet



Triplet



^{13}C-NMR



8. Notes and references

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