Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information (NMR Spectra)

Stereoselective synthesis of *trans*-THF rings using oxidative cyclisation–radical deoxygentaion sequence: Application to the formal synthesis of *trans*-(2*R*,5*R*)-linalool oxide

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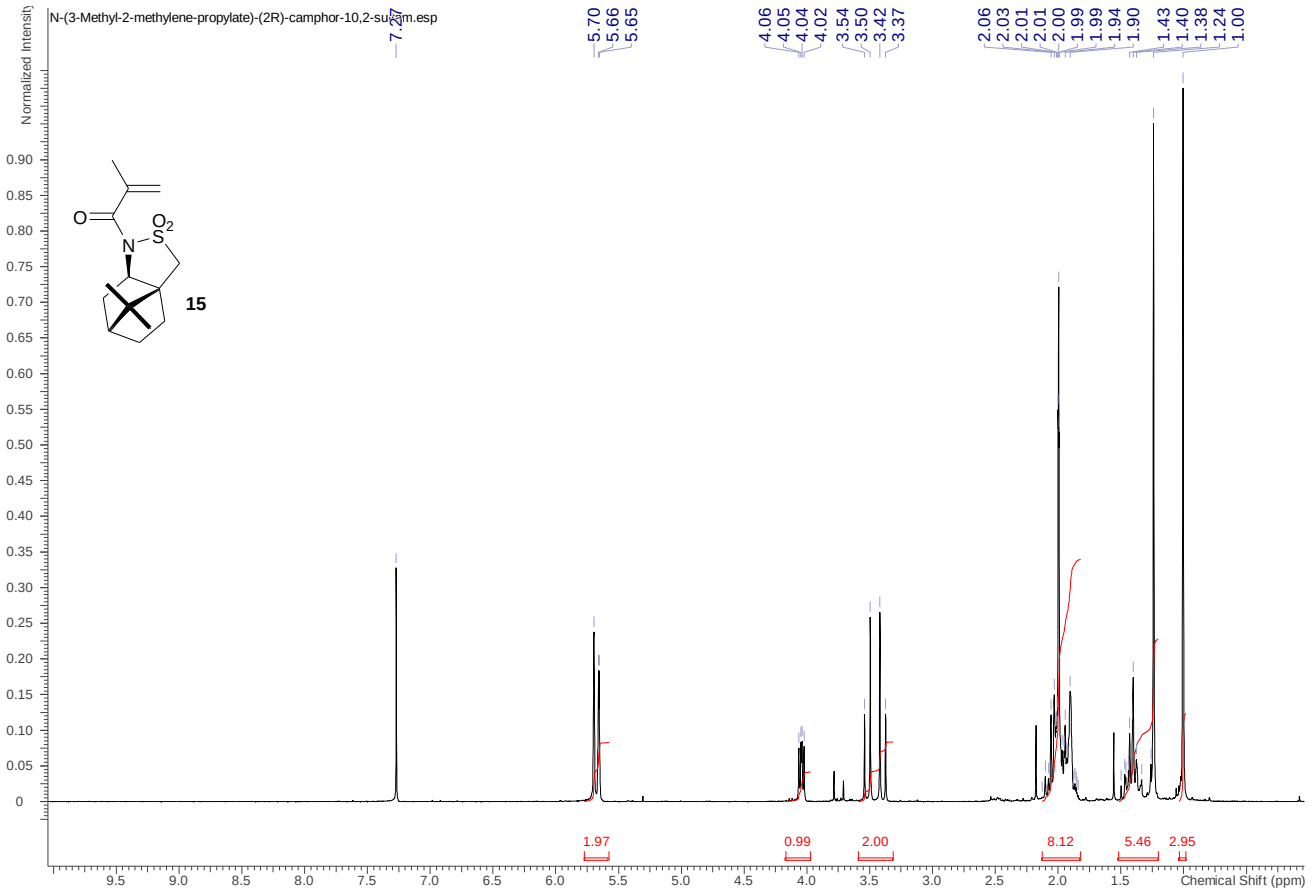
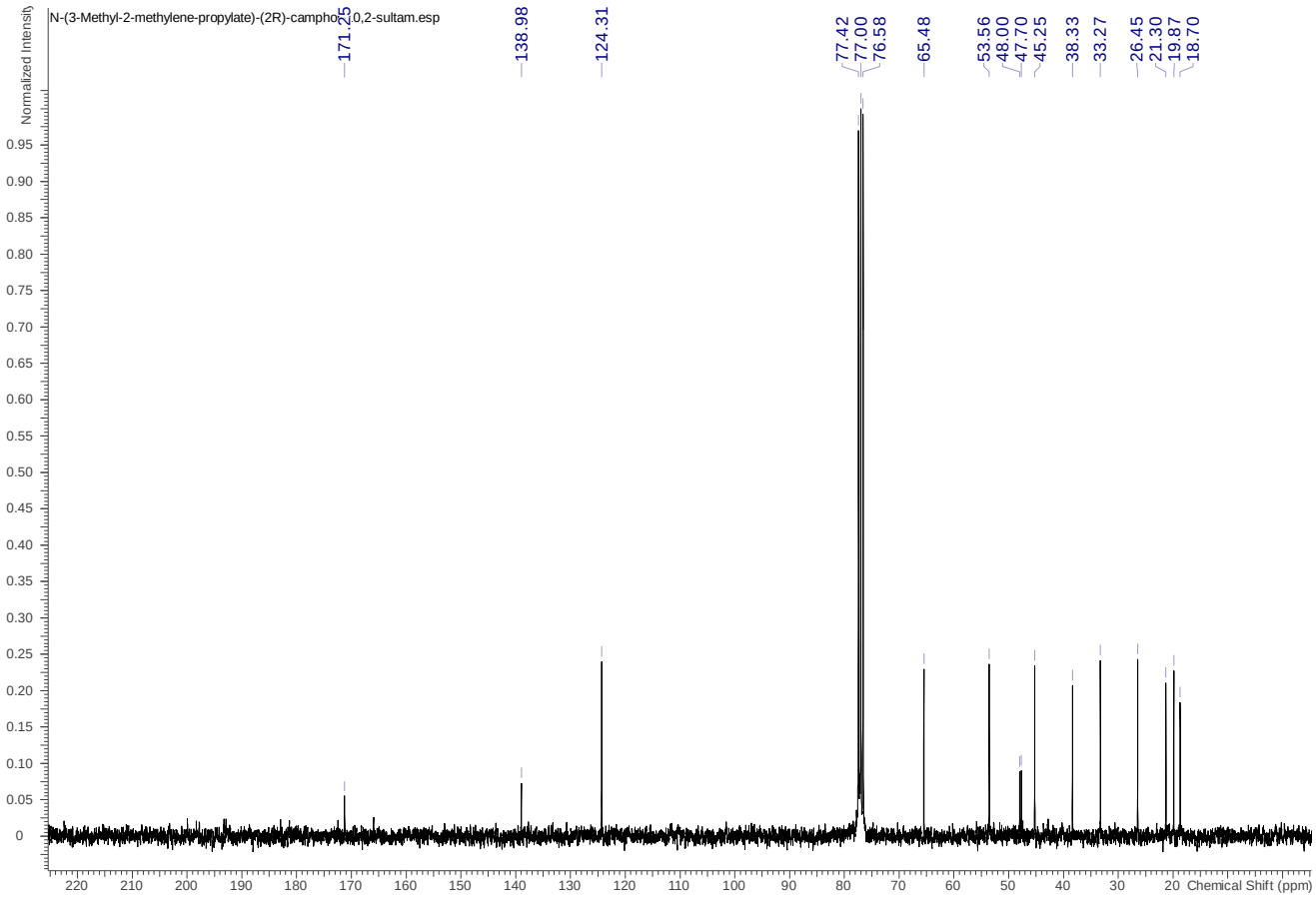
Copies of NMR spectra

N-(3-Methyl-2-methylene-propylate)-(2*R*)-camphor-10,2-sultam (15)

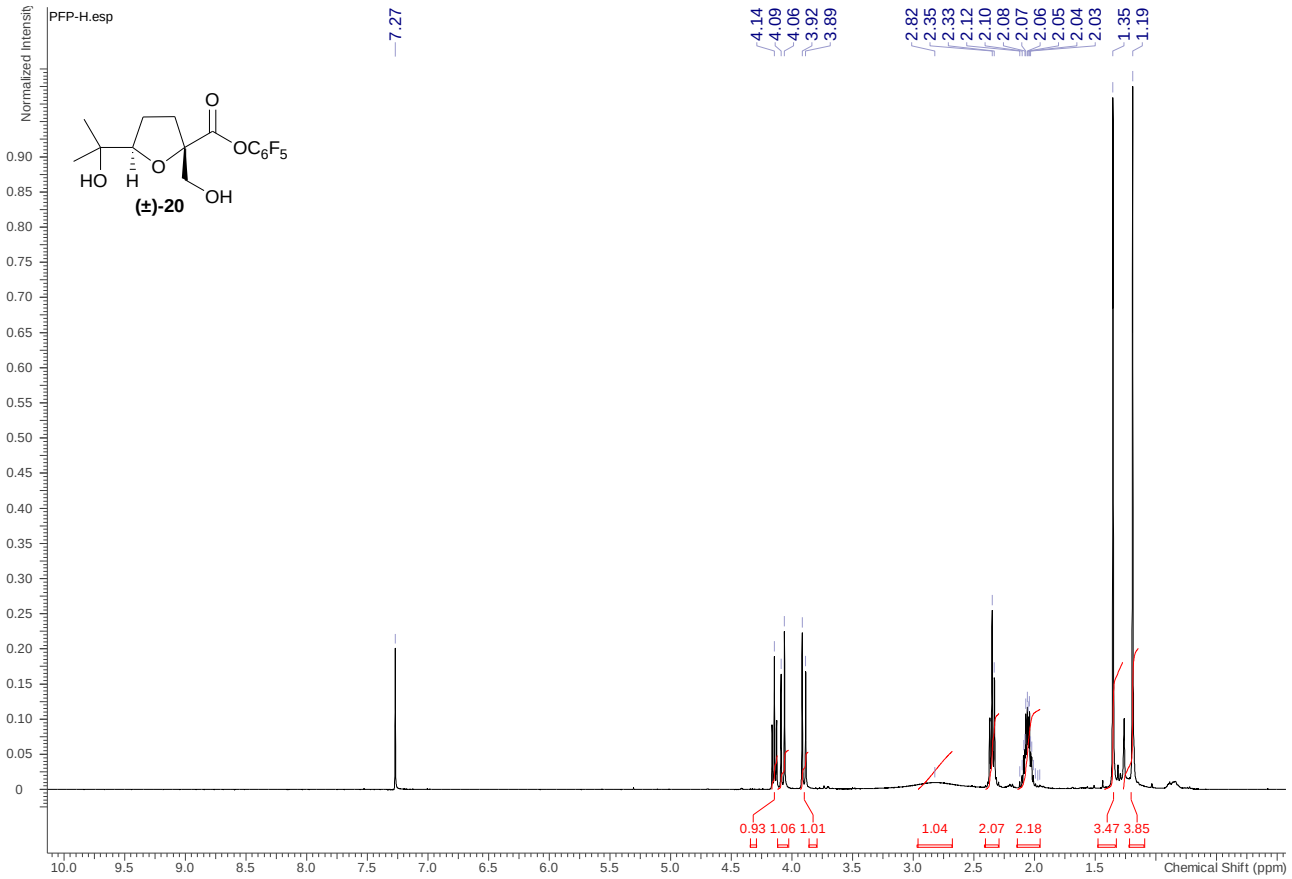
^{1}H NMR (300 MHz, $CDCl_3$) and ^{13}C NMR (75 MHz, $CDCl_3$)

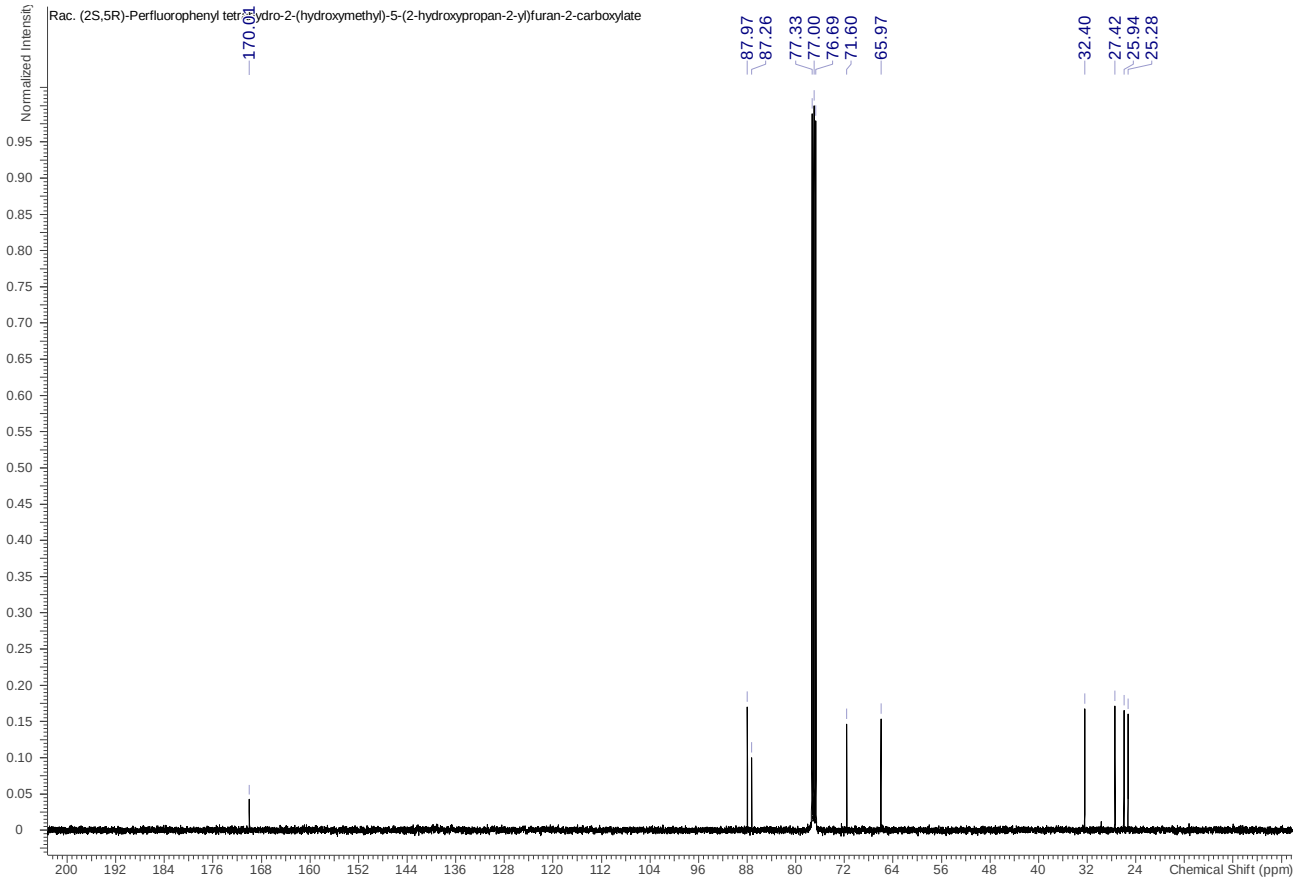




***Rac.* (2*S*,5*R*)-Perfluorophenyl tetrahydro-2-(hydroxymethyl)-5-(2-hydroxypropan-2-yl)furan-2-carboxylate (20)**

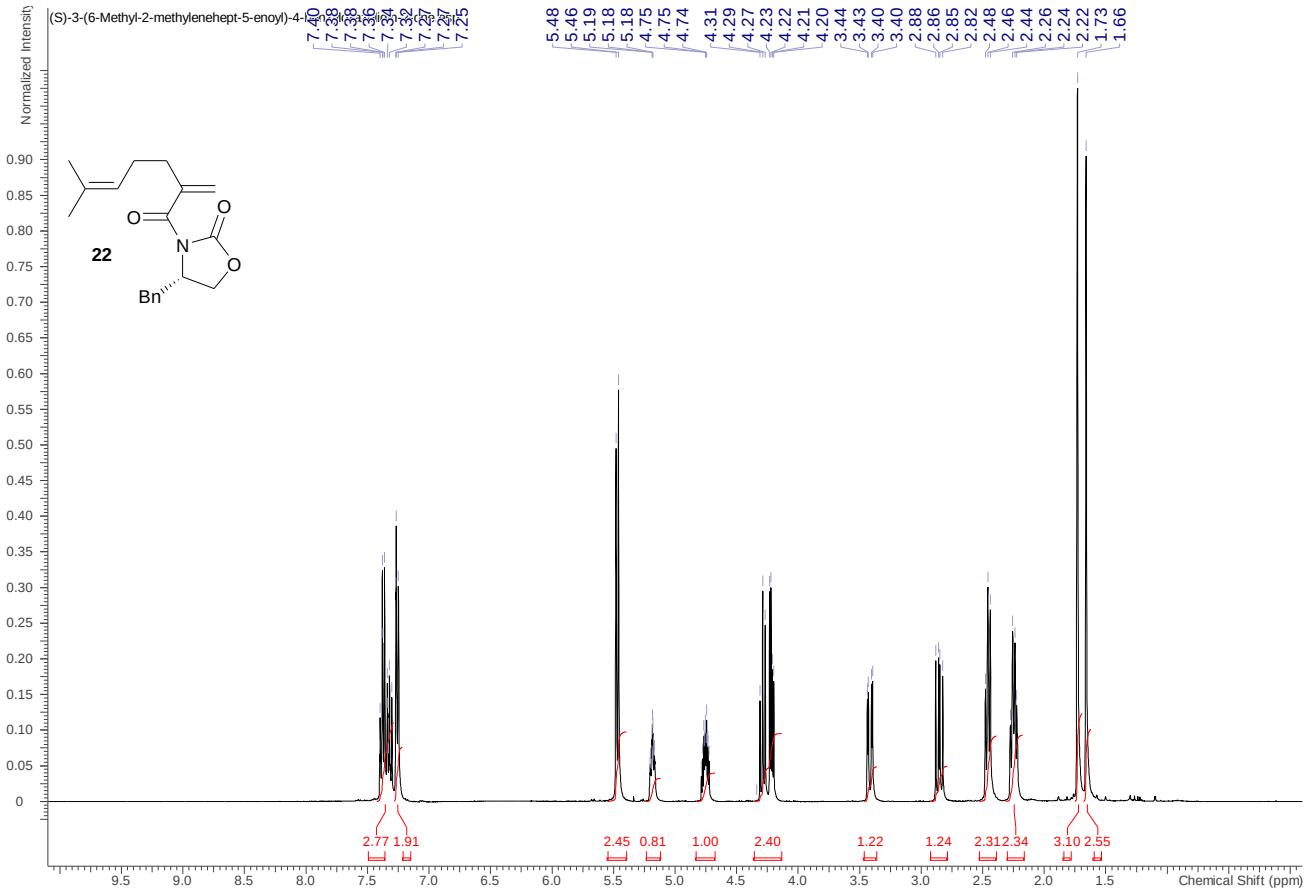
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$)

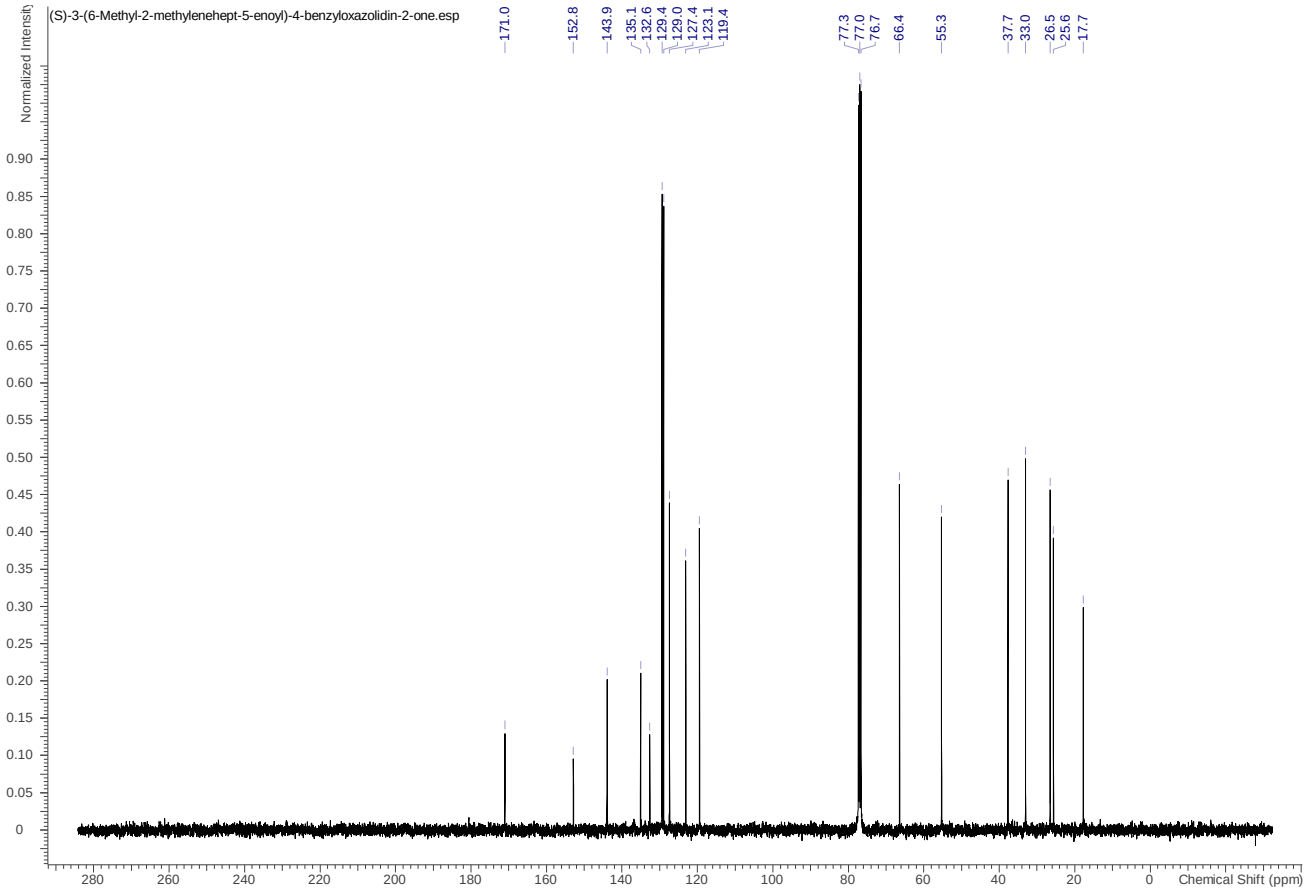




(*S*)-3-(6-Methyl-2-methylenehept-5-enoyl)-4-benzyloxazolidin-2-one (22)

^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$)

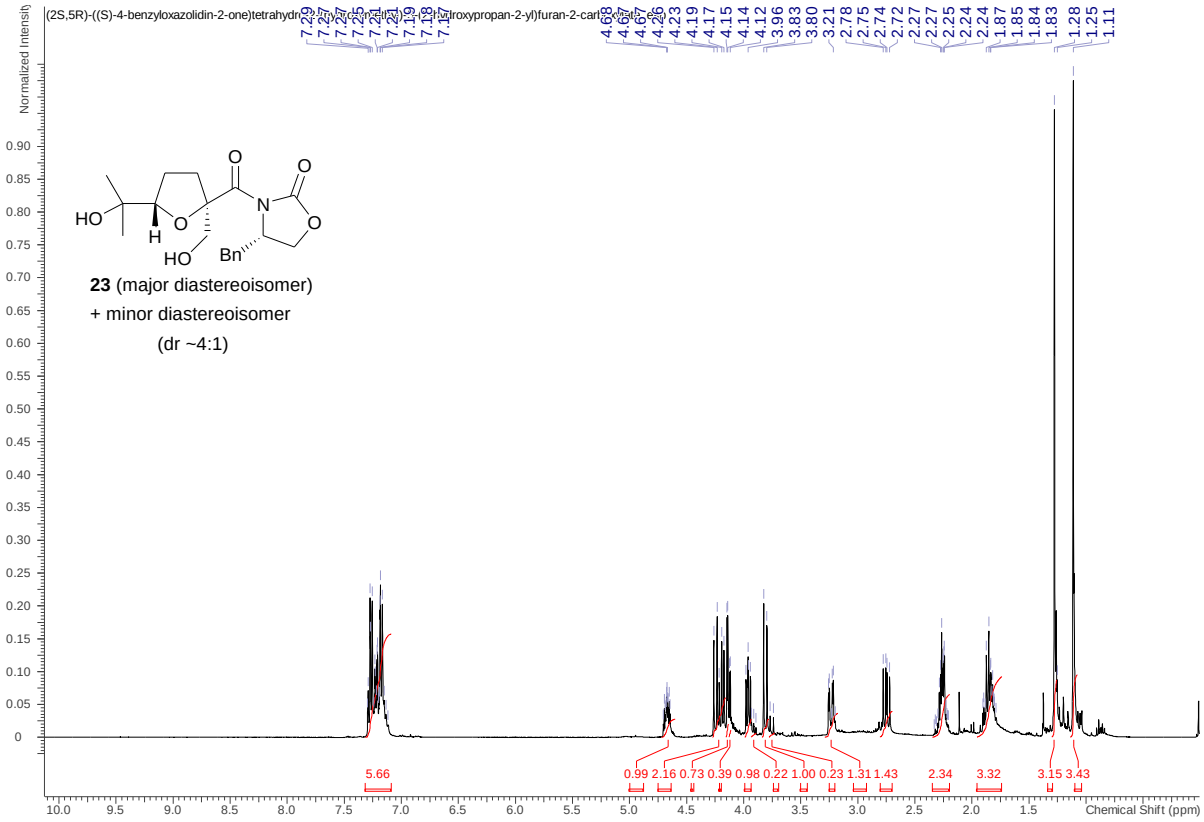


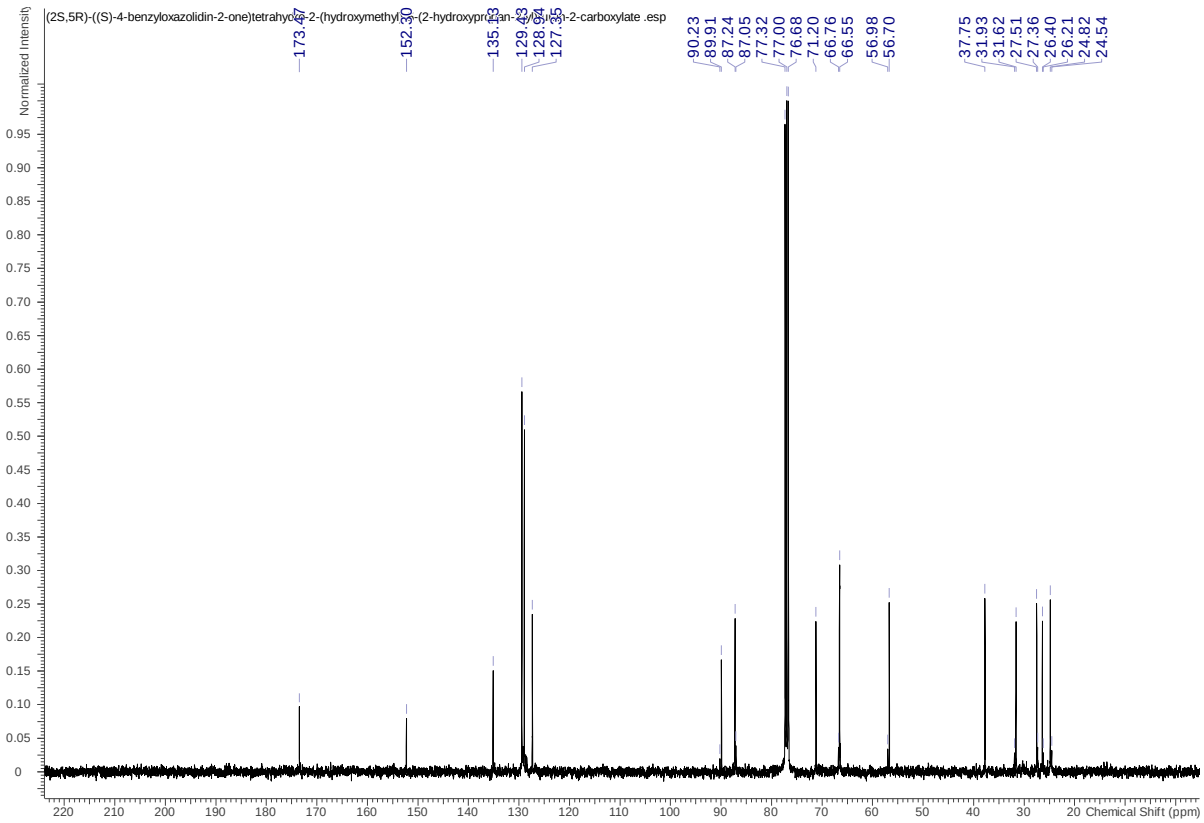


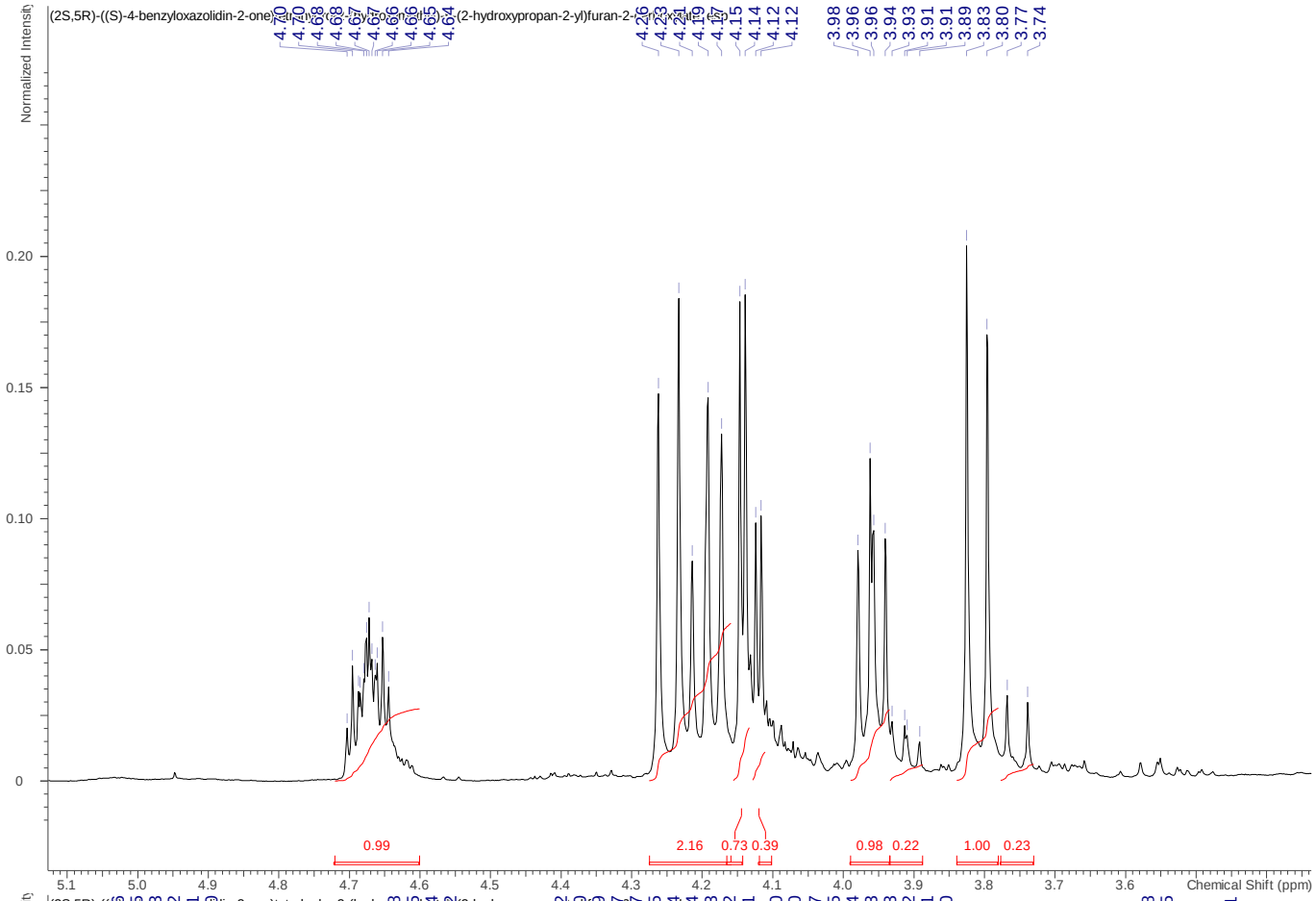
(2*R*,5*S*)-((*S*)-4-benzyloxazolidin-2-one)tetrahydro-2-(hydroxymethyl)-5-(2-hydroxypropan-2-yl)furan-2-carboxylate (23);

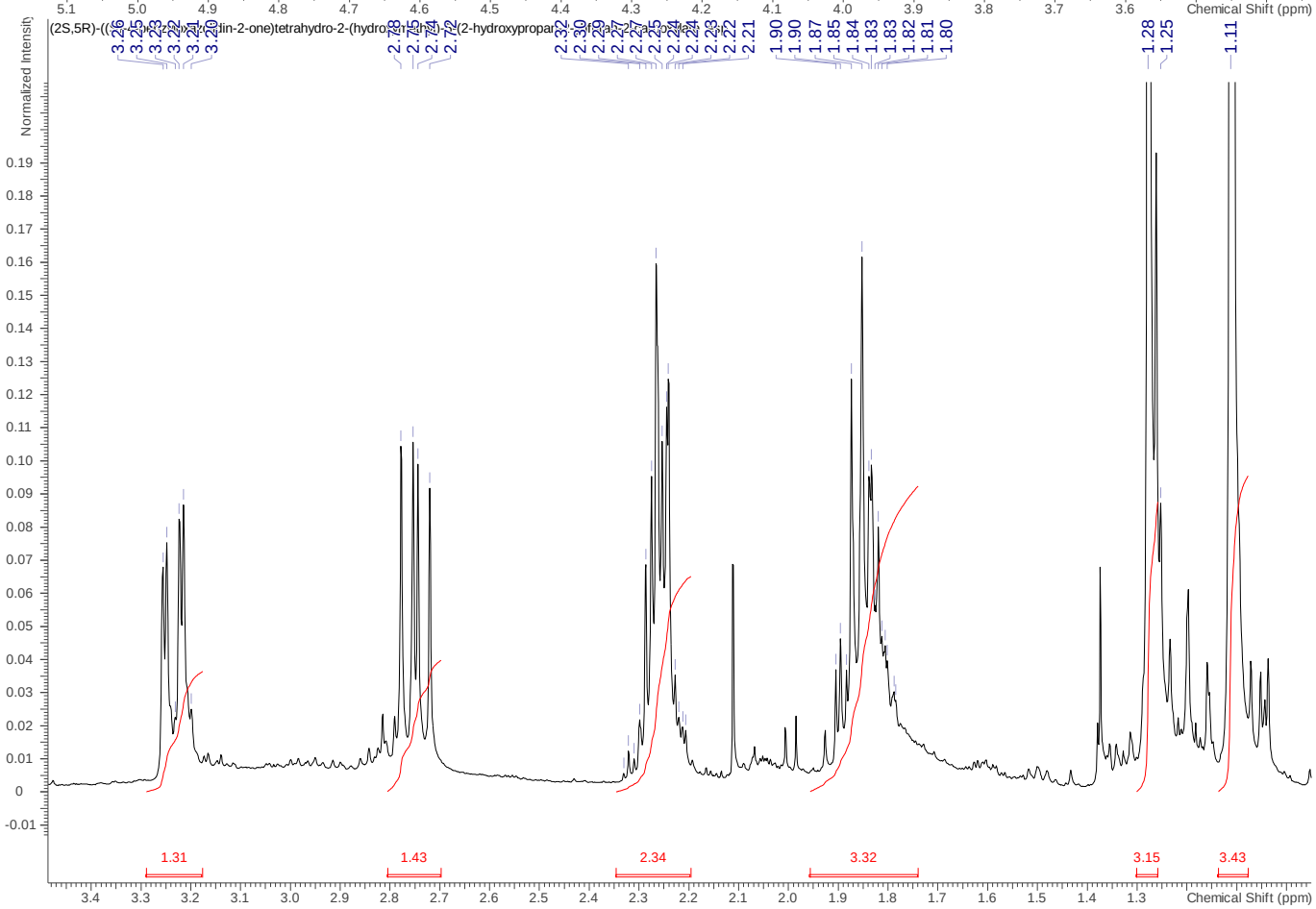
(2*S*,5*R*)-((*S*)-4-benzyloxazolidin-2-one)tetrahydro-2-(hydroxymethyl)-5-(2-hydroxypropan-2-yl)furan-2-carboxylate (minor diastereoisomer)

^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$)



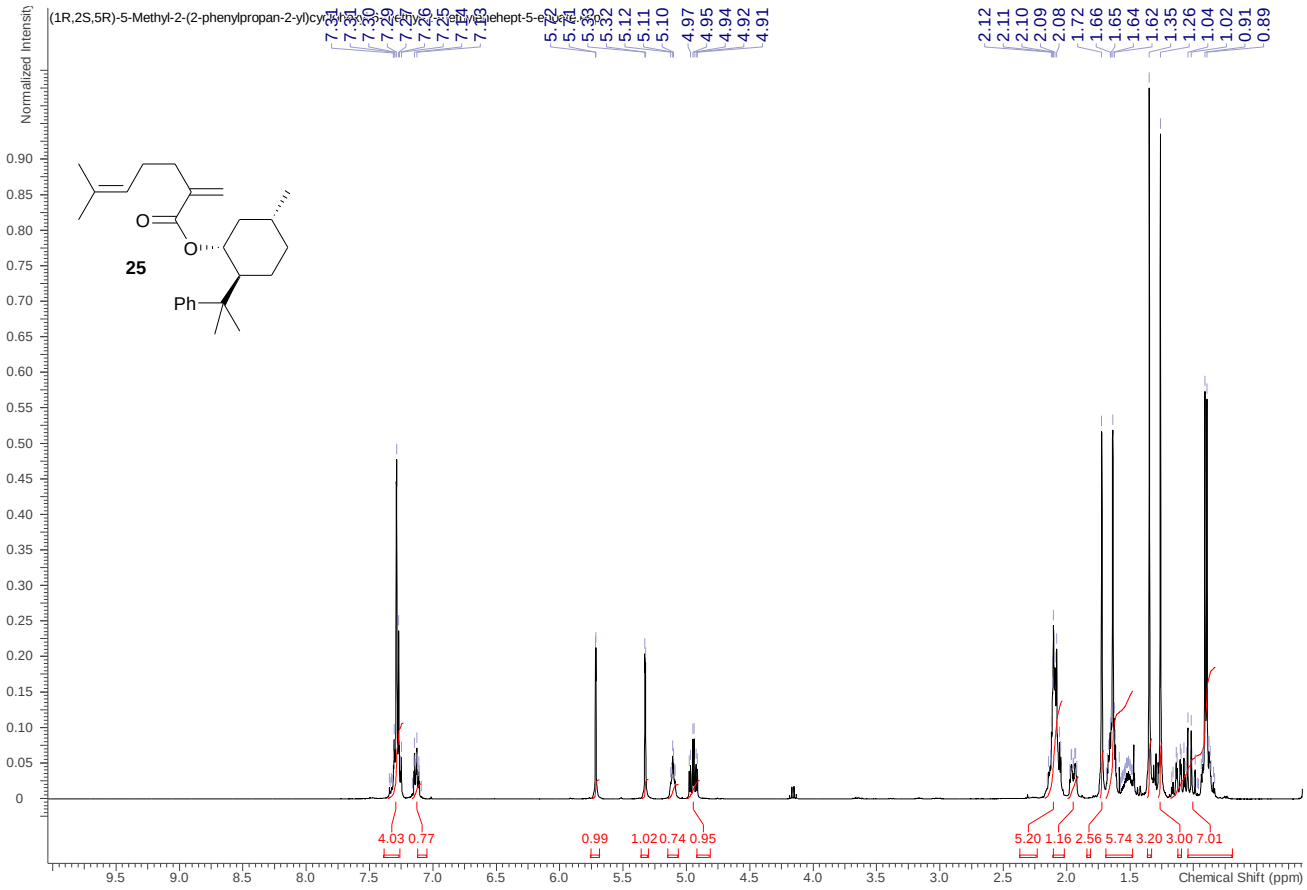


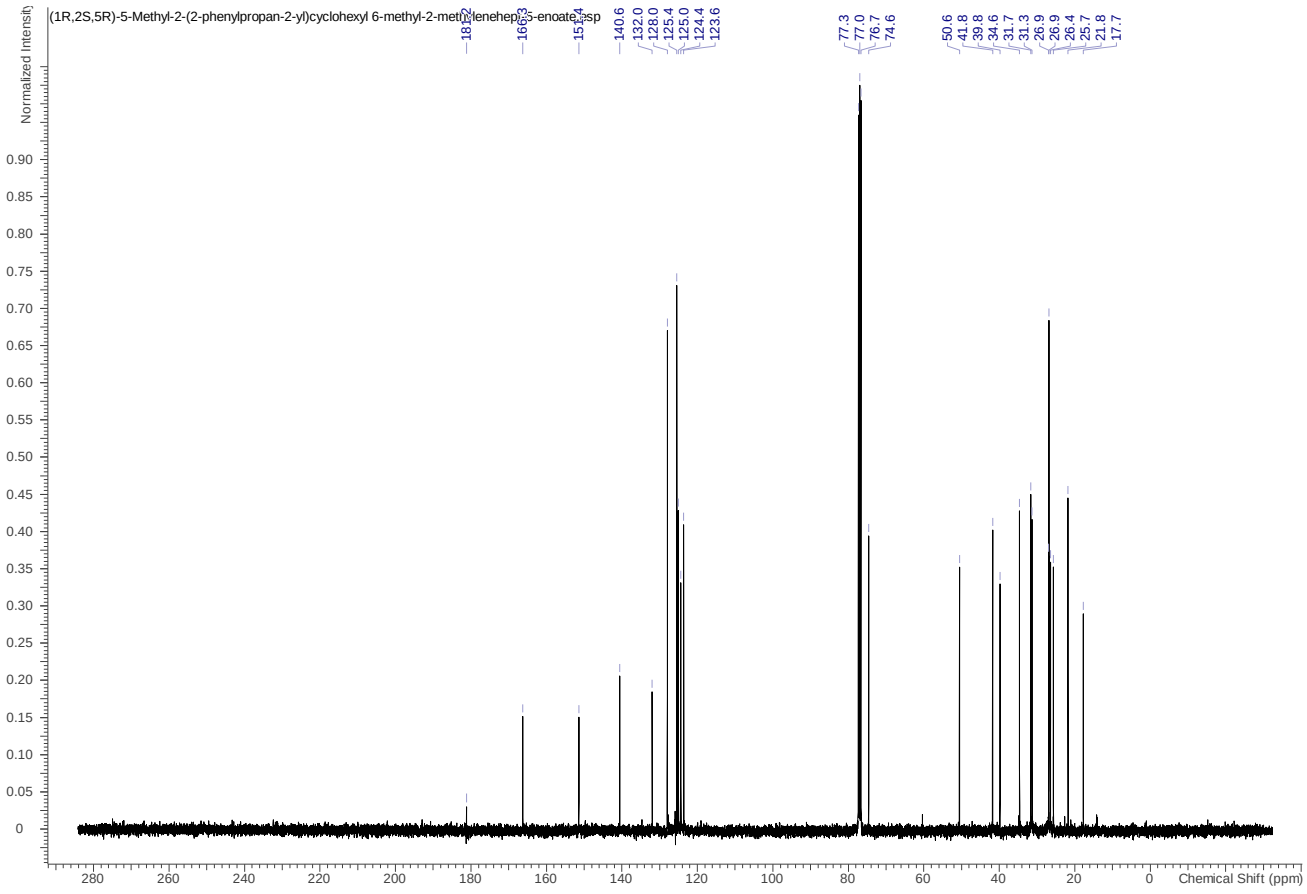

(2S,5R)-((S)-4-benzyloxazolidin-2-one)tetrahydro-2-(hydroxy(2-hydroxypropan-2-yl)furan-2-
4.70 4.70 4.68 4.68 4.67 4.67 4.67 4.66 4.66 4.65 4.65 4.64
4.26 4.23 4.21 4.19 4.17 4.15 4.14 4.12 4.12
3.98 3.96 3.96 3.94 3.93 3.91 3.91 3.89 3.83 3.80 3.77 3.74
Normalized Intensity
0.20
0.15
0.10
0.05
0
0.99
2.16
0.73
0.39
0.98
0.22
1.00
0.23
5.1 5.0 4.9 4.8 4.7 4.6 4.5 4.4 4.3 4.2 4.1 4.0 3.9 3.8 3.7 3.6
Chemical Shift (ppm)


(2S,5R)-((S)-4-benzyloxazolidin-2-one)tetrahydro-2-(hydroxy(2-hydroxypropan-2-yl)furan-2-
3.26 3.25 3.23 3.22 3.21 3.20
2.78 2.75 2.74 2.72
2.32 2.30 2.29 2.27 2.25 2.24 2.23 2.22 2.21
1.90 1.90 1.87 1.85 1.84 1.83 1.82 1.81 1.80
1.28 1.25
1.11
Normalized Intensity
0.19
0.18
0.17
0.16
0.15
0.14
0.13
0.12
0.11
0.10
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
-0.01
1.31
1.43
2.34
3.32
3.15
3.43
3.4 3.3 3.2 3.1 3.0 2.9 2.8 2.7 2.6 2.5 2.4 2.3 2.2 2.1 2.0 1.9 1.8 1.7 1.6 1.5 1.4 1.3
Chemical Shift (ppm)

(1*R*,2*S*,5*R*)-5-Methyl-2-(2-phenylpropan-2-yl)cyclohexyl 6-methyl-2-methylenehept-5-enoate (25)

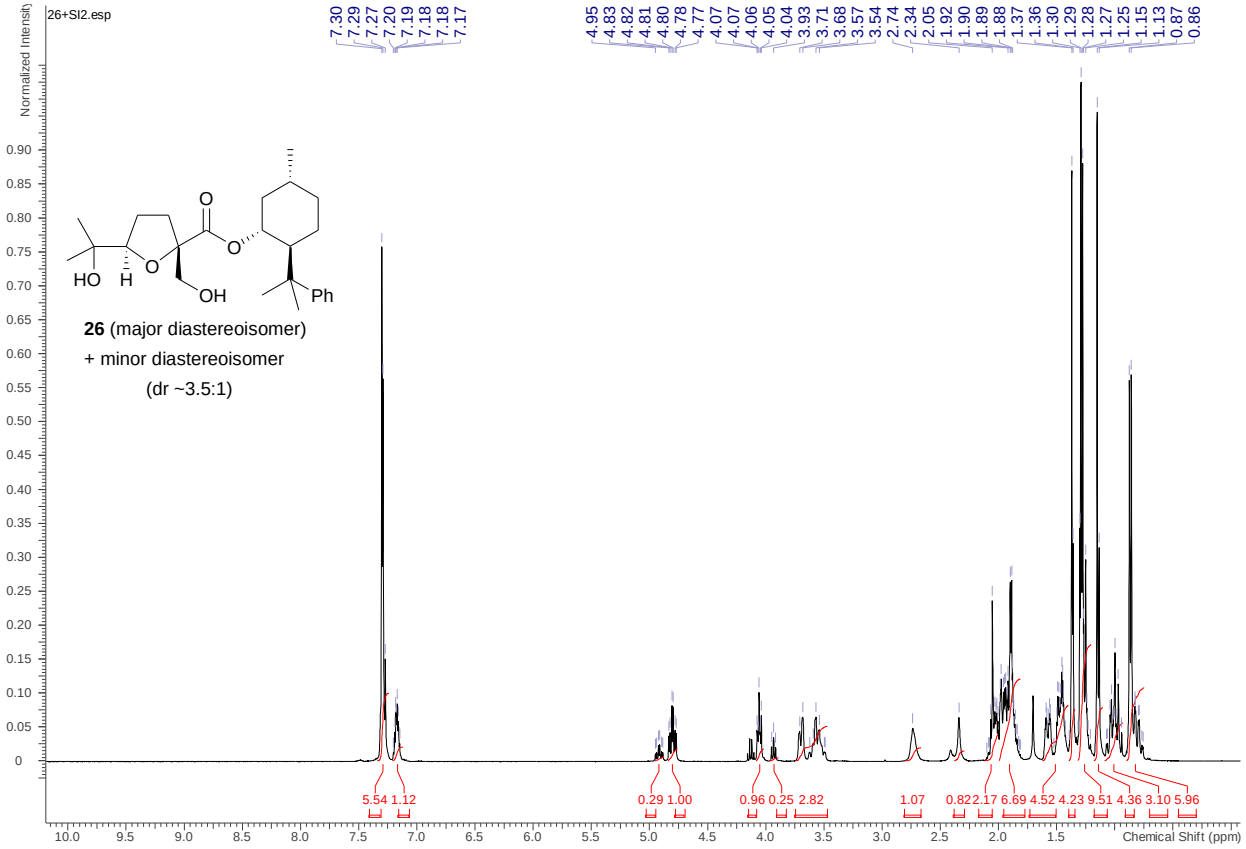
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$)

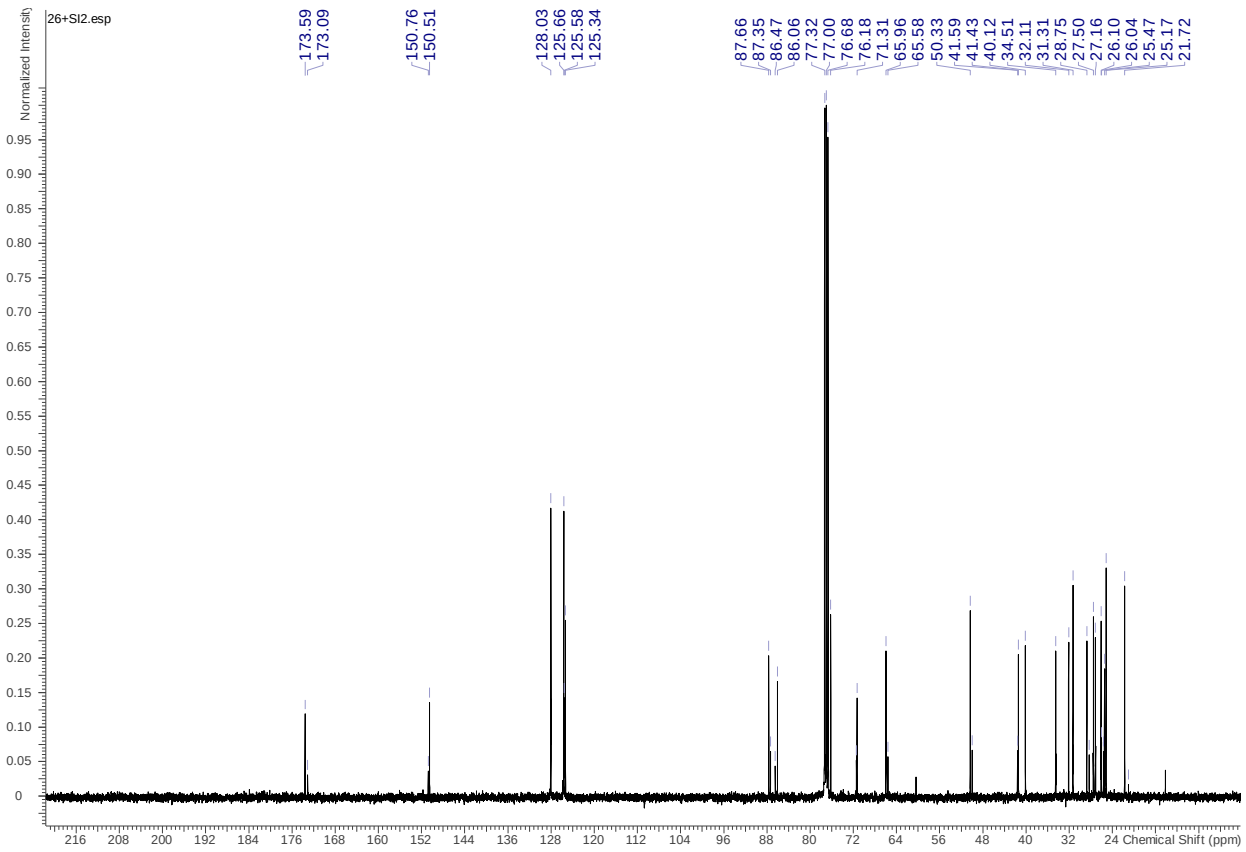


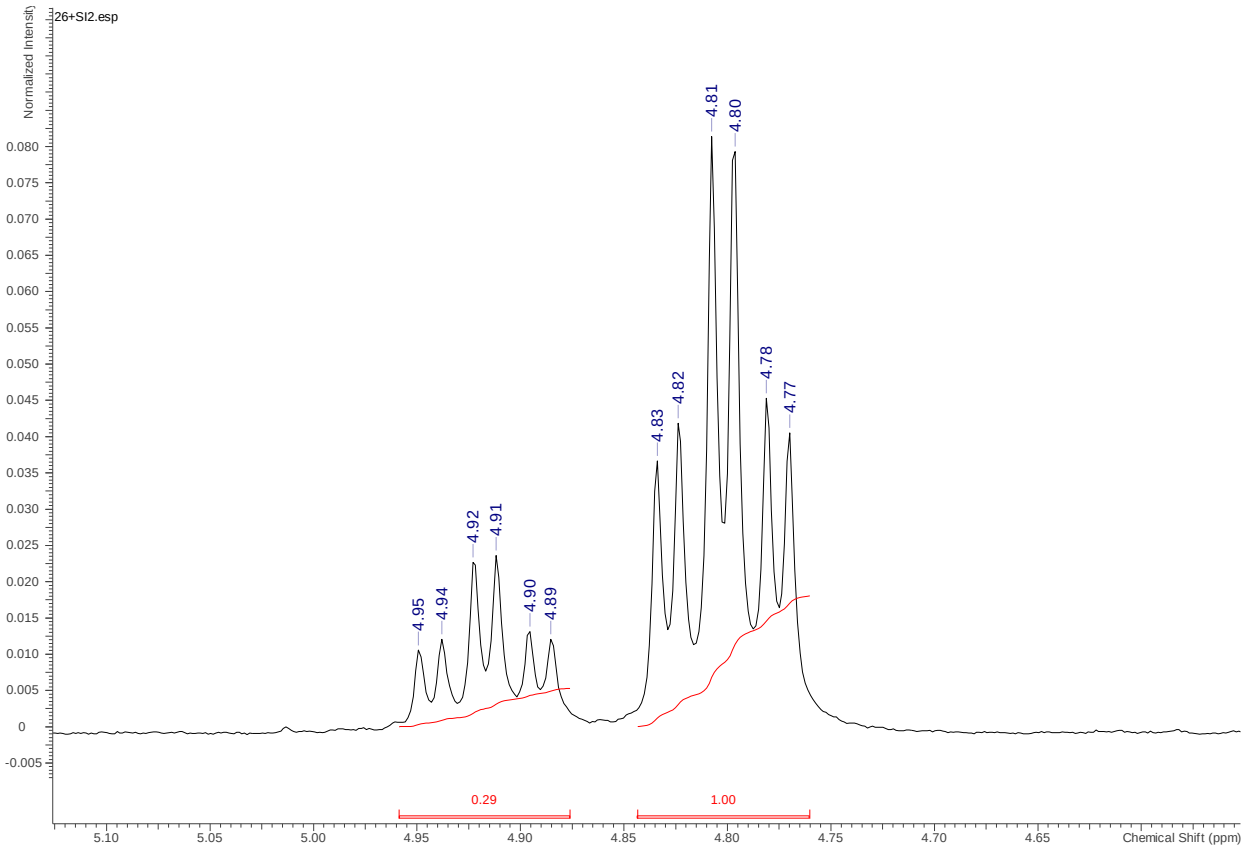


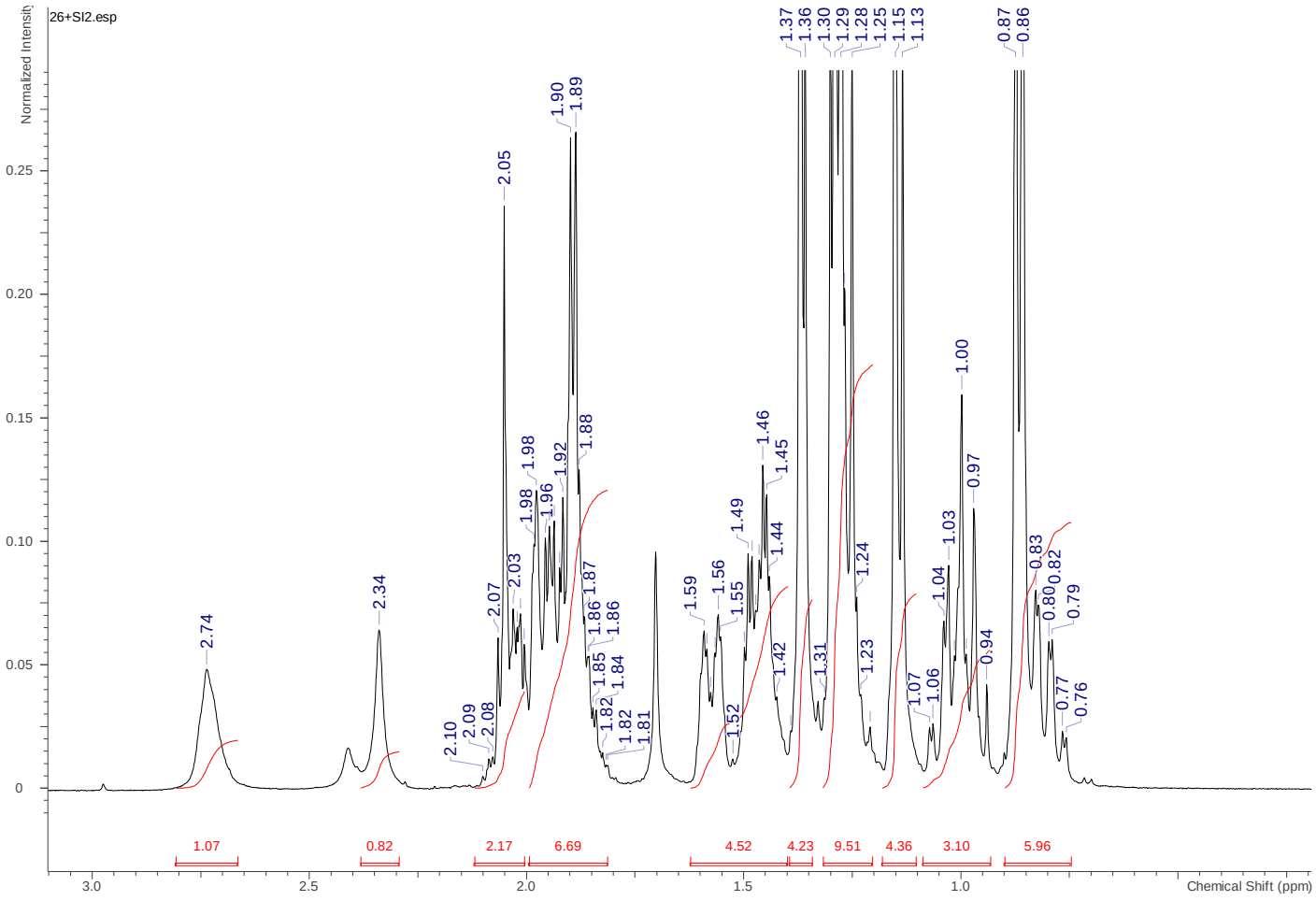
(2*S*,5*R*)-((1*R*,2*S*,5*R*)-5-Methyl-2-(2-phenylpropan-2-yl)cyclohexyl) tetrahydro-2-(hydroxymethyl)-5-(2-hydroxypropan-2-yl)furan-2-carboxylate (26);
(2*R*,5*S*)-((1*R*,2*S*,5*R*)-5-Methyl-2-(2-phenylpropan-2-yl)cyclohexyl) tetrahydro-2-(hydroxymethyl)-5-(2-hydroxypropan-2-yl)furan-2-carboxylate (minor diastereoisomer)

^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$)



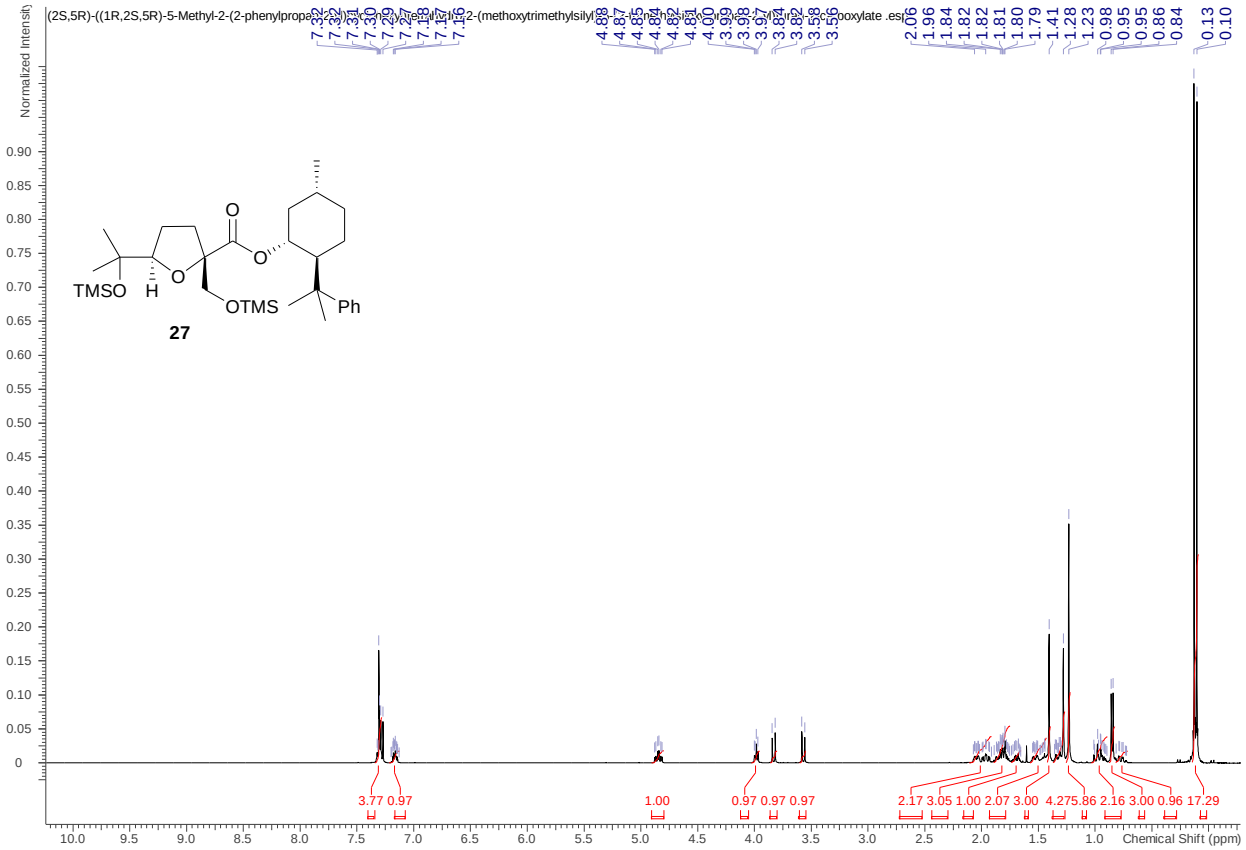


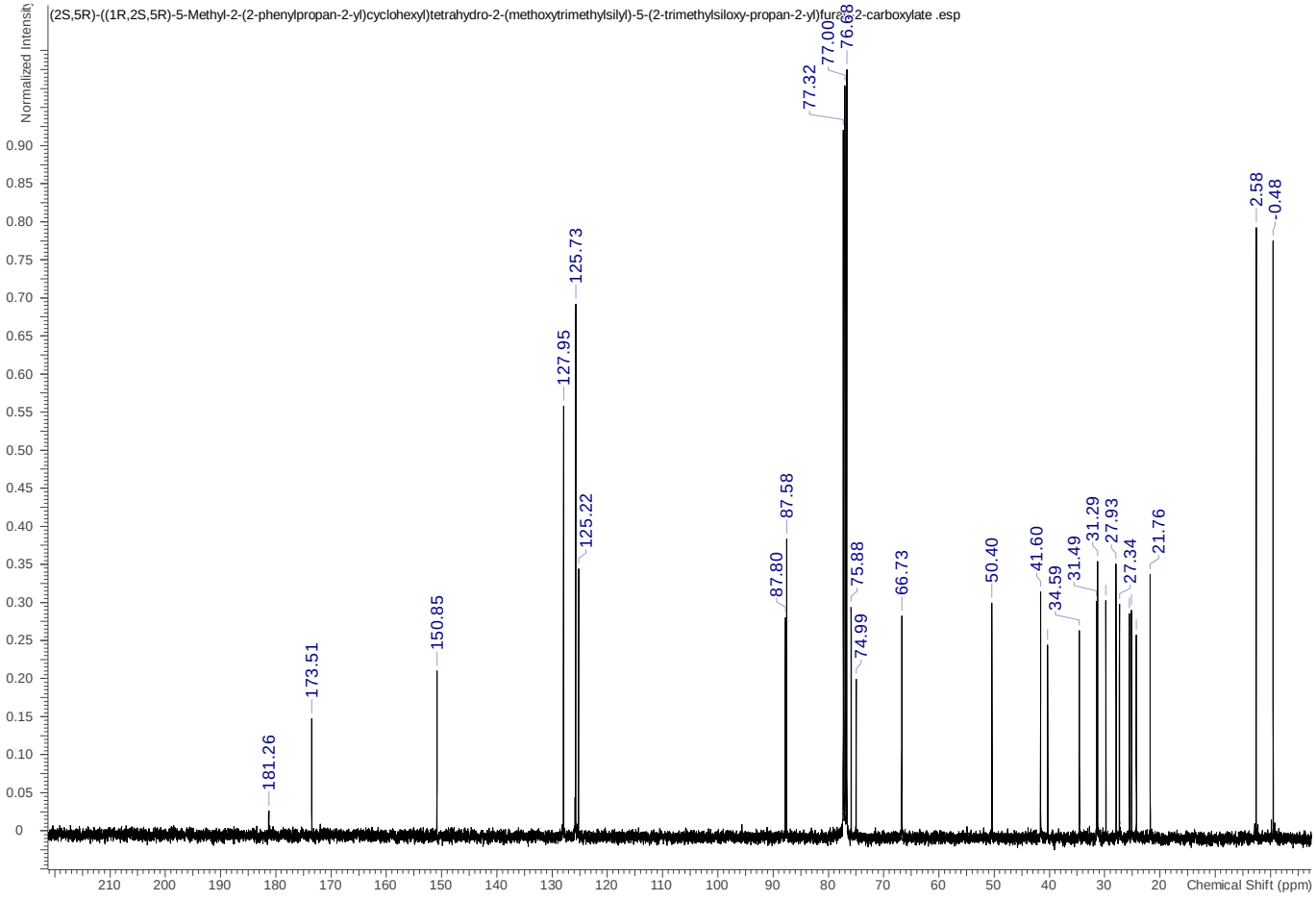

26+SI2.esp
Normalized Intensity
4.95
4.94
4.92
4.91
4.90
4.89
4.83
4.82
4.81
4.80
4.78
4.77
0.29
1.00
Chemical Shift (ppm)

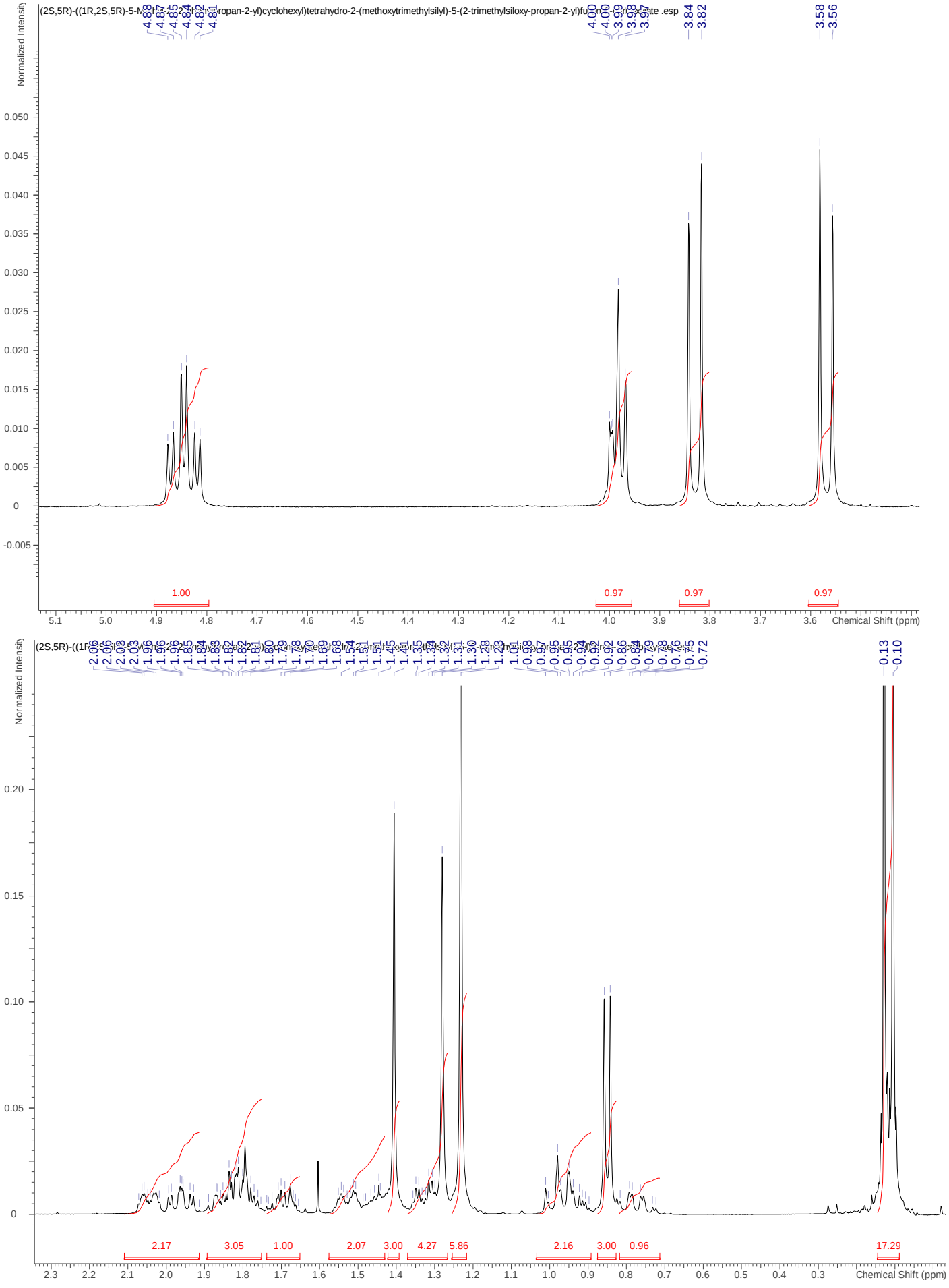

26+SI2.esp
Normalized Intensity
2.74
2.34
2.10
2.09
2.08
2.07
2.05
2.03
1.98
1.98
1.96
1.92
1.90
1.89
1.88
1.87
1.86
1.86
1.85
1.84
1.82
1.82
1.81
1.59
1.56
1.55
1.52
1.49
1.46
1.45
1.44
1.42
1.37
1.36
1.31
1.30
1.29
1.28
1.25
1.24
1.23
1.15
1.13
1.07
1.06
1.04
1.03
1.00
0.97
0.94
0.87
0.86
0.83
0.82
0.80
0.79
0.77
0.76
1.07
0.82
2.17
6.69
4.52
4.23
9.51
4.36
3.10
5.96
Chemical Shift (ppm)

(2*S*,5*R*)-((1*R*,2*S*,5*R*)-5-Methyl-2-(2-phenylpropan-2-yl)cyclohexyl)tetrahydro-2-(methoxytrimethylsilyl)-5-(2-trimethylsiloxy-propan-2-yl)furan-2-carboxylate (27)

^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$)

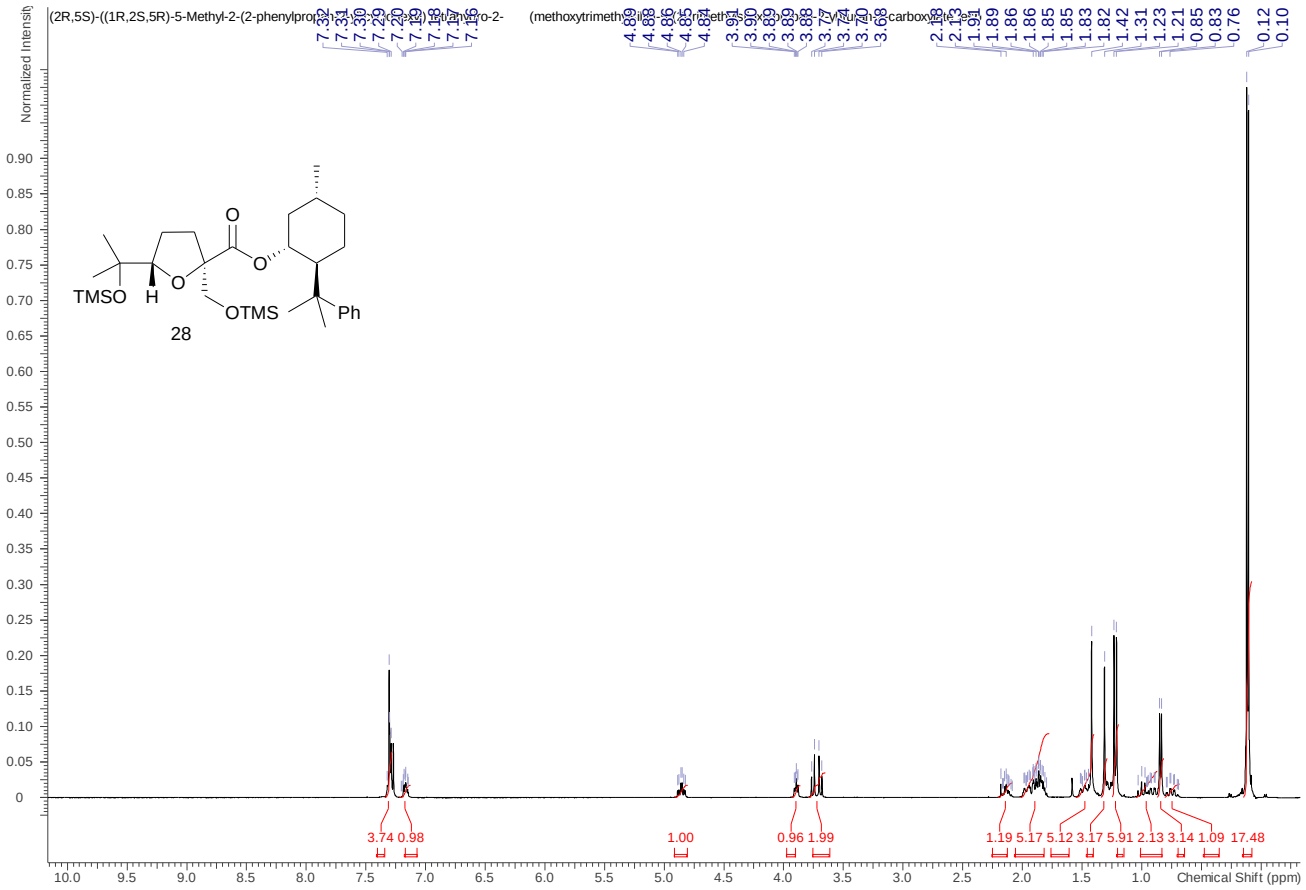


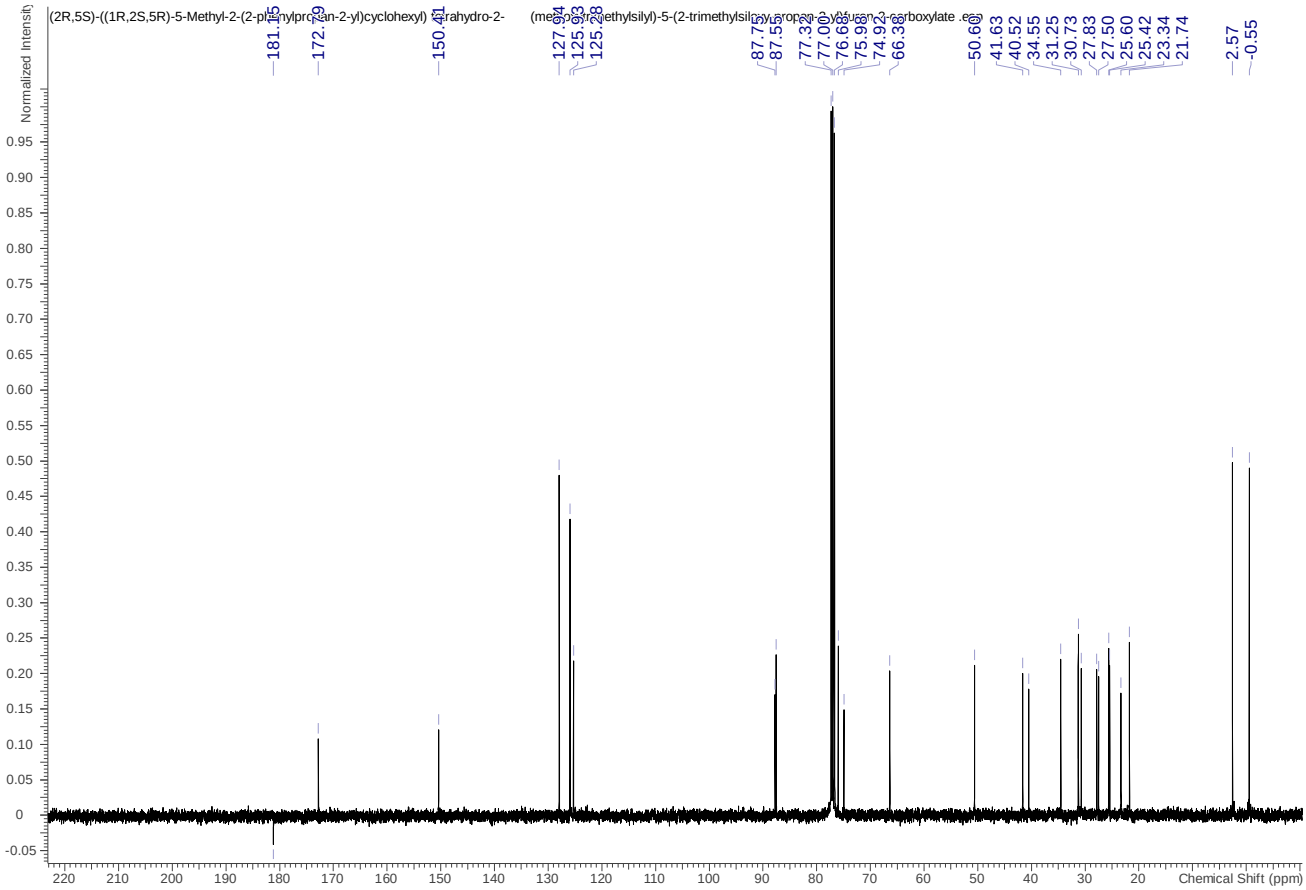


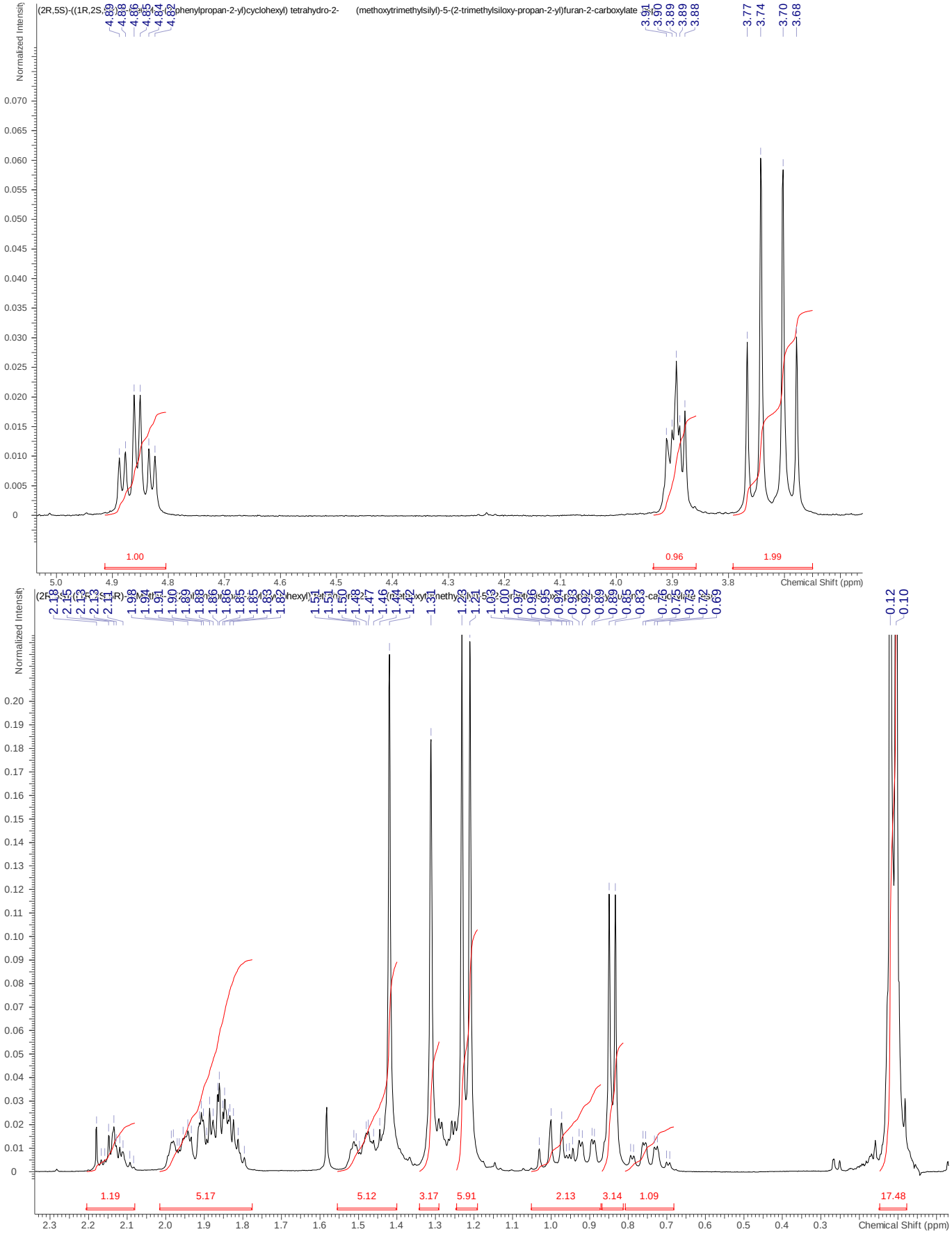

Normalized Intensity
Chemical Shift (ppm)
1.00
0.97
0.97
0.97
2.17
3.05
1.00
2.07
3.00
4.27
5.86
2.16
3.00
0.96
17.29

(2*R*,5*S*)-((1*R*,2*S*,5*R*)-5-Methyl-2-(2-phenylpropan-2-yl)cyclohexyl) tetrahydro-2-(methoxytrimethylsilyl)-5-(2-trimethylsiloxy-propan-2-yl)furan-2-carboxylate (28)

^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$)

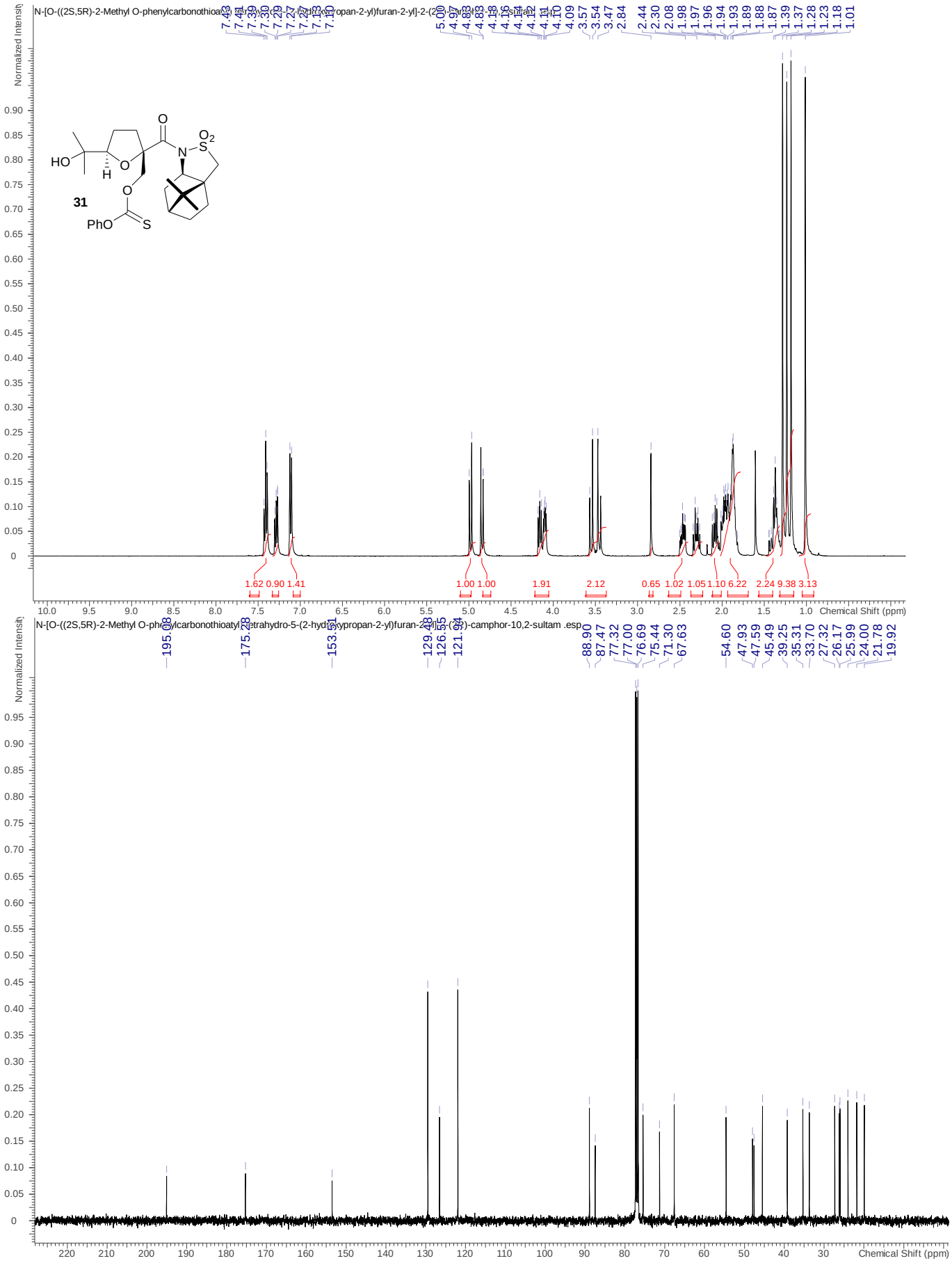





(2R,5S)-((1R,2S,5R)-2-isopropyl-5-methyl-2-(2-phenylpropan-2-yl)cyclohexyl) tetrahydro-2-(methoxytrimethylsilyl)-5-(2-trimethylsiloxy-propan-2-yl)furan-2-carboxylate
Normalized Intensity
Chemical Shift (ppm)
4.89
4.88
4.86
4.85
4.84
4.82
3.91
3.90
3.89
3.89
3.88
3.77
3.74
3.70
3.68
1.00
0.96
1.99
2.18
2.15
2.13
2.13
2.11
1.98
1.94
1.91
1.90
1.89
1.88
1.86
1.86
1.85
1.85
1.83
1.82
1.51
1.51
1.50
1.48
1.47
1.46
1.44
1.42
1.31
1.23
1.21
1.03
1.00
0.97
0.96
0.95
0.94
0.93
0.92
0.89
0.89
0.85
0.83
0.76
0.75
0.73
0.72
0.69
0.12
0.10
1.19
5.17
5.12
3.17
5.91
2.13
3.14
1.09
17.48

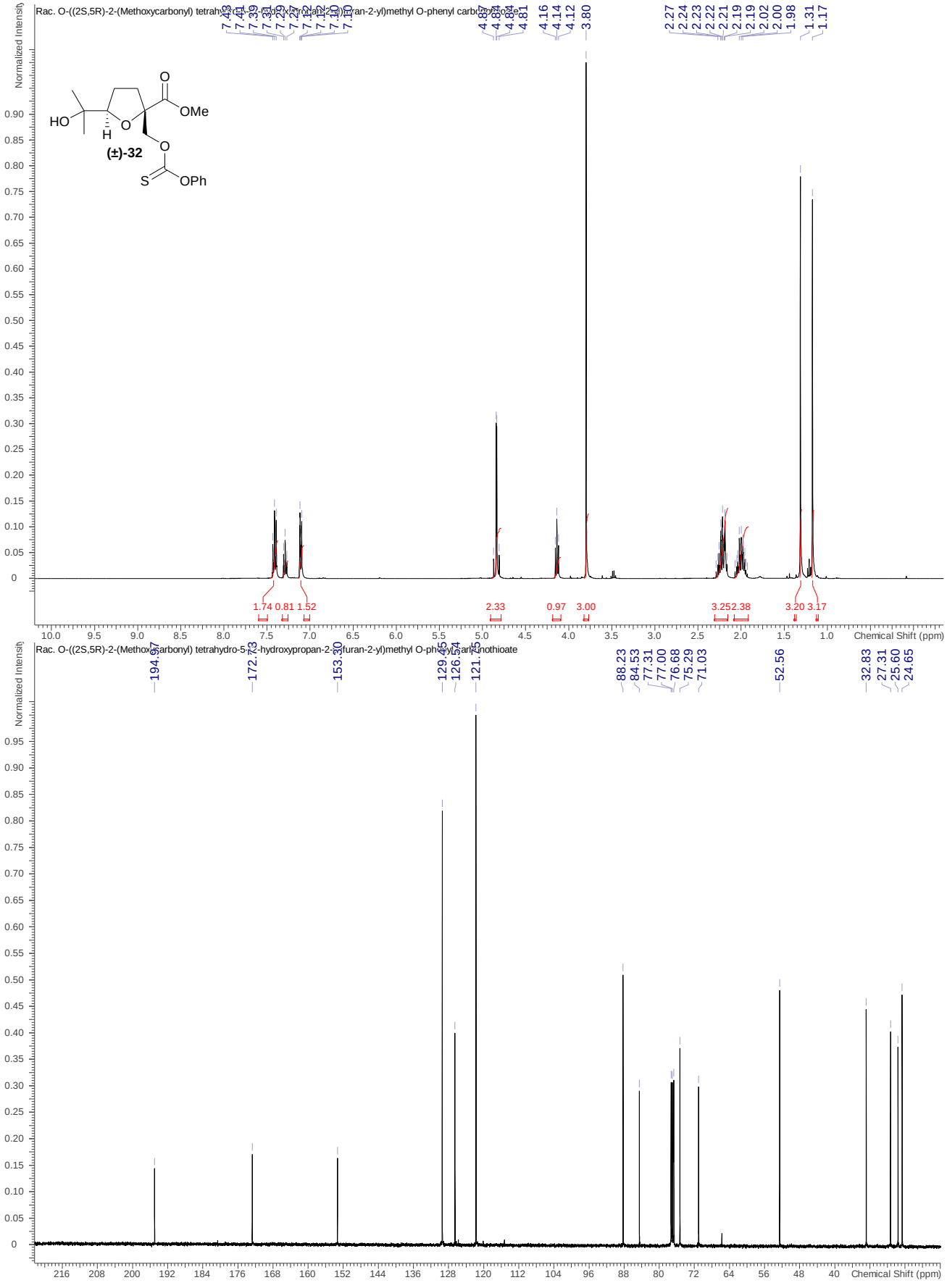
_N_-[_O_-((2_S_,5_R_)-2-Methyl _O_-phenylcarbonothioatyl) tetrahydro-5-(2-hydroxypropan-2-yl)furan-2-yl]-2-(2_R_)-camphor-10,2-sultam (31)

^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$)



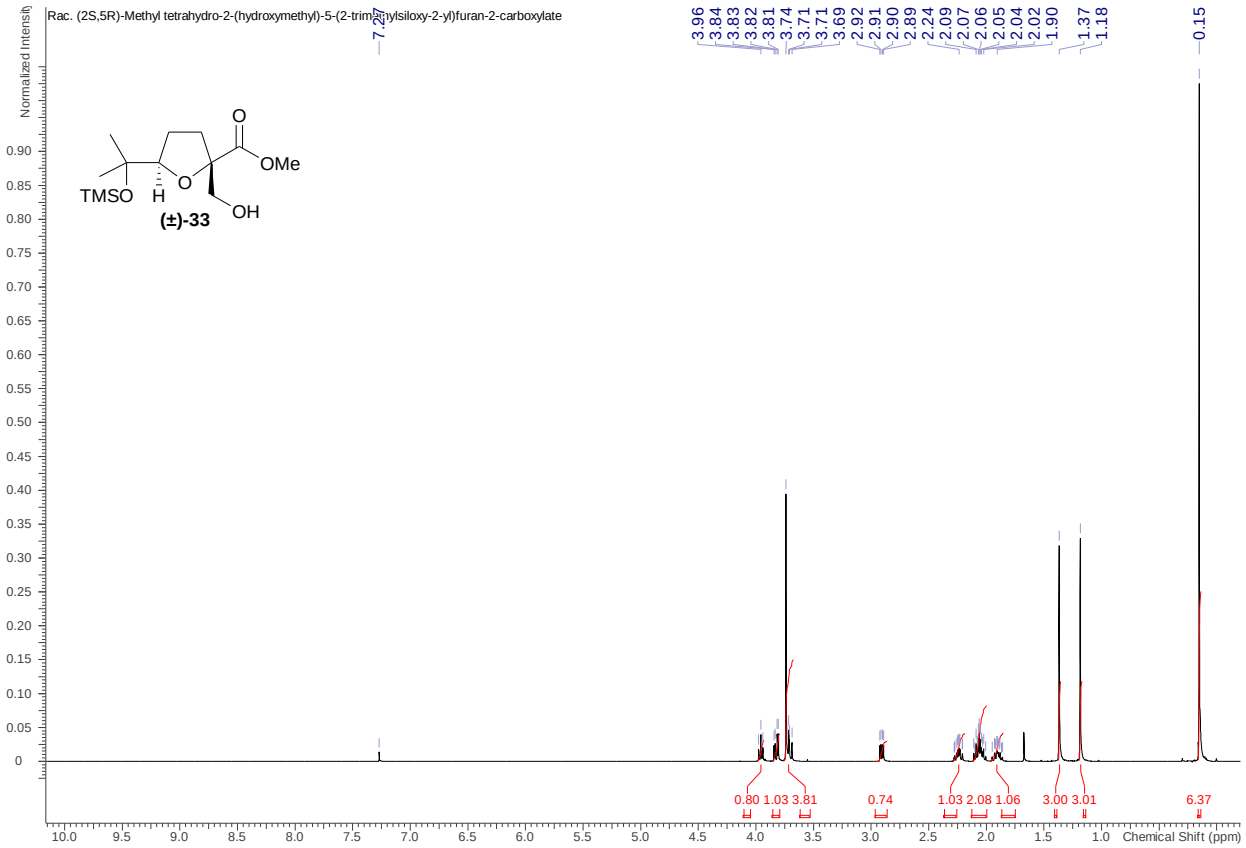
***Rac.* *O*-((2*S*,5*R*)-2-(Methoxycarbonyl) tetrahydro-5-(2-hydroxypropan-2-yl)furan-2-yl)methyl *O*-phenyl carbonothioate (32)**

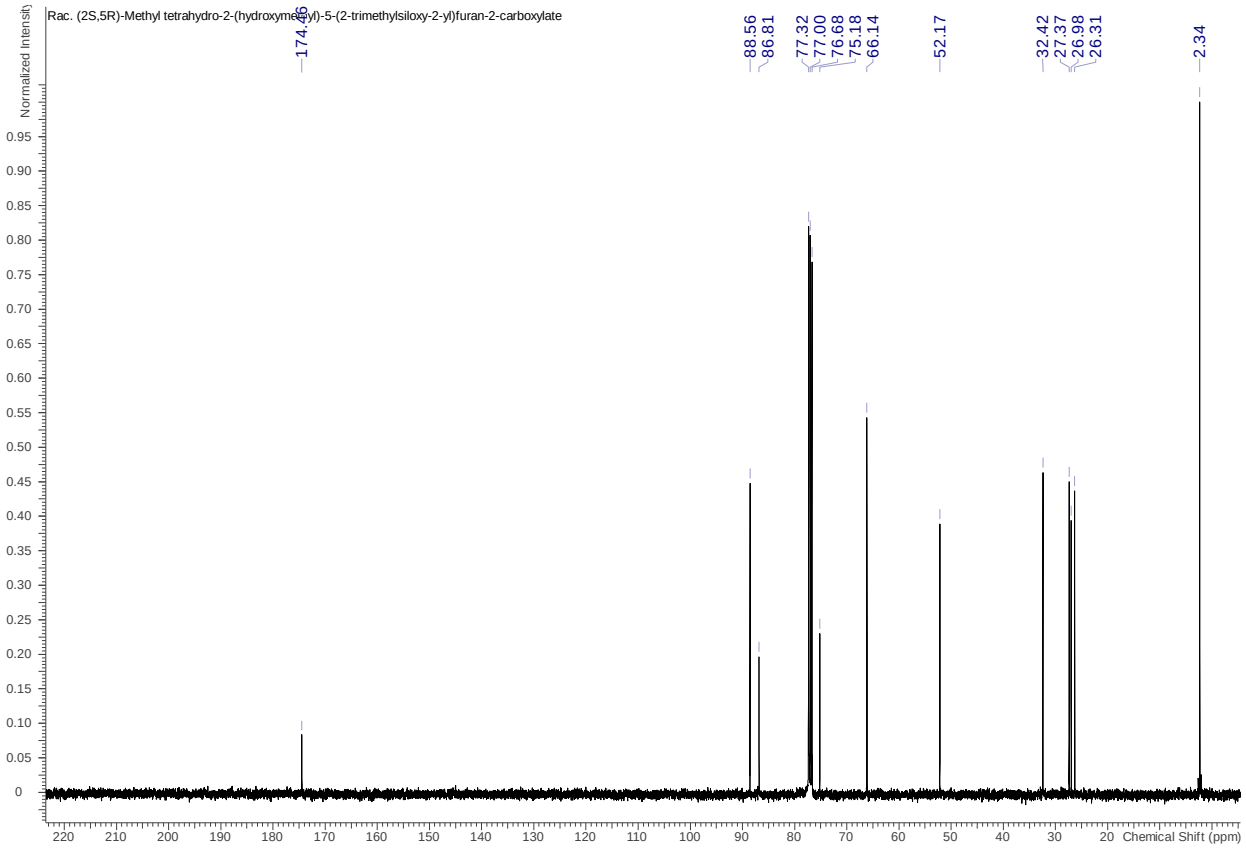
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$)

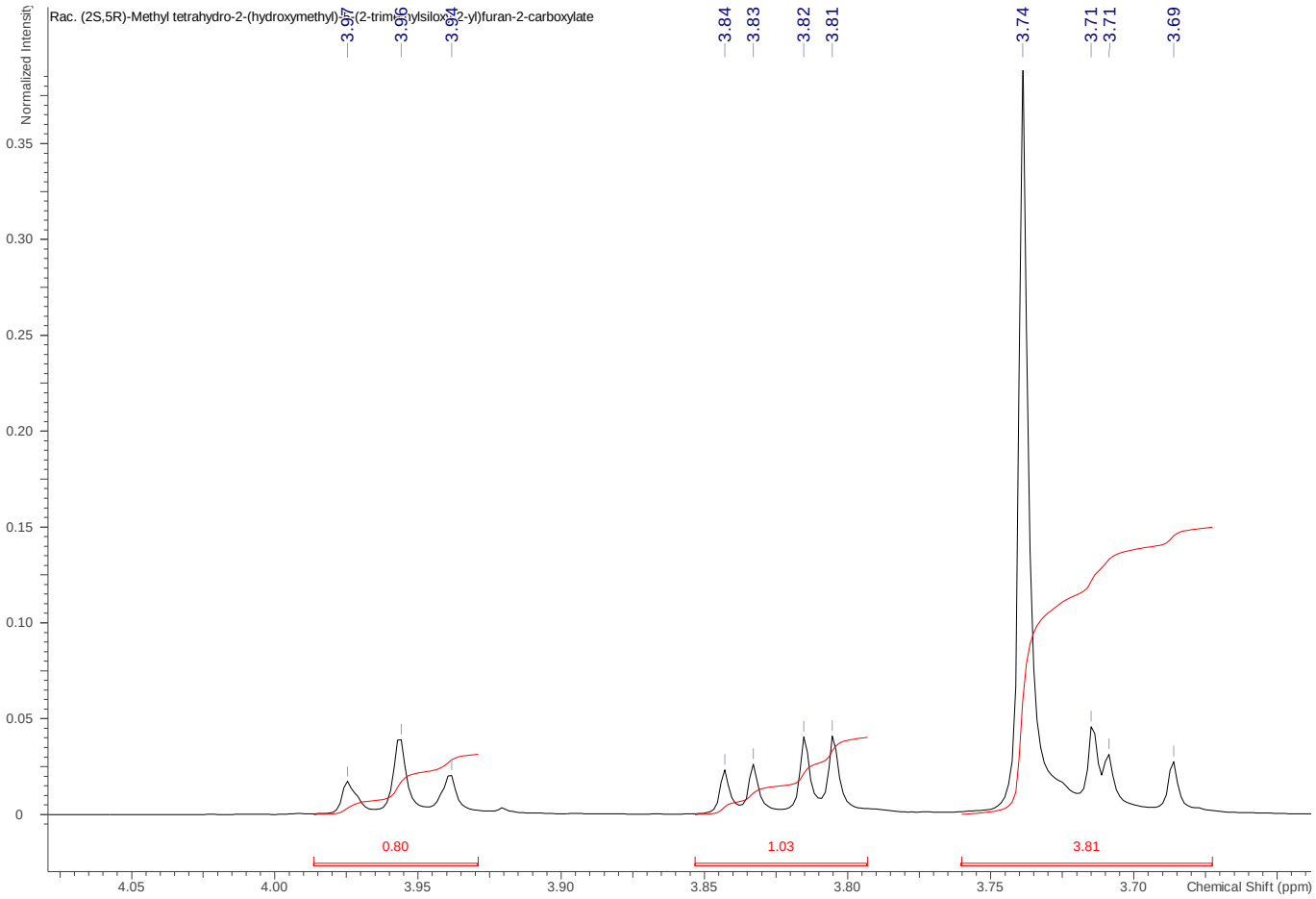


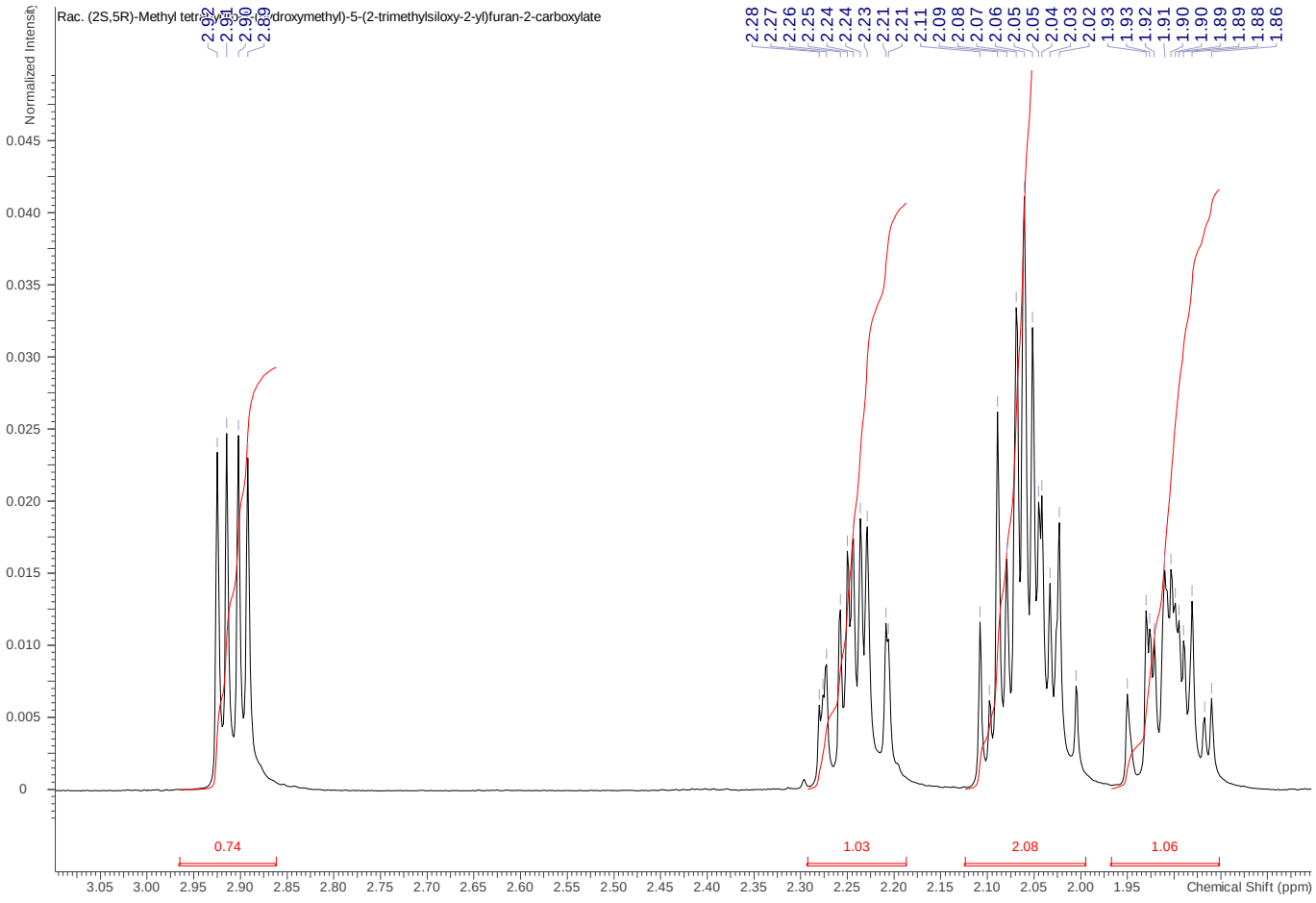
Rac. (2*S*,5*R*)-Methyl tetrahydro-2-(hydroxymethyl)-5-(2-trimethylsiloxy-2-yl)furan-2-carboxylate (33)

^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$)



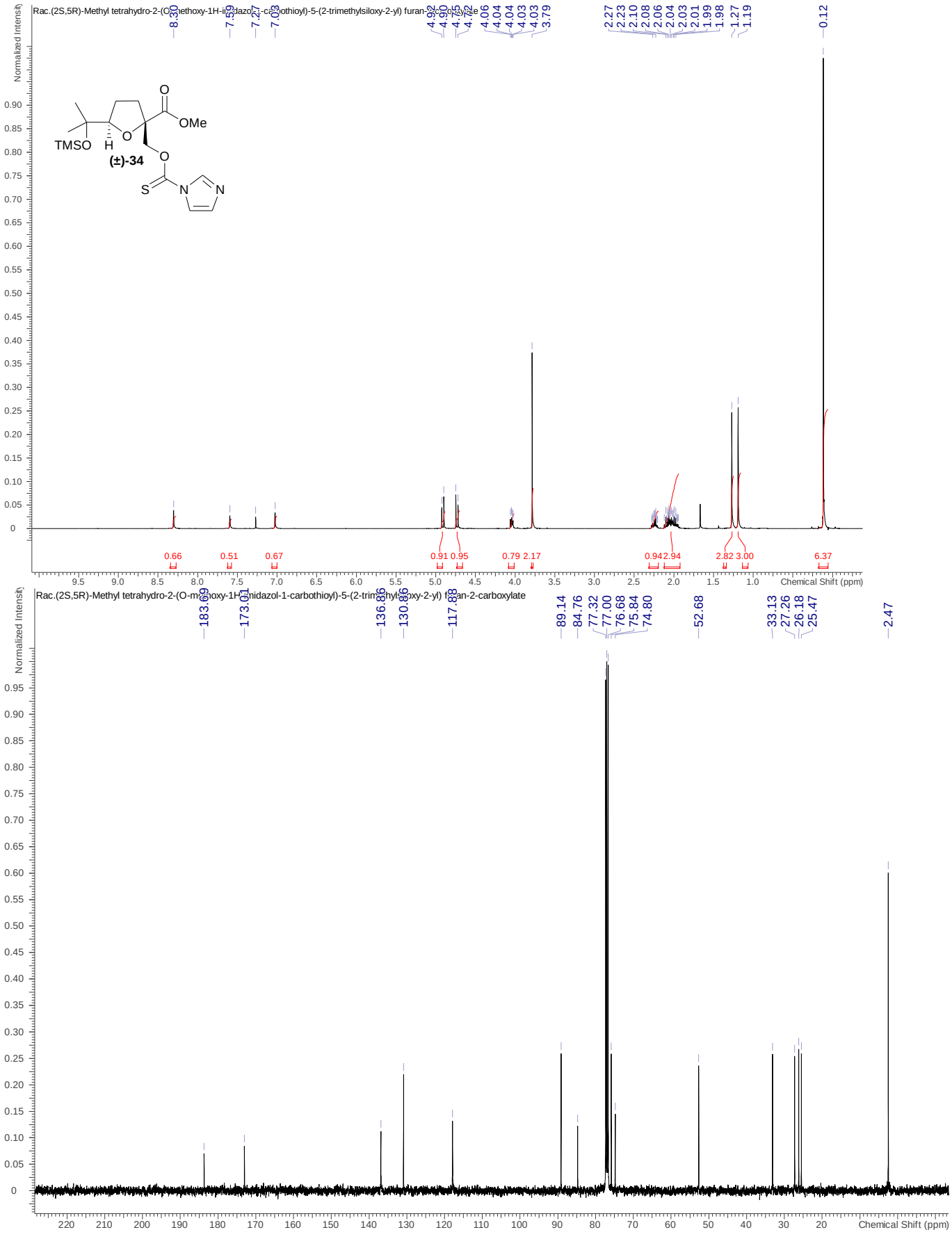


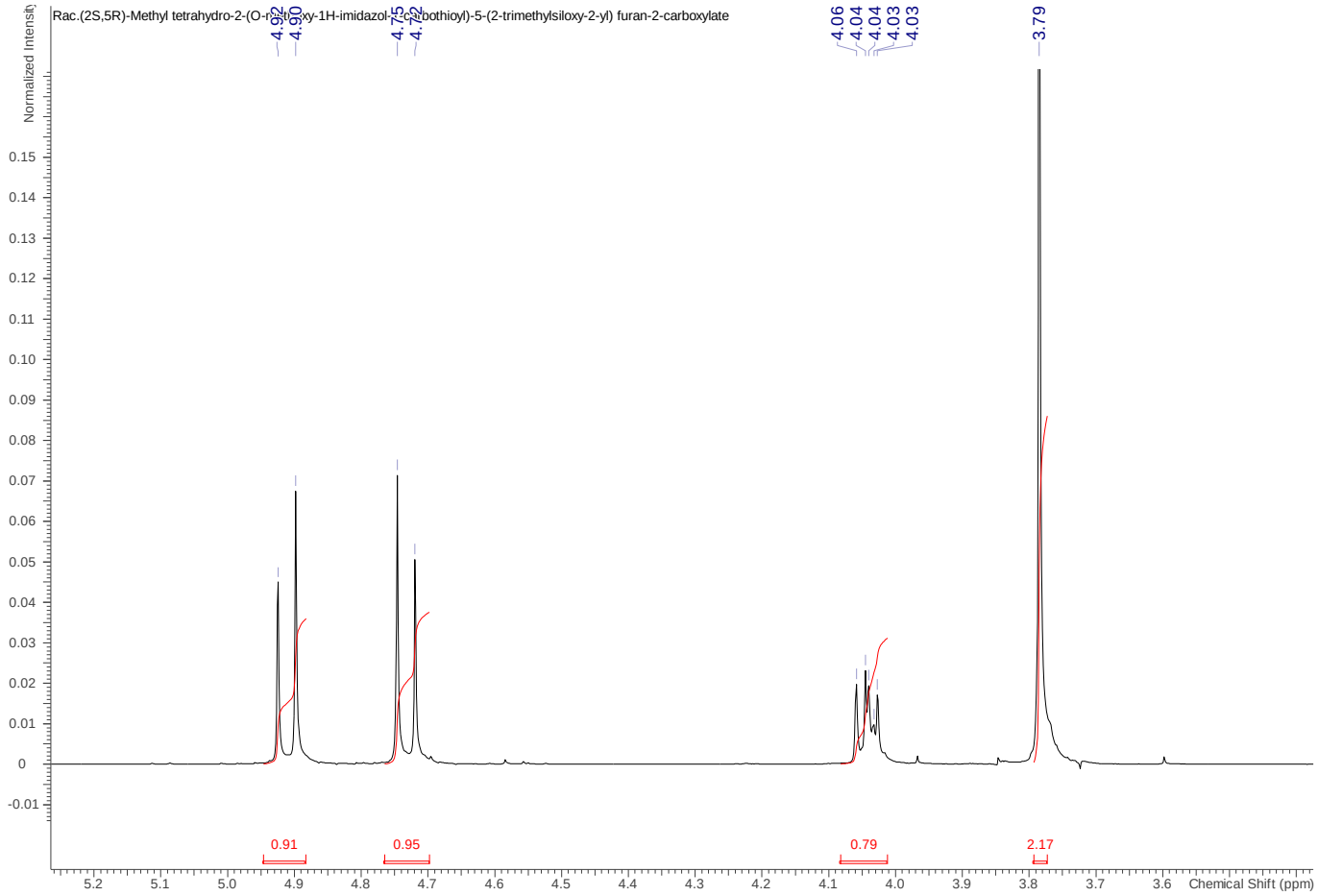

Rac. (2S,5R)-Methyl tetrahydro-2-(hydroxymethyl)-5-(2-trimethylsiloxy-2-yl)furan-2-carboxylate
Normalized Intensity
3.97
3.96
3.94
3.84
3.83
3.82
3.81
3.74
3.71
3.71
3.69
0.80
1.03
3.81
Chemical Shift (ppm)

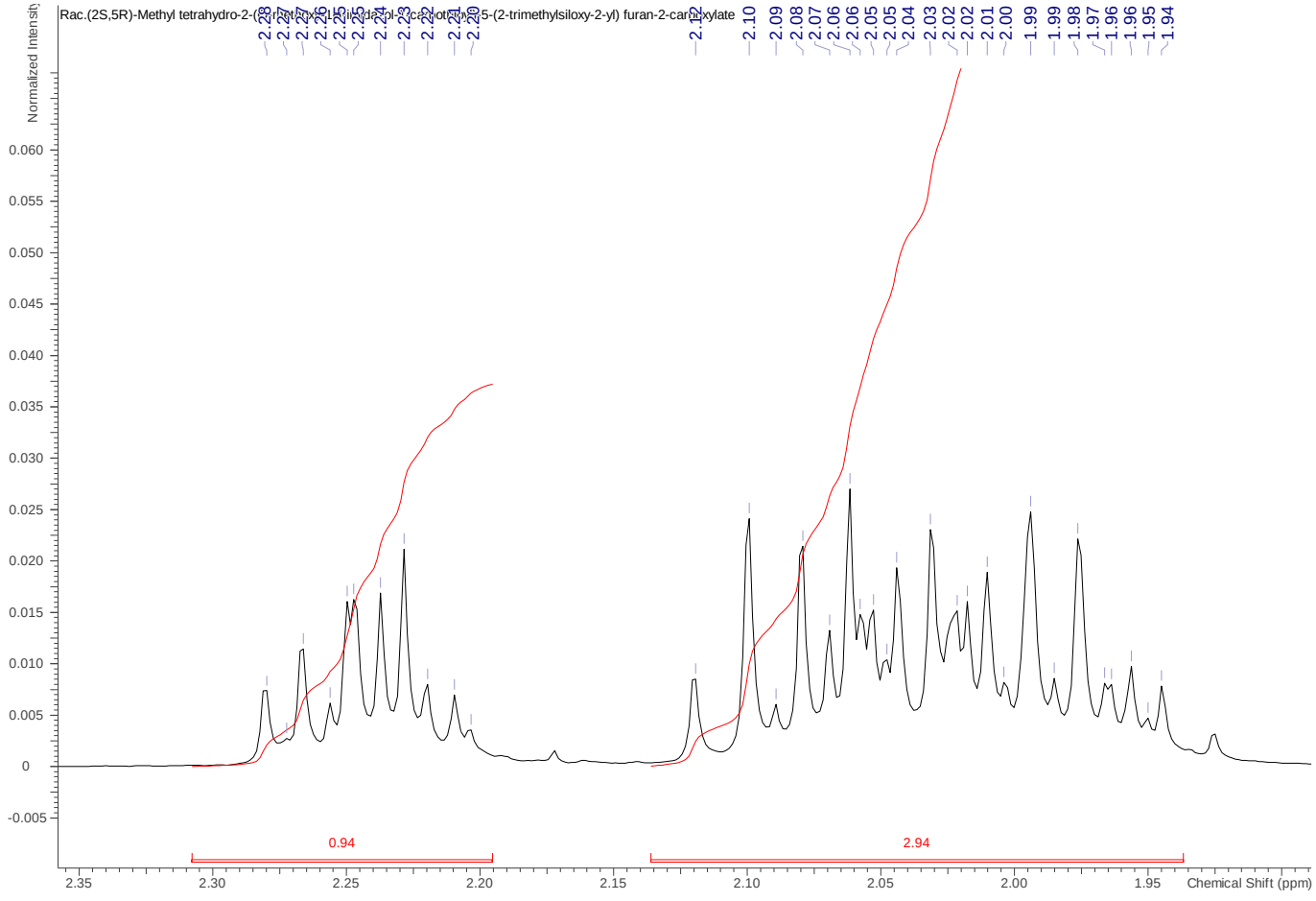

Rac. (2S,5R)-Methyl tetrahydro-2-(hydroxymethyl)-5-(2-trimethylsiloxy-2-yl)furan-2-carboxylate
Normalized Intensity
2.92
2.91
2.90
2.89
2.28
2.27
2.26
2.25
2.24
2.24
2.23
2.21
2.21
2.11
2.09
2.08
2.07
2.06
2.05
2.05
2.04
2.03
2.02
1.93
1.93
1.92
1.91
1.90
1.90
1.89
1.89
1.88
1.86
0.74
1.03
2.08
1.06
Chemical Shift (ppm)

Rac.(2_S_,5_R_)-Methyl tetrahydro-2-(_O_-methoxy-1_H_-imidazol-1-carbothioyl)-5-(2-trimethylsiloxy-2-yl) furan-2-carboxylate (34)

^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$)

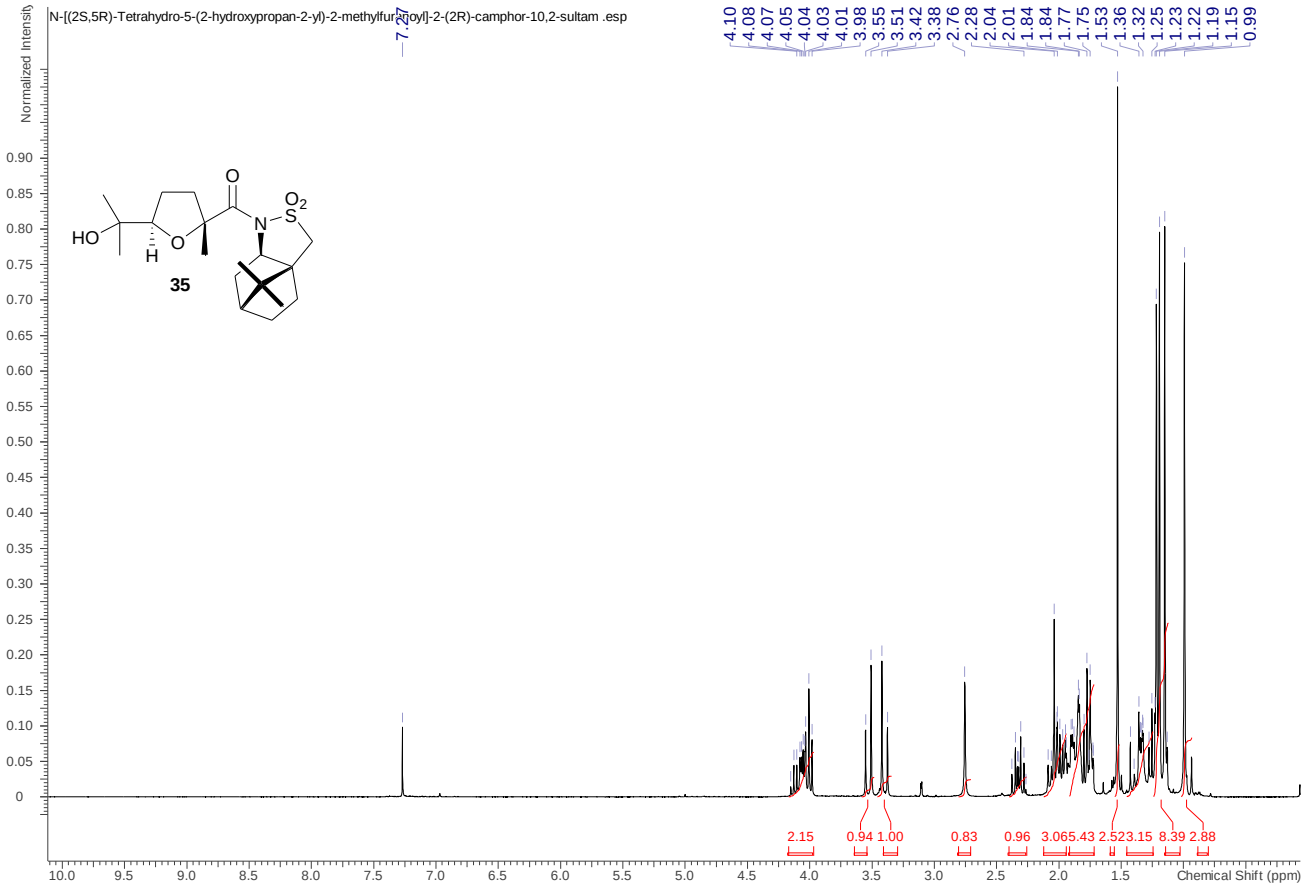


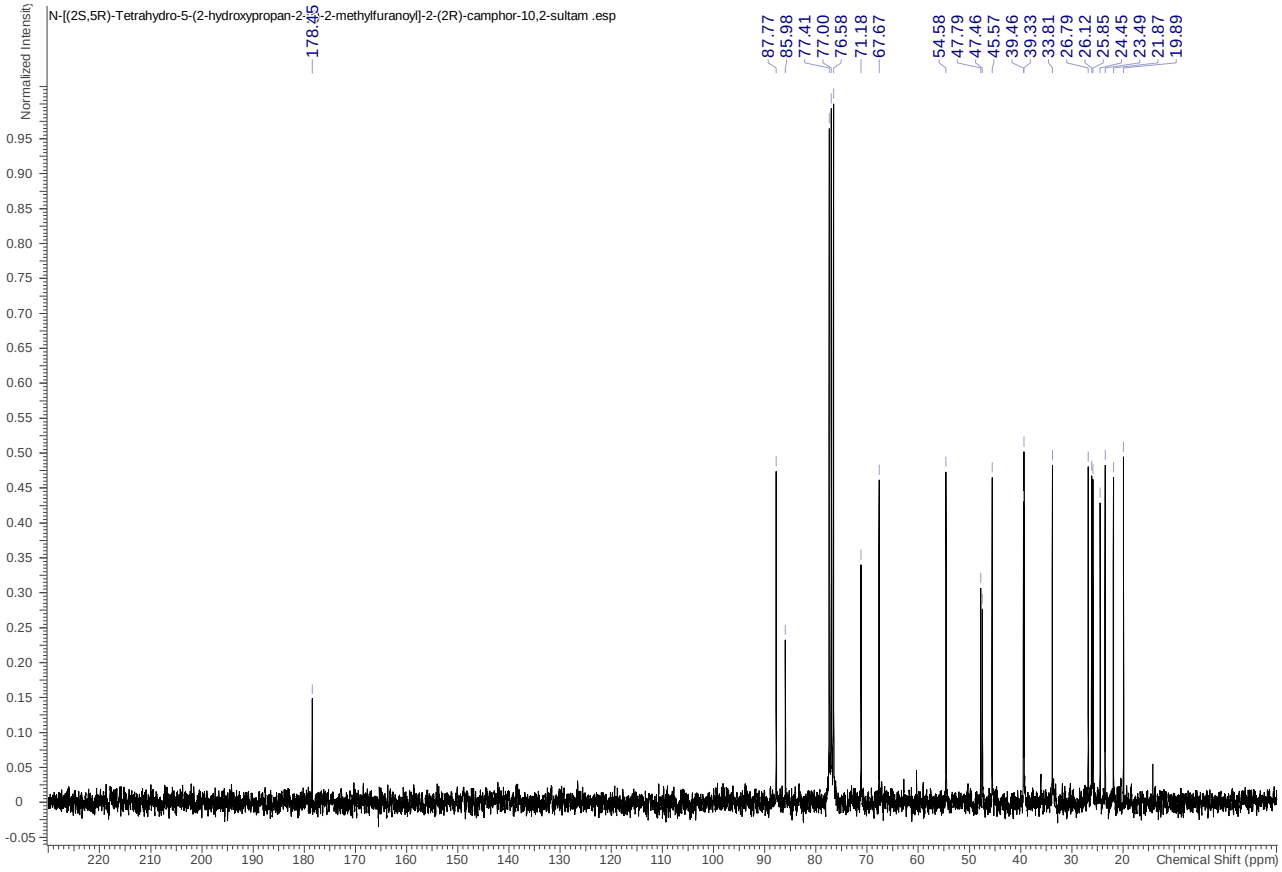

Rac.(2S,5R)-Methyl tetrahydro-2-(O-methoxy-1H-imidazol-1-carbothioyl)-5-(2-trimethylsiloxy-2-yl) furan-2-carboxylate
4.92
4.90
4.75
4.72
4.06
4.04
4.04
4.03
4.03
3.79
Normalized Intensity
0.91
0.95
0.79
2.17
Chemical Shift (ppm)


Rac.(2S,5R)-Methyl tetrahydro-2-(O-methoxy-1H-imidazol-1-carbothioyl)-5-(2-trimethylsiloxy-2-yl) furan-2-carboxylate
2.28
2.27
2.27
2.26
2.25
2.25
2.24
2.23
2.22
2.21
2.20
2.12
2.10
2.09
2.08
2.07
2.06
2.06
2.05
2.05
2.04
2.03
2.02
2.02
2.01
2.00
1.99
1.99
1.98
1.97
1.96
1.96
1.95
1.94
Normalized Intensity
0.94
2.94
Chemical Shift (ppm)

_N_-[(2_S_,5_R_)-Tetrahydro-5-(2-hydroxypropan-2-yl)-2-methylfuranoyl]-2-(2_R_)-camphor-10,2-sultam (35)

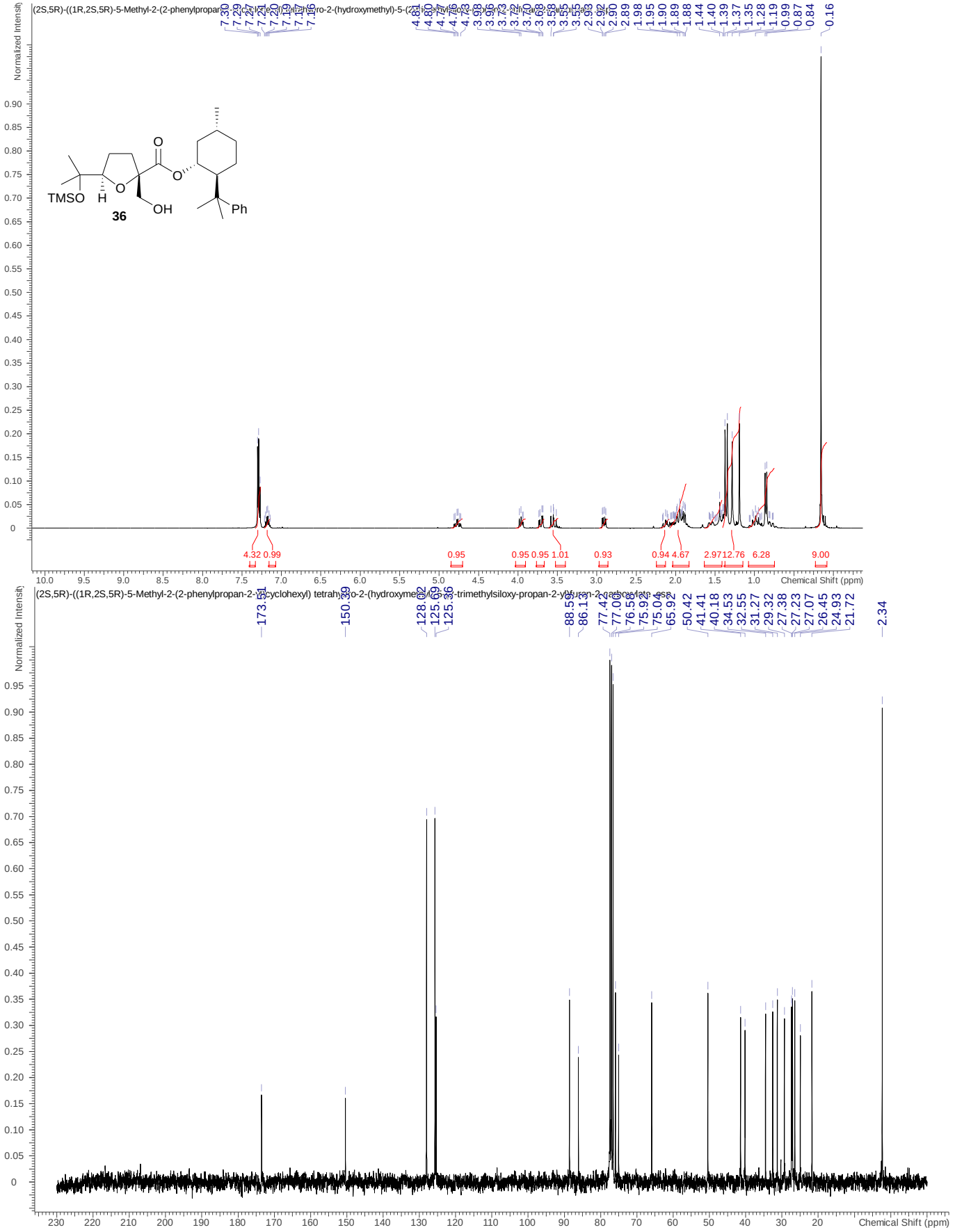
^{1}H NMR (300 MHz, $CDCl_3$) and ^{13}C NMR (75 MHz, $CDCl_3$)

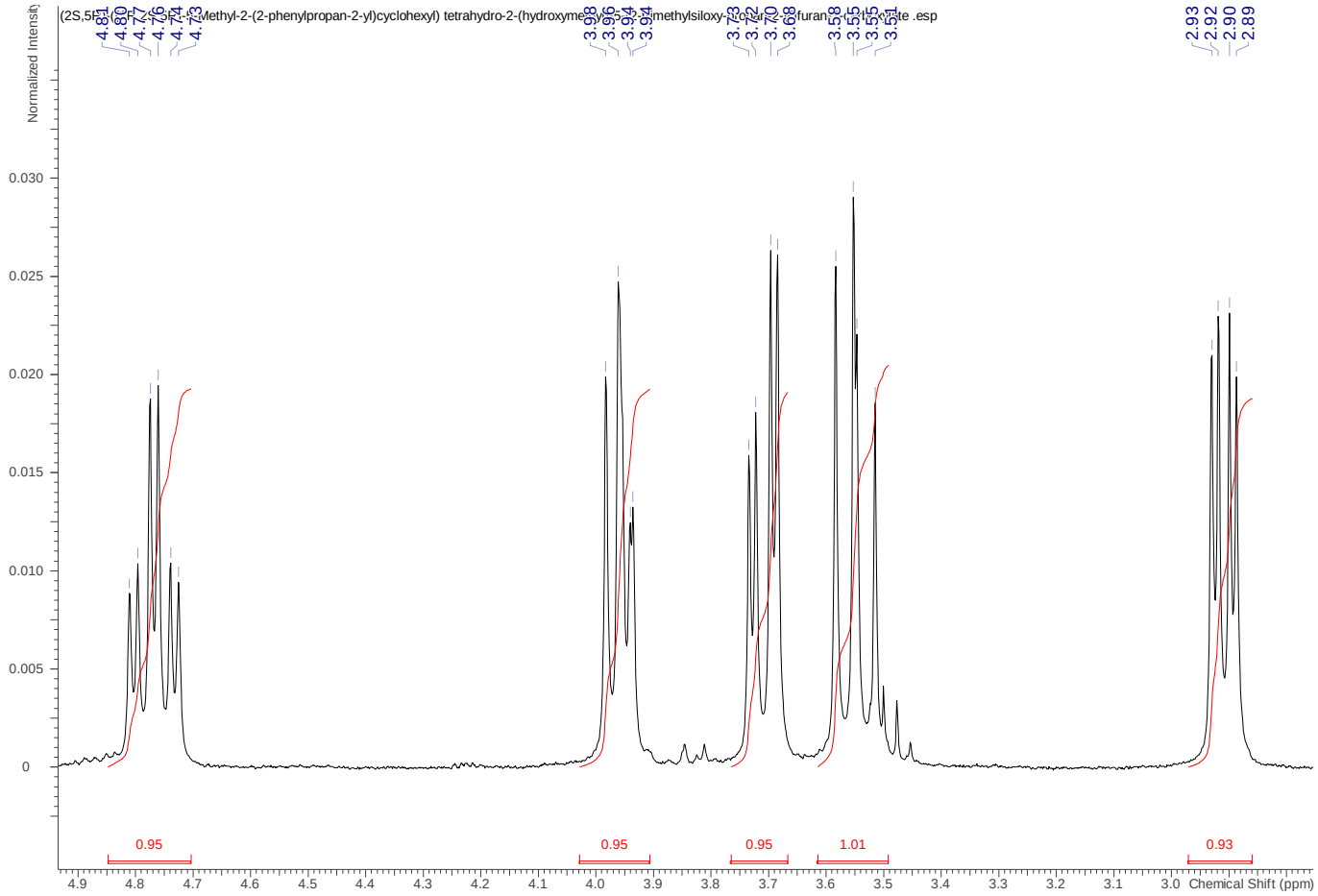


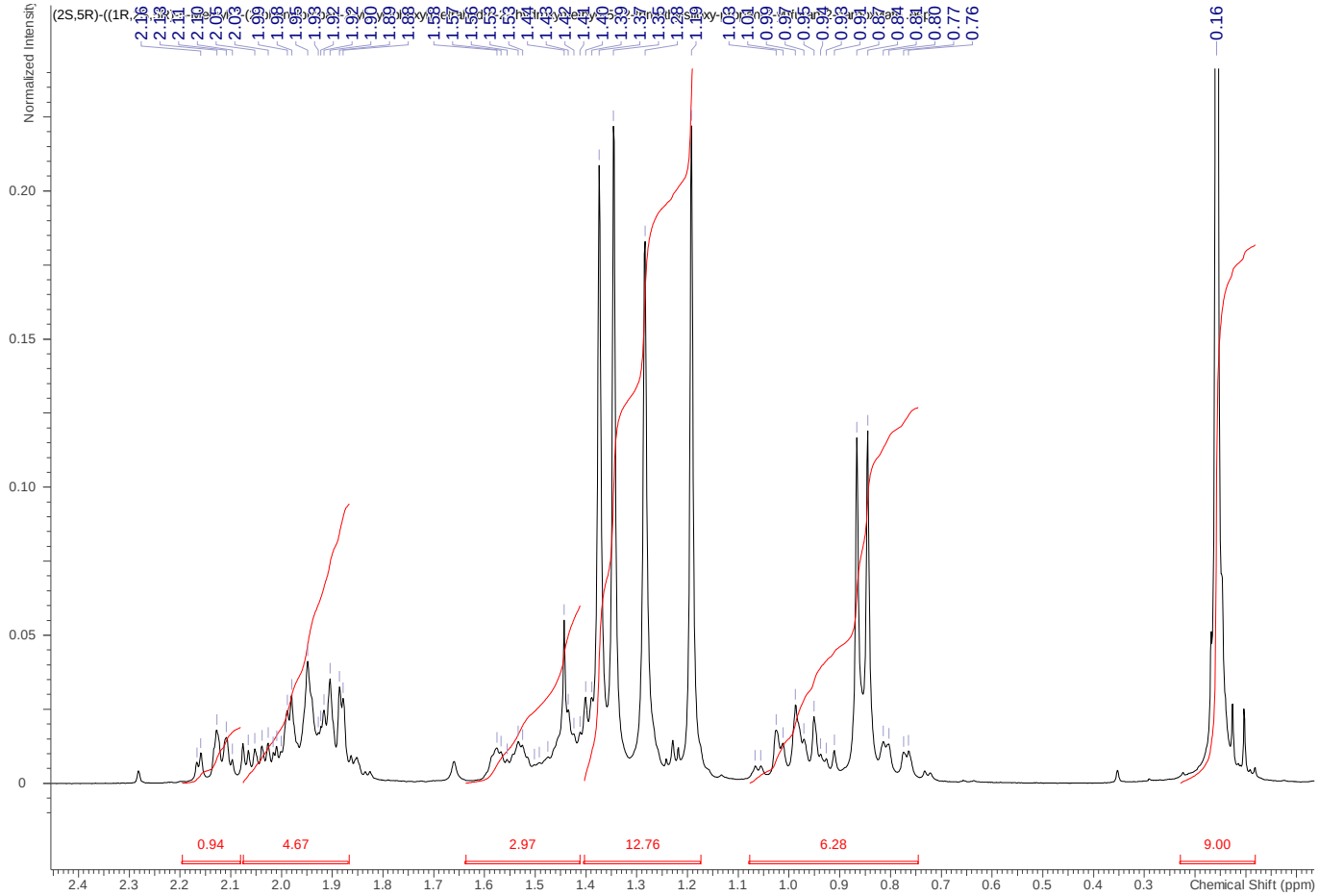


(2*S*,5*R*)-((1*R*,2*S*,5*R*)-5-Methyl-2-(2-phenylpropan-2-yl)cyclohexyl) tetrahydro-2-(hydroxymethyl)-5-(2-trimethylsiloxy-propan-2-yl)furan-2-carboxylate (36)

^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$)

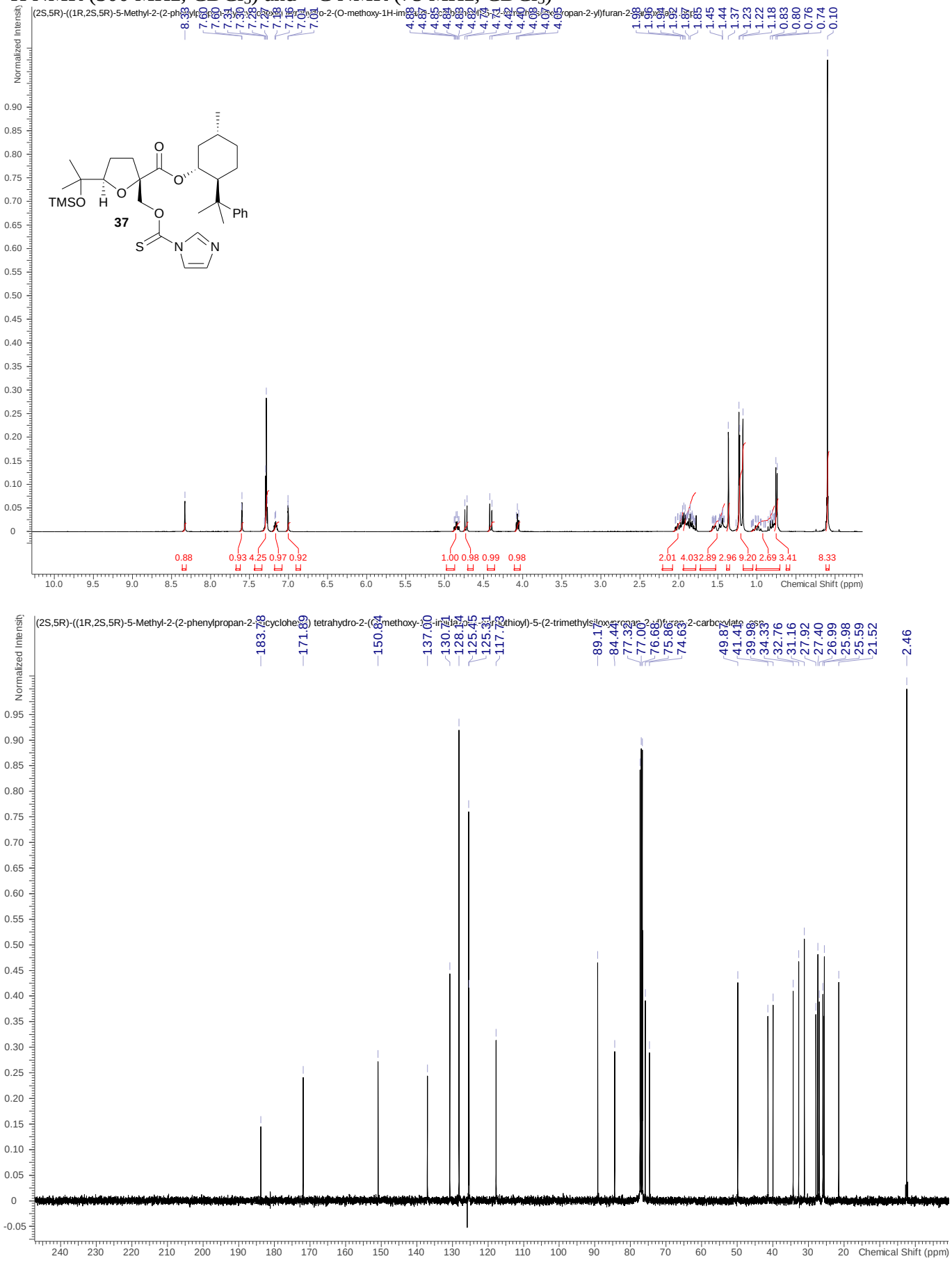


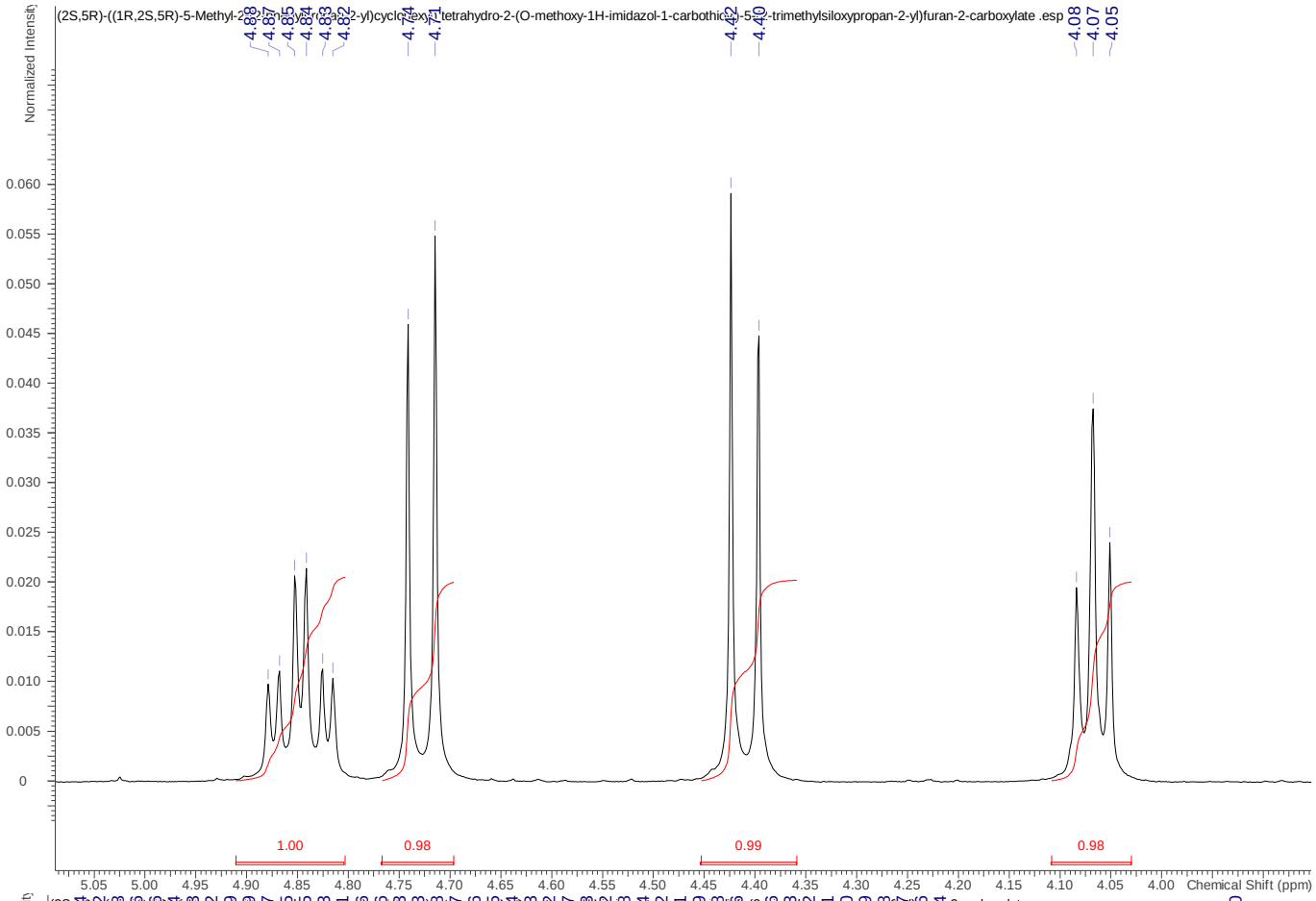

Normalized Intensity
Chemical Shift (ppm)
0.95
0.95
0.95
1.01
0.93

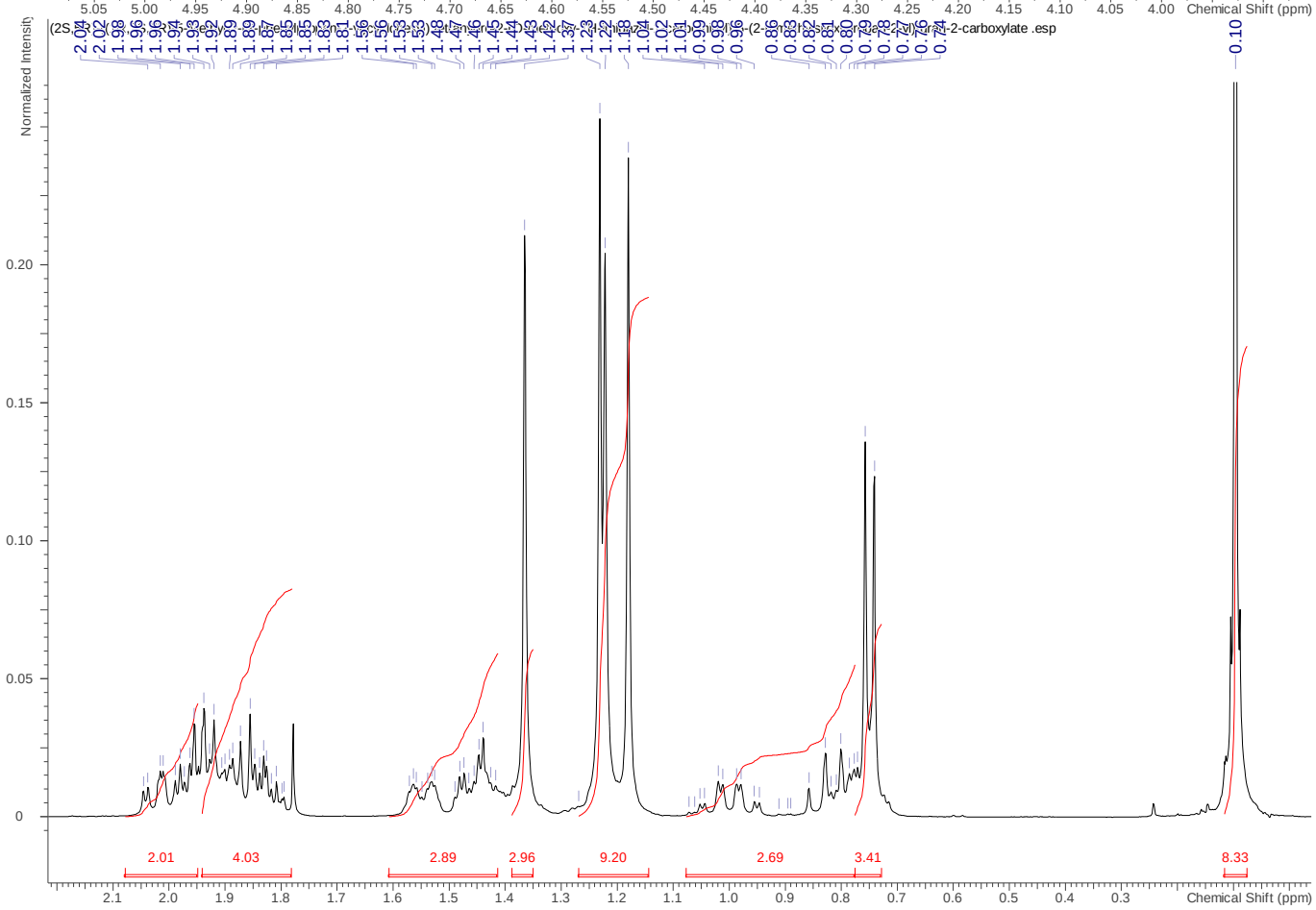

Normalized Intensity
Chemical Shift (ppm)
0.94
4.67
2.97
12.76
6.28
9.00

(2*S*,5*R*)-((1*R*,2*S*,5*R*)-5-Methyl-2-(2-phenylpropan-2-yl)cyclohexyl)tetrahydro-2-(*O*-methoxy-1*H*-imidazol-1-carbothioyl)-5-(2-trimethylsiloxypropan-2-yl)furan-2-carboxylate (37)

^{1}H NMR (300 MHz, $CDCl_3$) and ^{13}C NMR (75 MHz, $CDCl_3$)

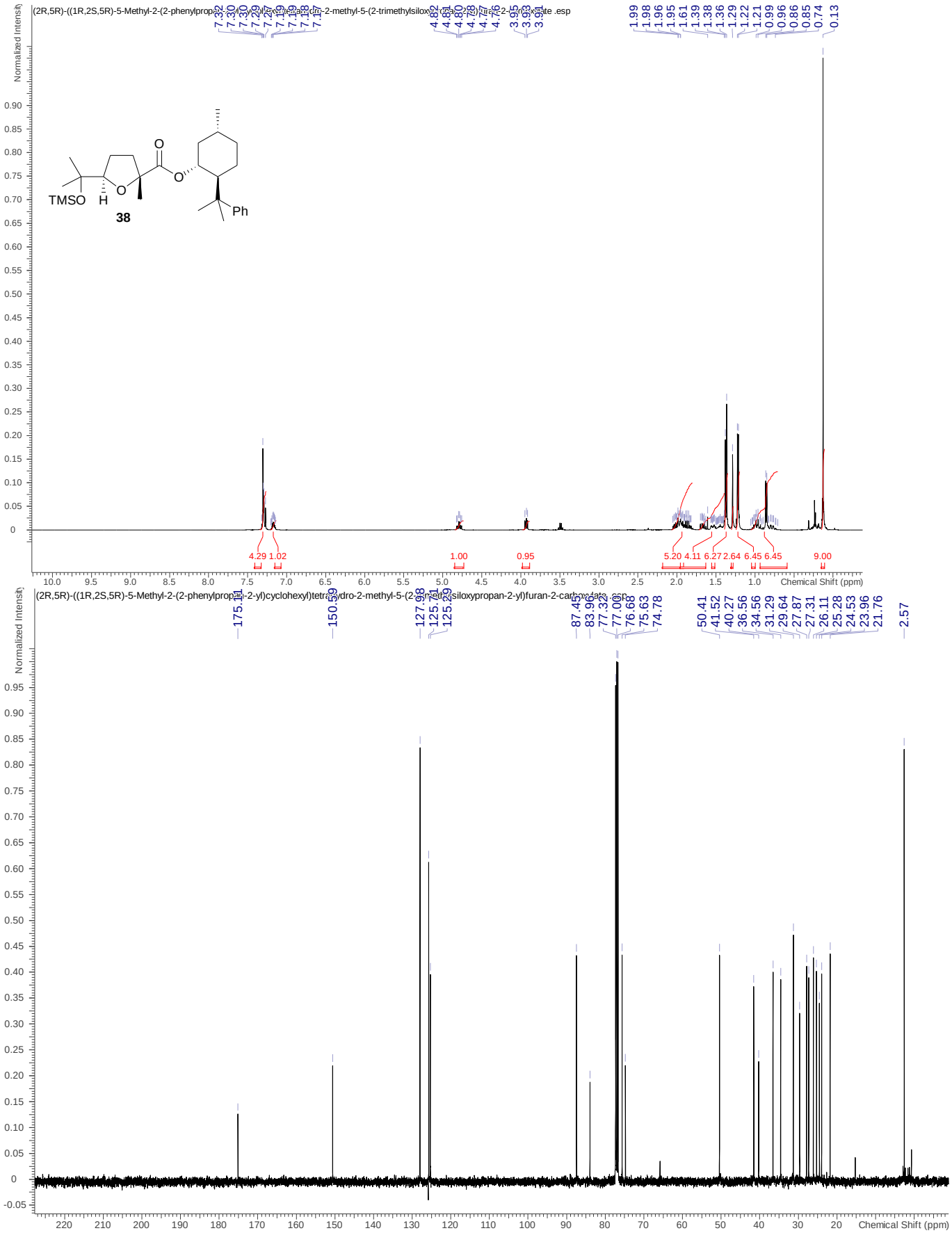


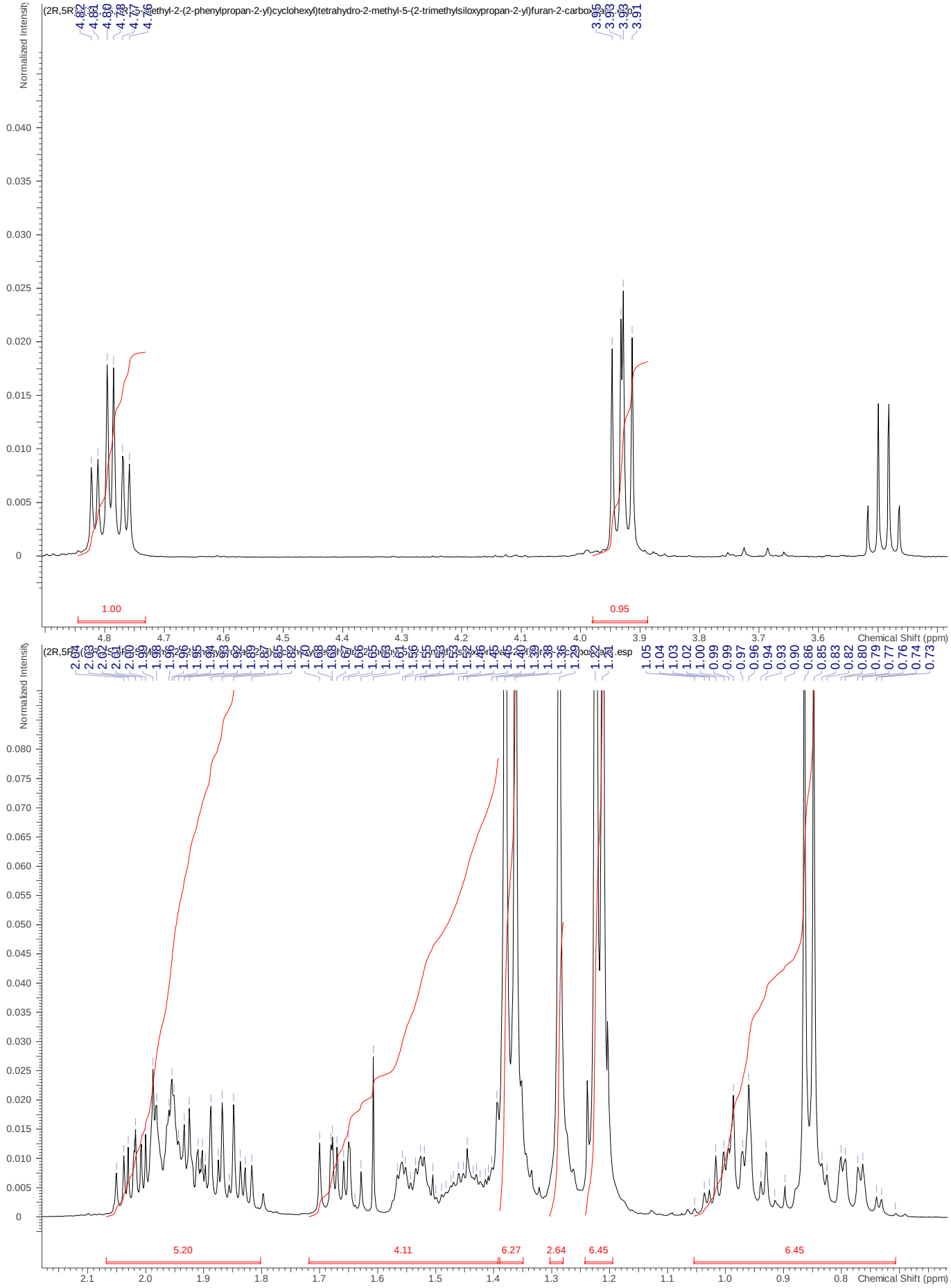

(2S,5R)-((1R,2S,5R)-5-Methyl-2-(
4.88
4.87
4.85
4.84
4.83
4.82
4.74
4.71
4.42
4.40
4.08
4.07
4.05
Normalized Intensity
1.00
0.98
0.99
0.98
Chemical Shift (ppm)


(2S
2.04
2.02
1.98
1.96
1.96
1.94
1.93
1.92
1.89
1.87
1.85
1.85
1.83
1.81
1.56
1.53
1.53
1.48
1.47
1.46
1.45
1.44
1.43
1.42
1.37
1.23
1.22
1.18
1.04
1.02
1.01
0.99
0.98
0.96
0.86
0.83
0.82
0.81
0.80
0.79
0.78
0.77
0.76
0.74
0.10
Normalized Intensity
2.01
4.03
2.89
2.96
9.20
2.69
3.41
8.33
Chemical Shift (ppm)

(2*R*,5*R*)-((1*R*,2*S*,5*R*)-5-Methyl-2-(2-phenylpropan-2-yl)cyclohexyl)tetrahydro-2-methyl-5-(2-trimethylsiloxypropan-2-yl)furan-2-carboxylate (38)

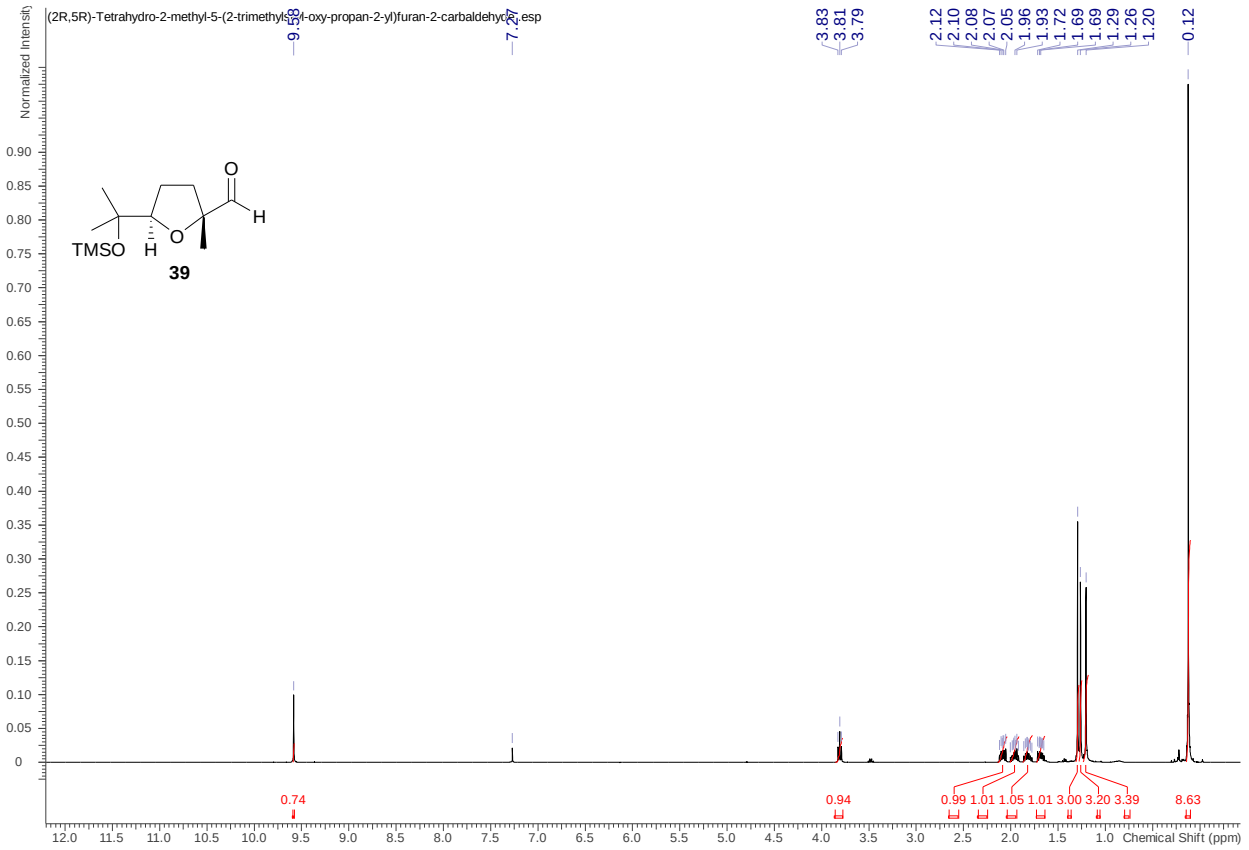
^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (100 MHz, $CDCl_3$)

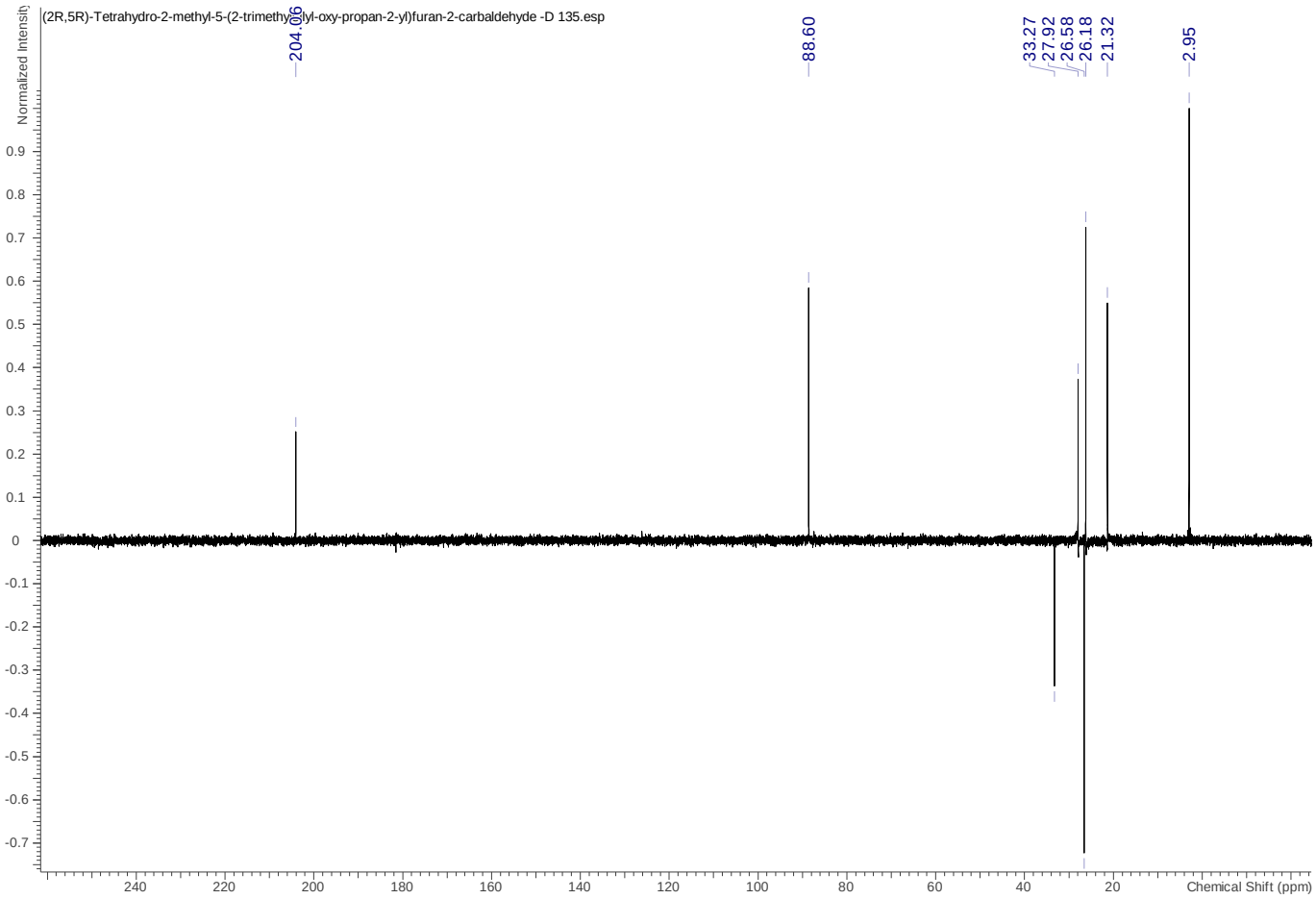


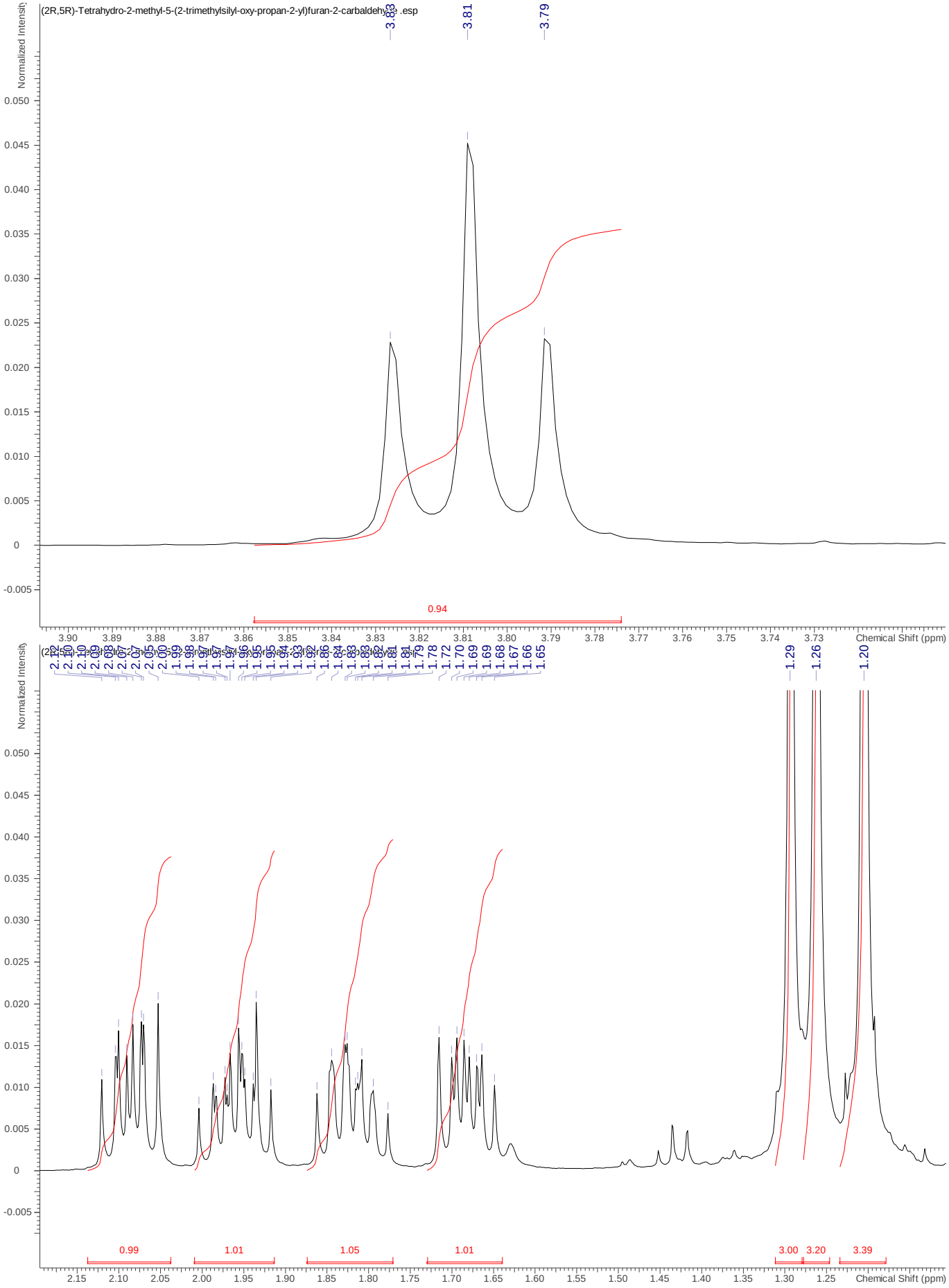

(2R,5R)-5-(1-methyl-2-(2-phenylpropan-2-yl)cyclohexyl)tetrahydro-2-methyl-5-(2-trimethylsiloxypropan-2-yl)furan-2-carboxylate
Normalized Intensity
Chemical Shift (ppm)
1.00
0.95
5.20
4.11
6.27
2.64
6.45
6.45

(2*R*,5*R*)-Tetrahydro-2-methyl-5-(2-trimethylsilyl-oxy-propan-2-yl)furan-2-carbaldehyde (39)

^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C DEPT135 NMR (100 MHz, $CDCl_3$)






(2R,5R)-Tetrahydro-2-methyl-5-(2-trimethylsilyl-oxy-propan-2-yl)furan-2-carbaldehyde .esp
Normalized Intensity
3.83
3.81
3.79
0.94
Chemical Shift (ppm)
2.12
2.10
2.09
2.08
2.07
2.07
2.05
2.00
1.99
1.98
1.97
1.97
1.97
1.96
1.95
1.95
1.94
1.93
1.92
1.86
1.84
1.83
1.83
1.82
1.81
1.81
1.79
1.78
1.72
1.70
1.69
1.69
1.68
1.67
1.66
1.65
1.29
1.26
1.20
0.99
1.01
1.05
1.01
3.00
3.20
3.39