

Supporting information

(NMR Spectral Data)

Total synthesis of (+)-monocerin via tandem dihydroxylation-S_N2 cyclization and copper mediated tandem cyanation-lactonization approach

U. Nookaraju, Eeshwaraiah Begari* and Pradeep Kumar*

*Division of Organic Chemistry, CSIR-National Chemical Laboratory, Pune
411008, India.*

Telephone number: +91-20-25902050

Fax number: +91-20-25902629

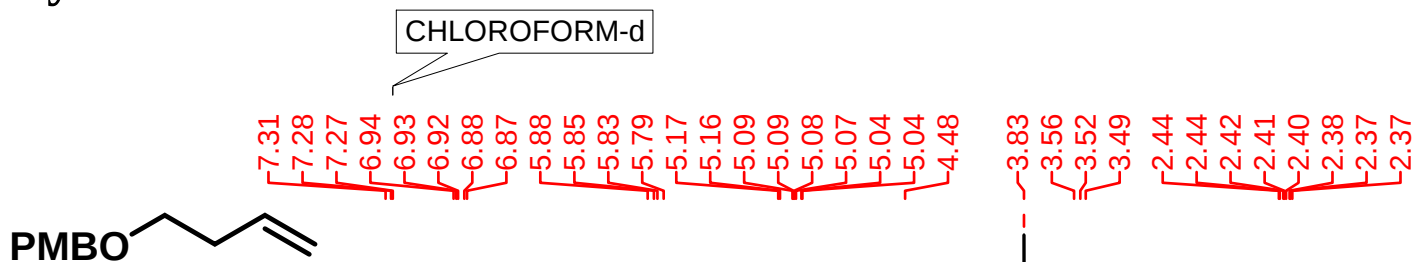
E-mail : e.begari@ncl.res.in

Table of Contents.

¹ H NMR and ¹³ C NMR spectra	1 - 53
--	--------

NMR Spectral Data

^1H NMR (200MHz, CDCl_3) of the compound 1-((but-3-en-1-yloxy)methyl)-4-methoxybenzene :

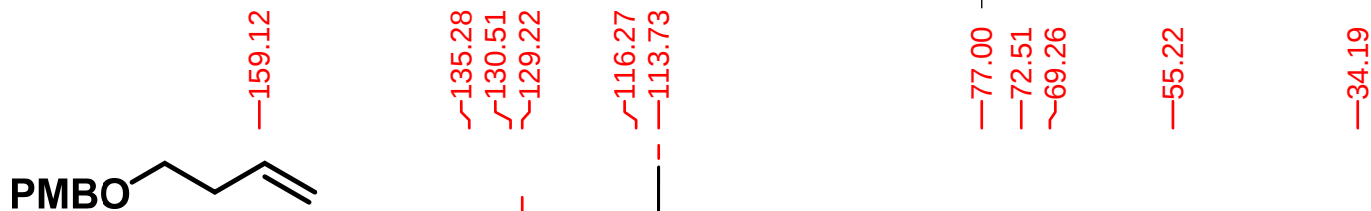


7.31 7.28 7.27 6.94 6.93 6.92 6.88 6.87 5.88 5.85 5.83 5.79 5.17 5.16 5.09 5.09 5.08 5.07 5.04 5.04 4.48 3.83 3.56 3.52 3.49 2.44 2.44 2.42 2.41 2.40 2.38 2.37 2.37

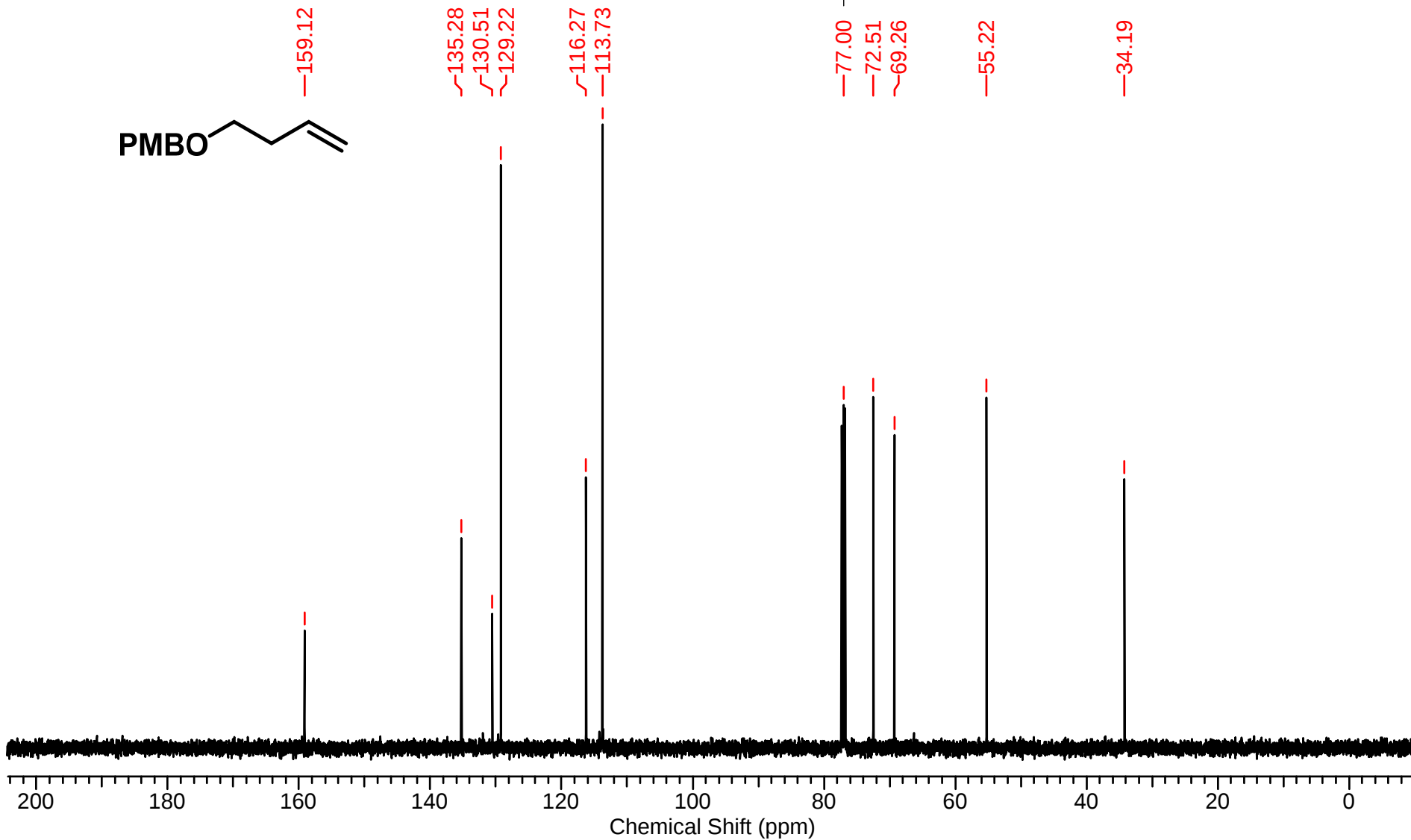
2.20 2.18 1.00 2.06 2.16 3.06 2.14 2.13

Chemical Shift (ppm)

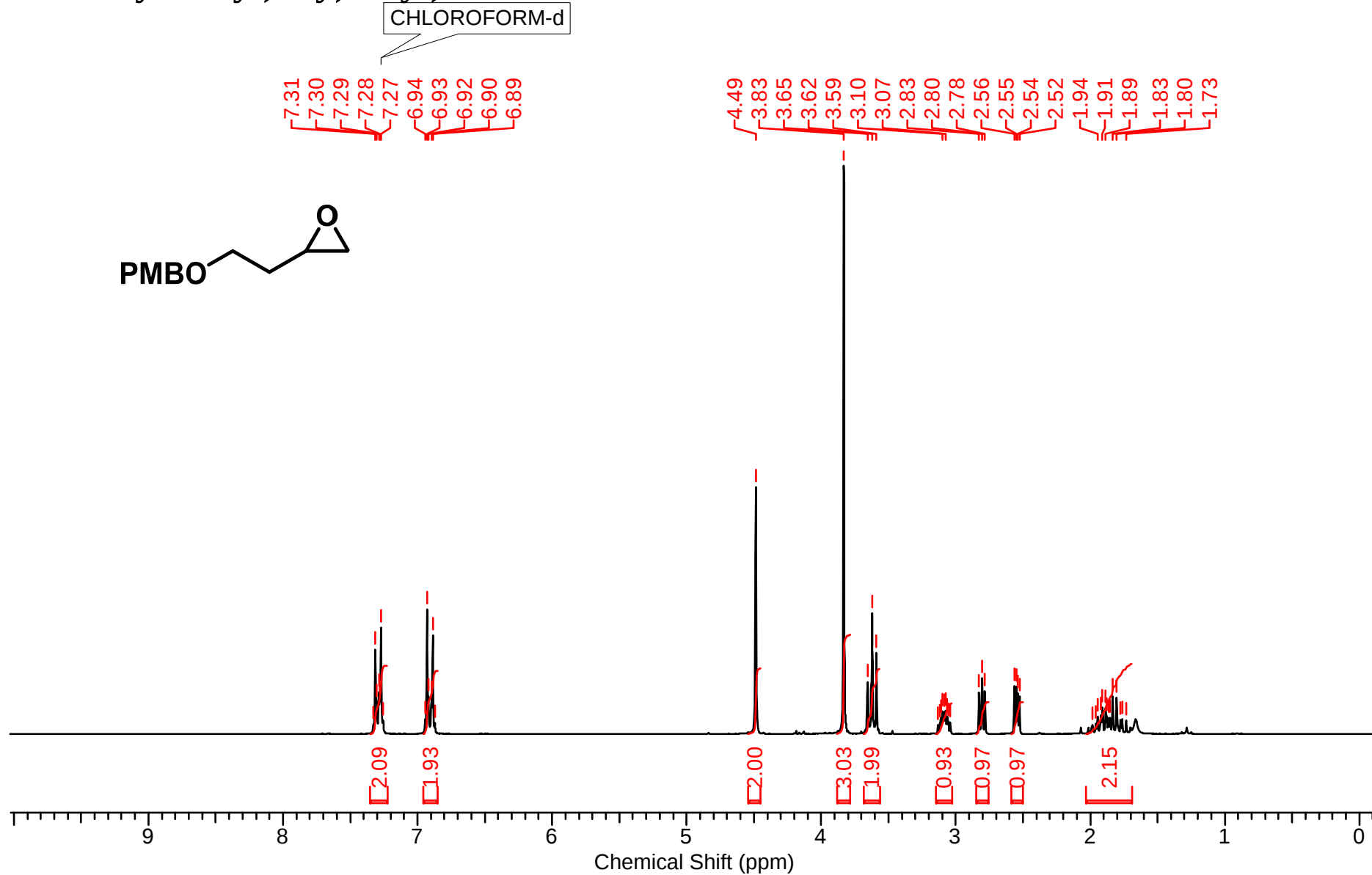
^{13}C NMR (126MHz, CDCl_3) of the compound 1-((but-3-en-1-yloxy)methyl)-4-methoxybenzene :



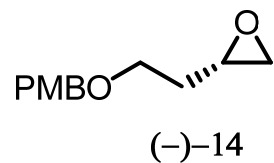
CHLOROFORM-d



^1H NMR (500MHz, CDCl_3) Of the compound 2-((4-methoxybenzyl)oxy)ethyl oxirane



^1H NMR (200MHz, CDCl_3) Of the compound (S)-2-(2-((4-methoxybenzyl)oxy)ethyl)oxirane :



CHLOROFORM-d

7.30
7.27
7.25
6.91
6.87

4.47
3.82
3.64
3.60
3.57
3.09
3.08
3.05
3.04
2.81
2.79
2.77
2.55
2.53
2.52
2.51
1.90
1.89
1.82
1.79

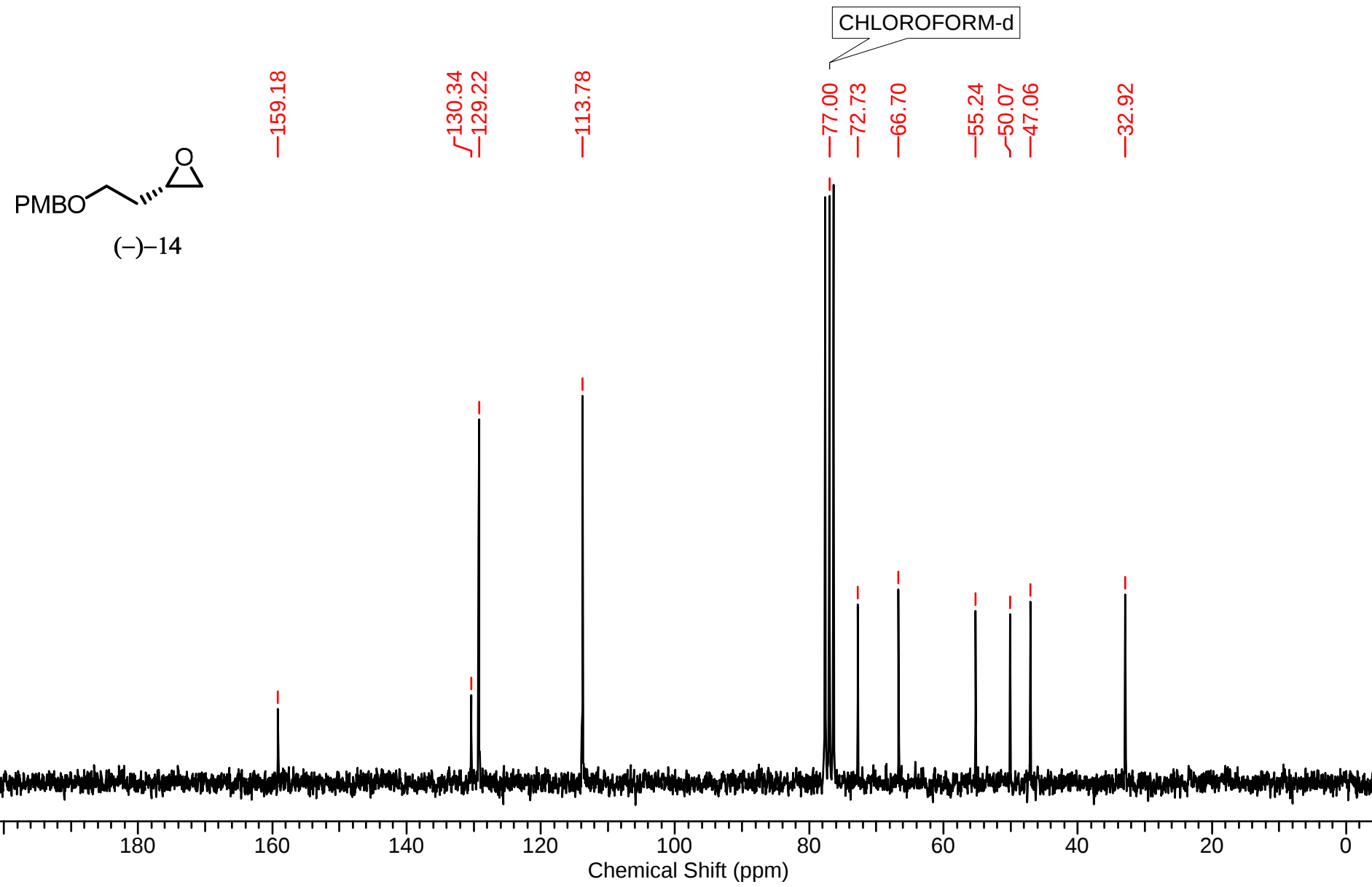
2.45
2.00

2.00
3.02
2.01

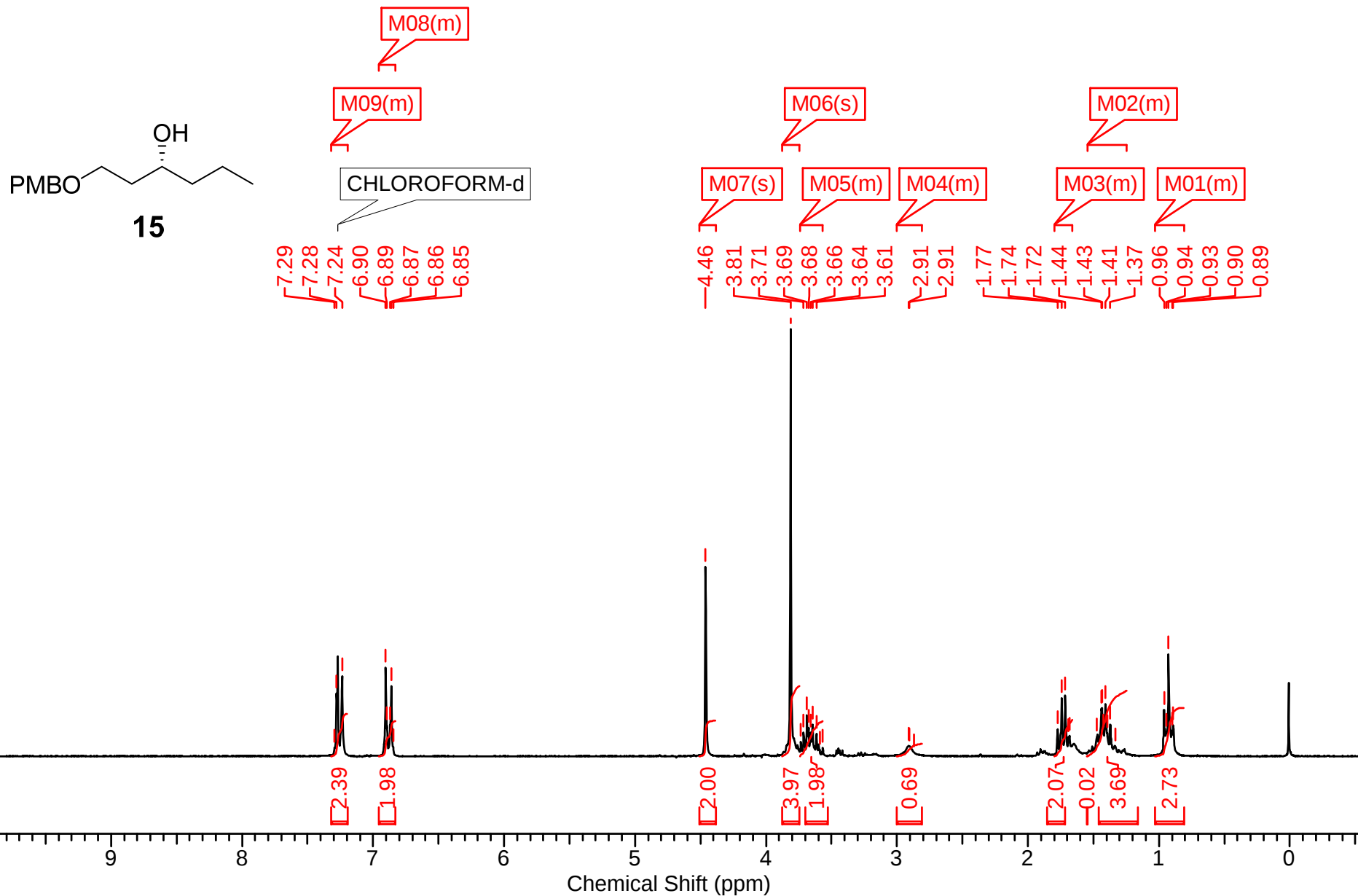
0.99
1.00
1.01
2.11

Chemical Shift (ppm)

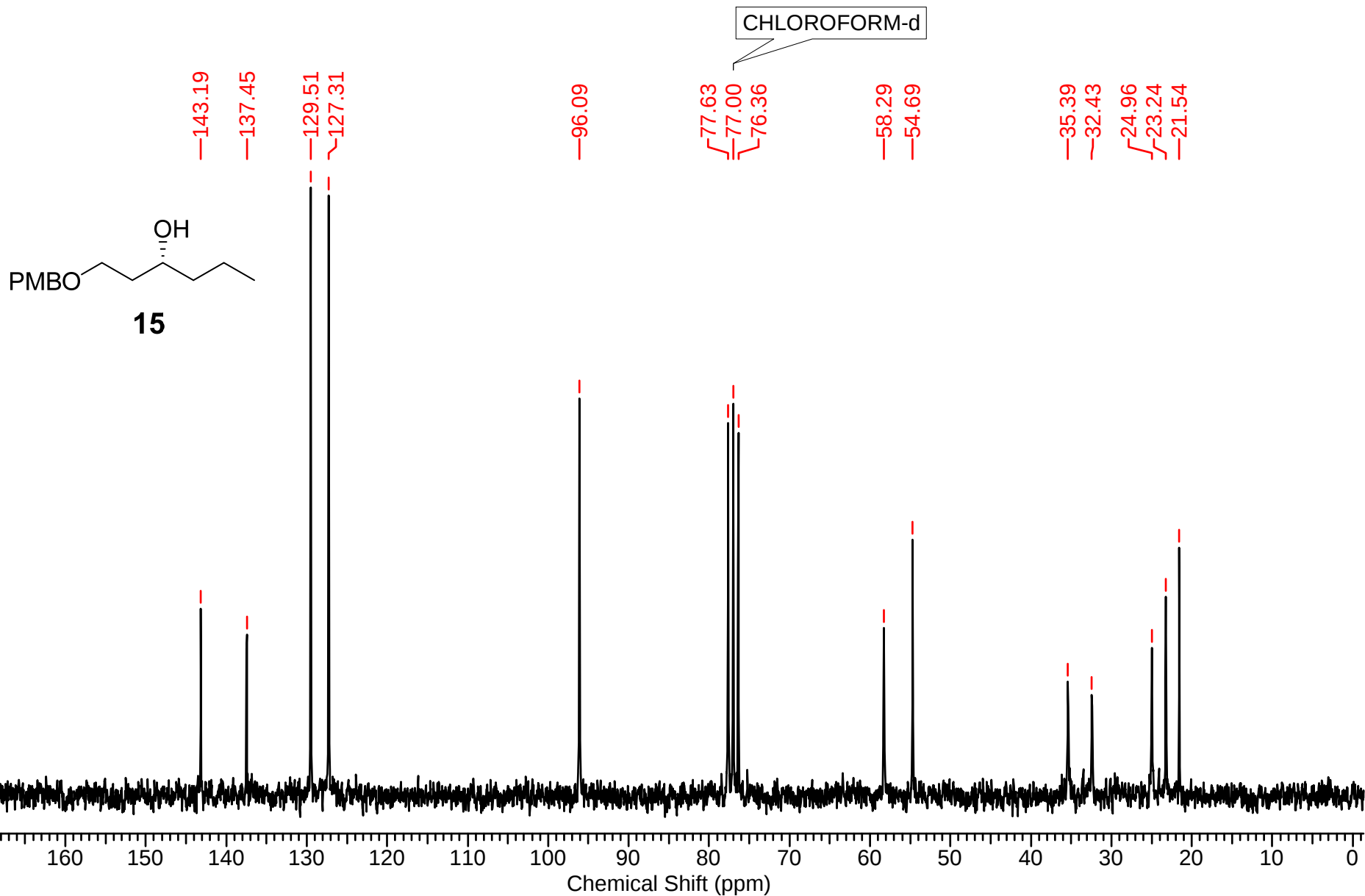
¹³C NMR (50MHz ,CDCl₃) Of the compound (S)-2-(2-((4-methoxybenzyl)oxy)ethyl)oxirane :



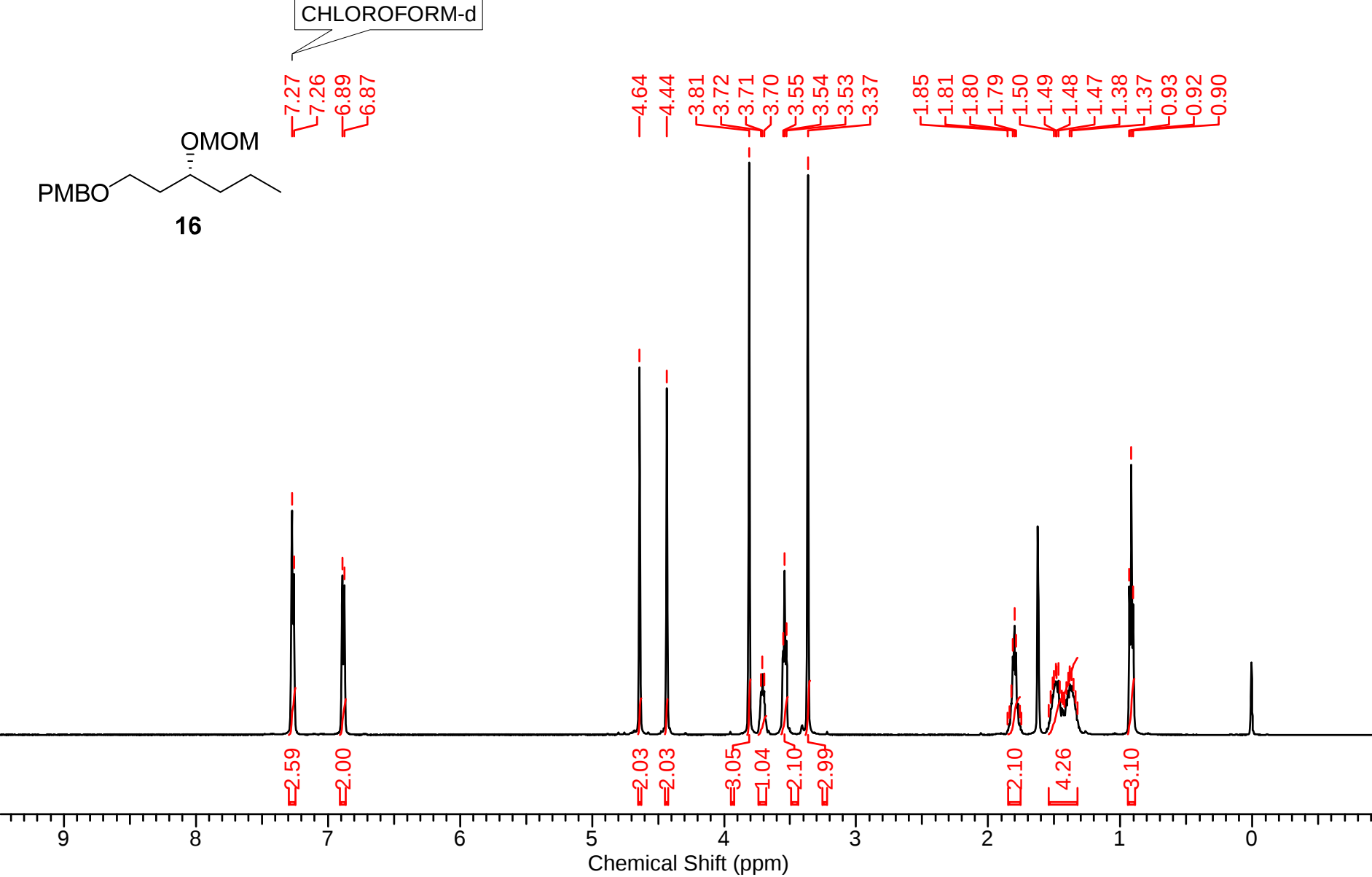
^1H NMR (200MHz, CDCl_3) Of the compound (*R*)-1-((4-methoxybenzyl)oxy)hexan-3-ol:



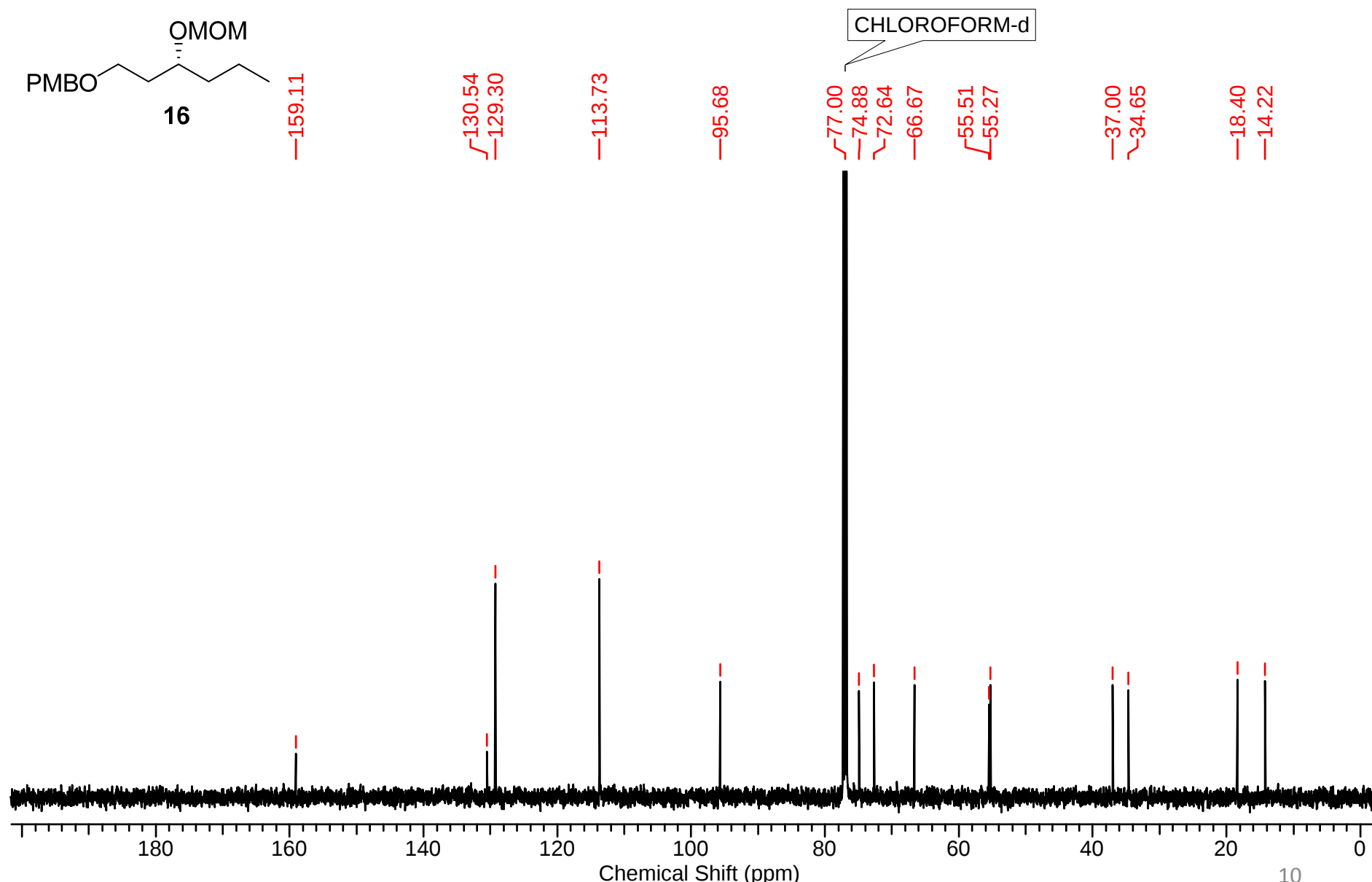
^{13}C NMR (125MHz, CDCl_3) Of the compound (*R*)-1-((4-methoxybenzyl)oxy)hexan-3-ol



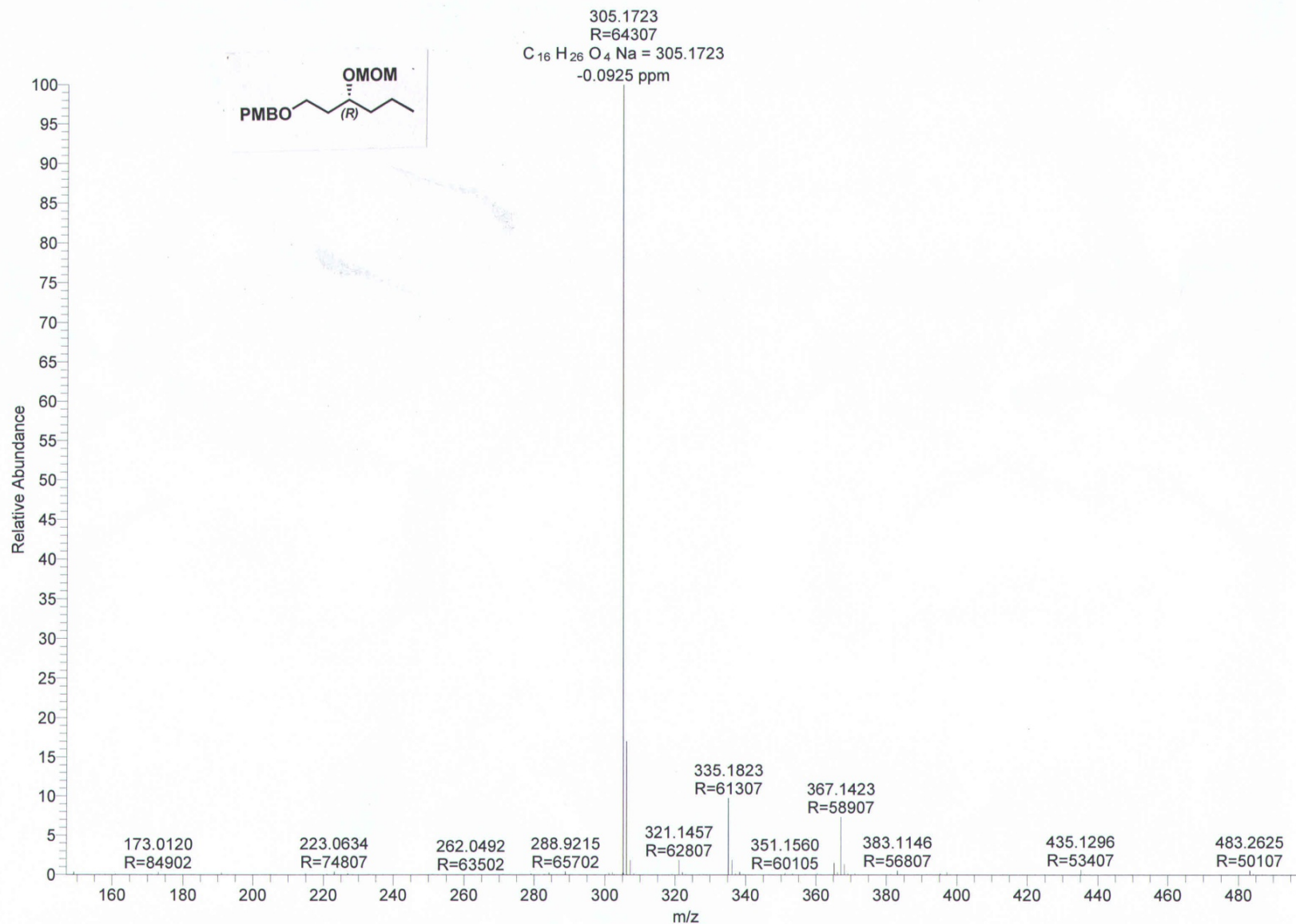
¹H NMR (500MHz ,CDCl₃) Of the compound (*R*)-1-methoxy-4-(((3-(methoxymethoxy)hexyl)oxy)methyl)benzene :



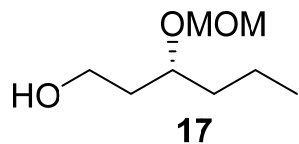
¹³C NMR (125MHz ,CDCl₃) Of the compound (*R*)-1-methoxy-4-(((3 (methoxymethoxy)hexyl)oxy)methyl)benzene :



UNR-A-3 #1225 RT: 5.49 AV: 1 NL: 1.66E9
T: FTMS + p ESI Full ms [100.00-700.00]



^1H NMR (500MHz, CDCl_3) Of the compound (*R*)-3-(methoxymethoxy)hexan-1-ol



CHLOROFORM-d

-7.27

4.71
4.69
4.67
4.65

3.83
3.82
3.81
3.80
3.78
3.77
3.74
3.73
3.41

2.04
1.82
1.81
1.56
1.49
1.48
1.38
1.37
1.36
1.35
1.35
0.95
0.93
0.92

1.01
1.00

3.08

3.01

1.05
1.15

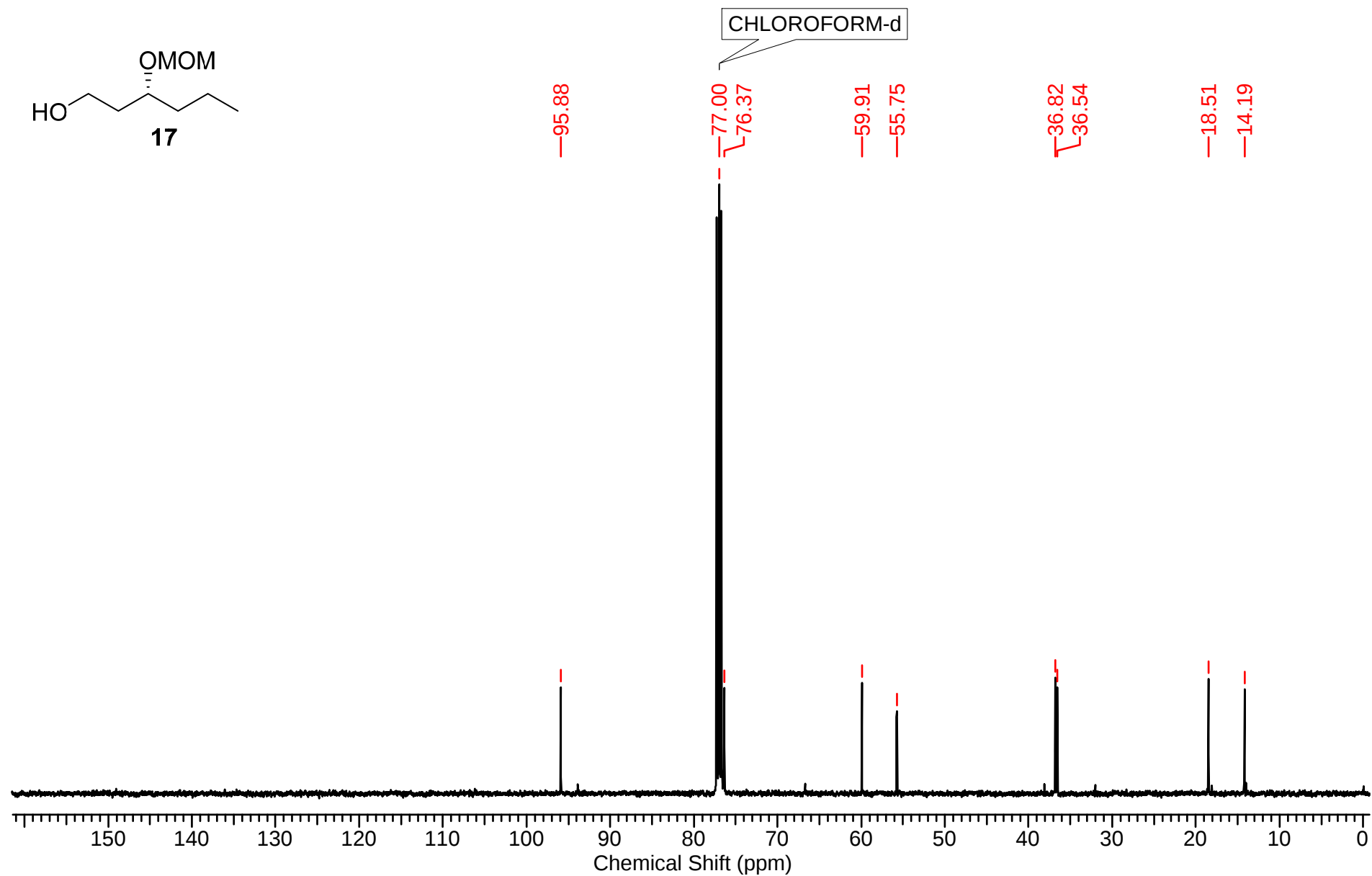
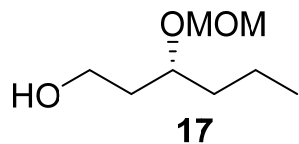
1.13
1.13

1.24
2.13

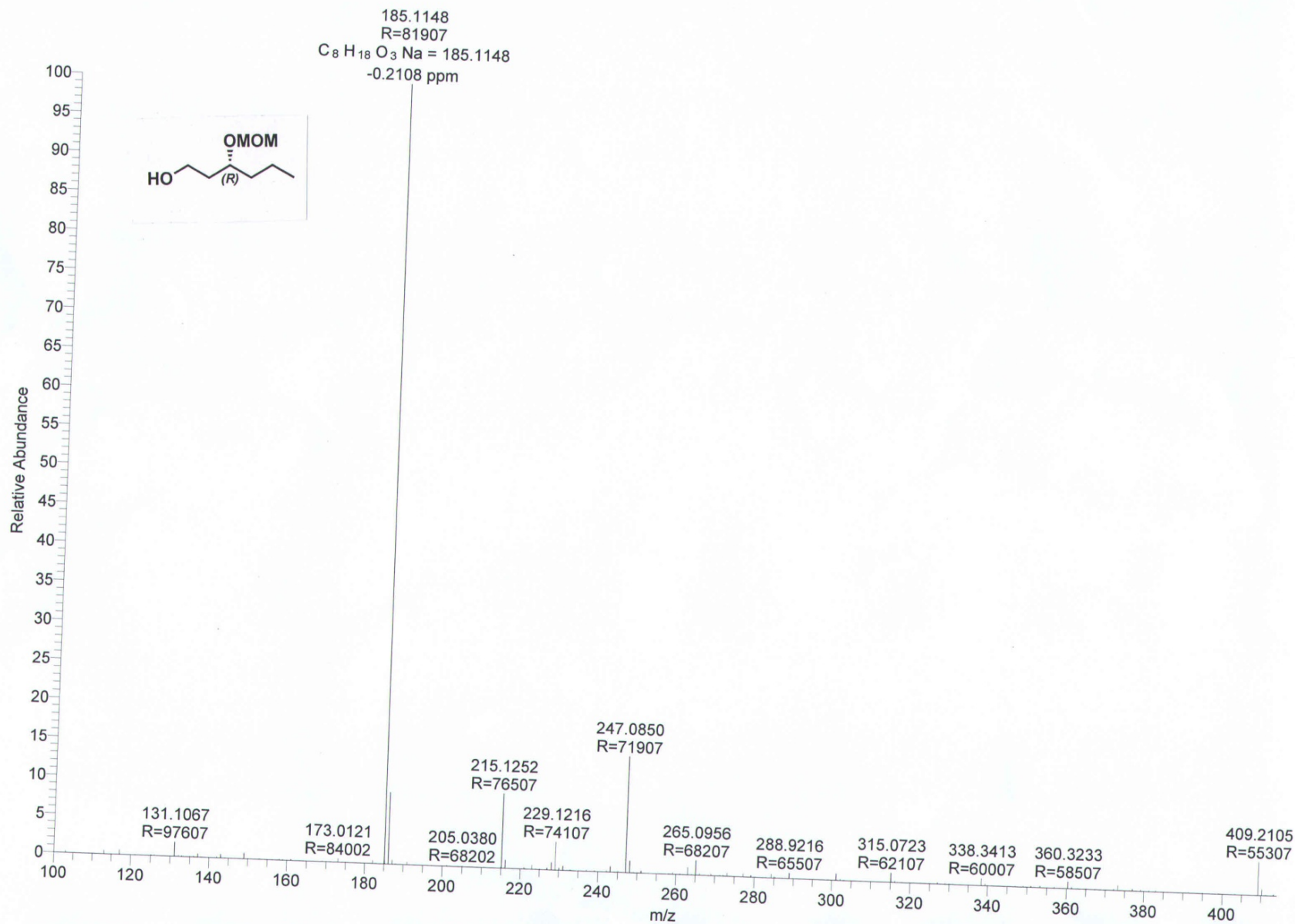
3.10

Chemical Shift (ppm)

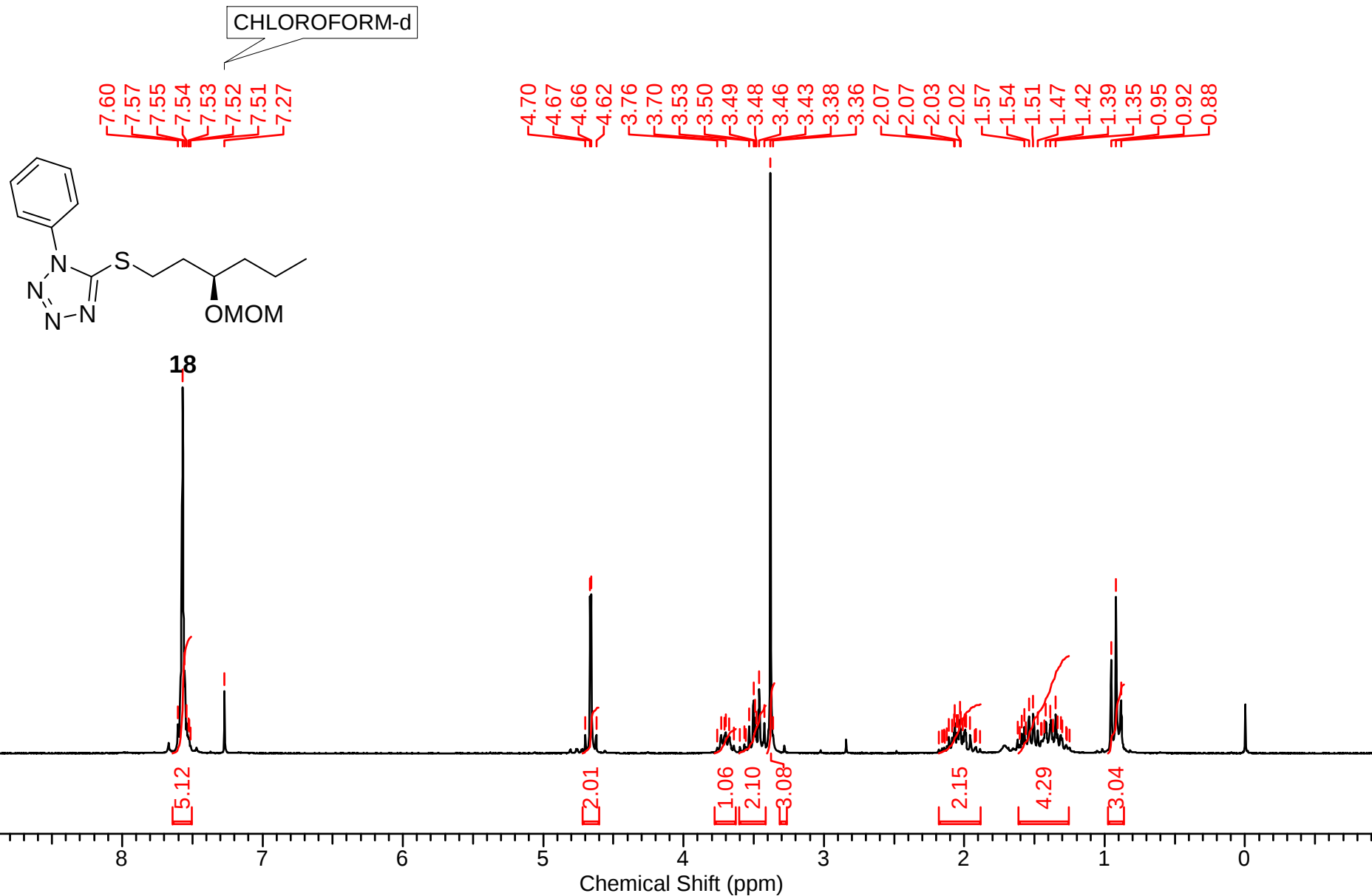
^{13}C NMR (125MHz, CDCl_3) Of the compound (*R*)-3-(methoxymethoxy)hexan-1-ol



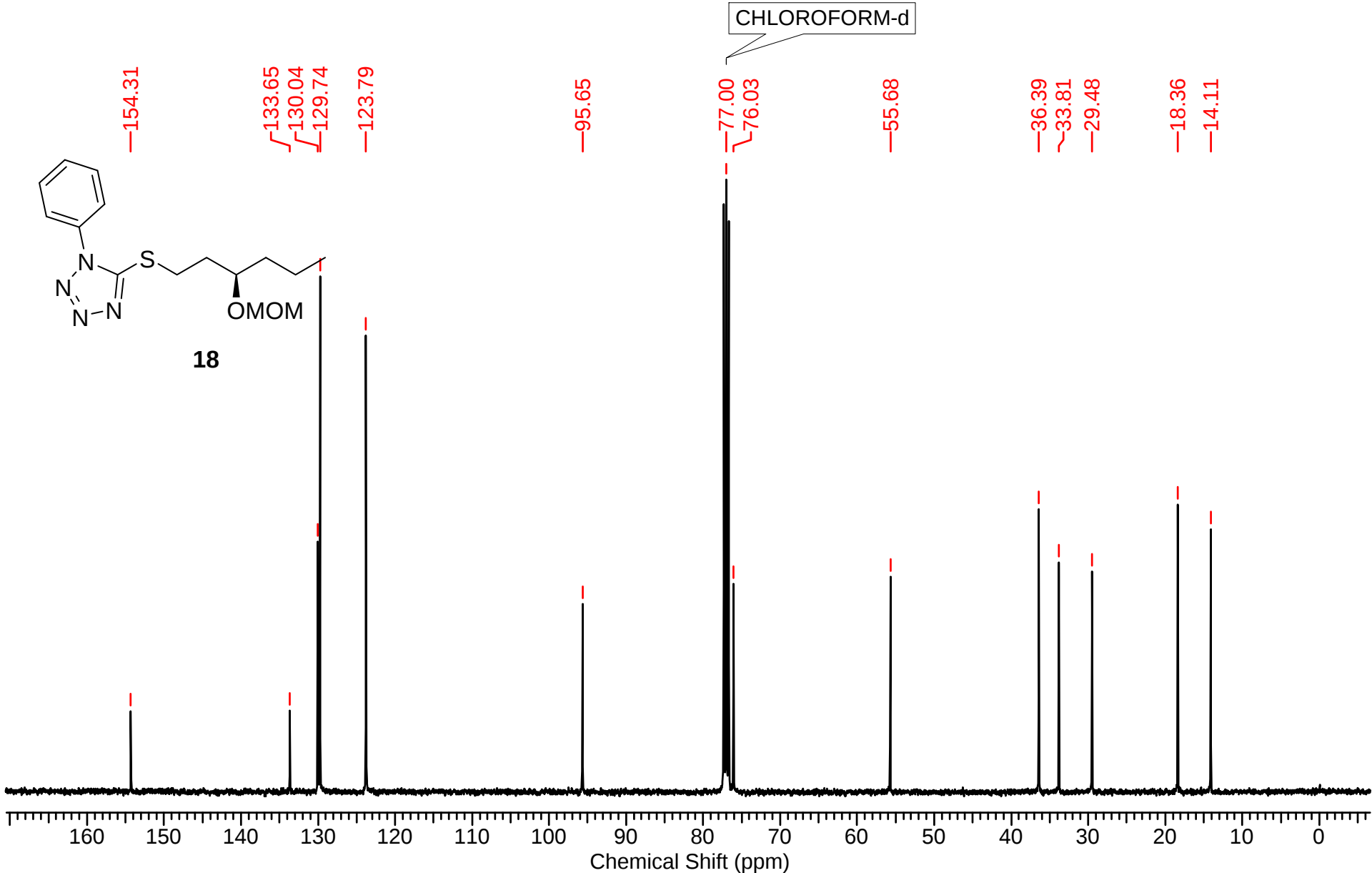
UNR-A-4 #916 RT: 4.10 AV: 1 NL: 8.42E8
T: FTMS + p ESI Full ms [100.00-700.00]



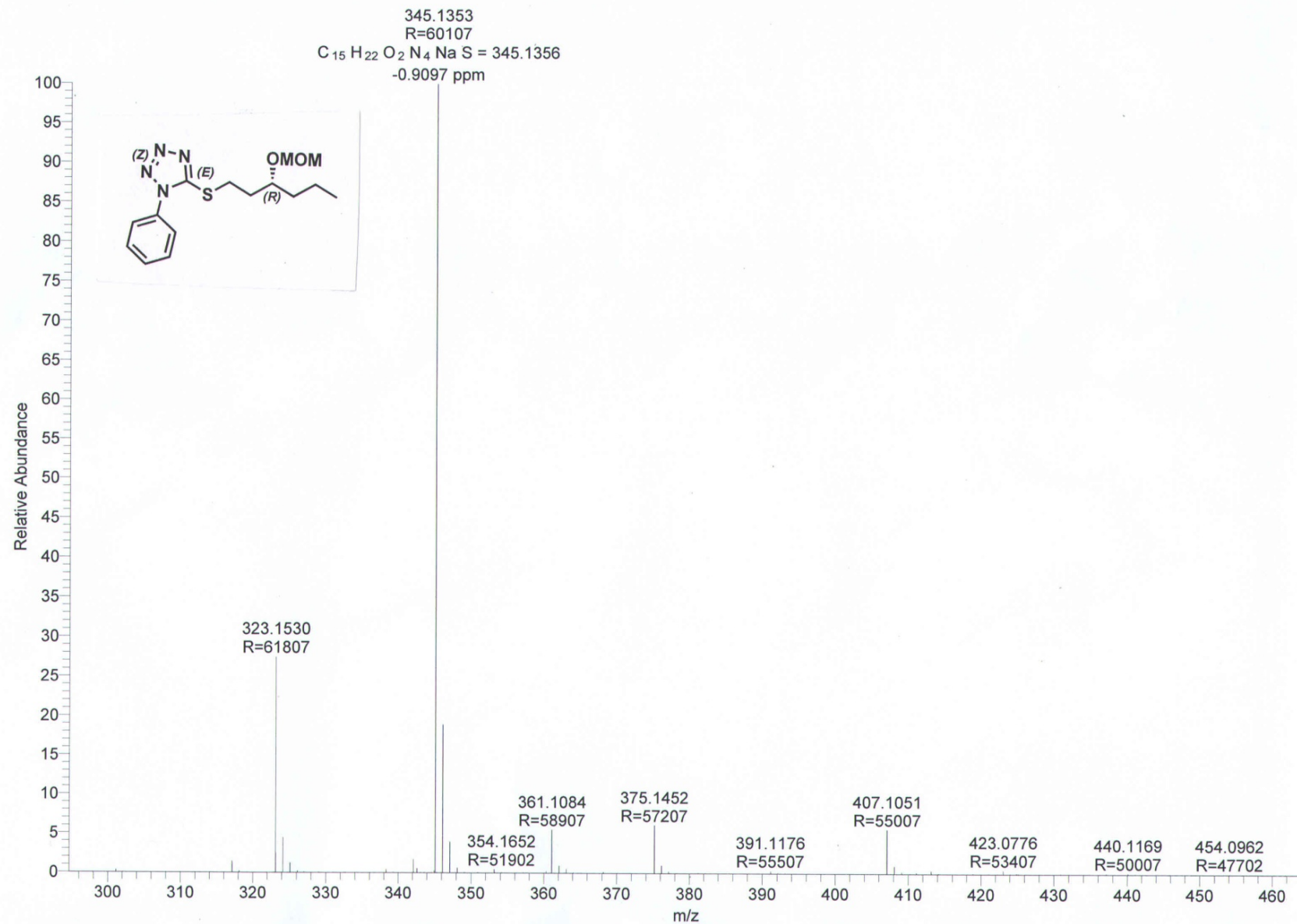
¹H NMR (200MHz ,CDCl₃) Of the compound (*R*)-5-((3-(methoxymethoxy)hexyl)thio)-1-phenyl-1H-tetrazole



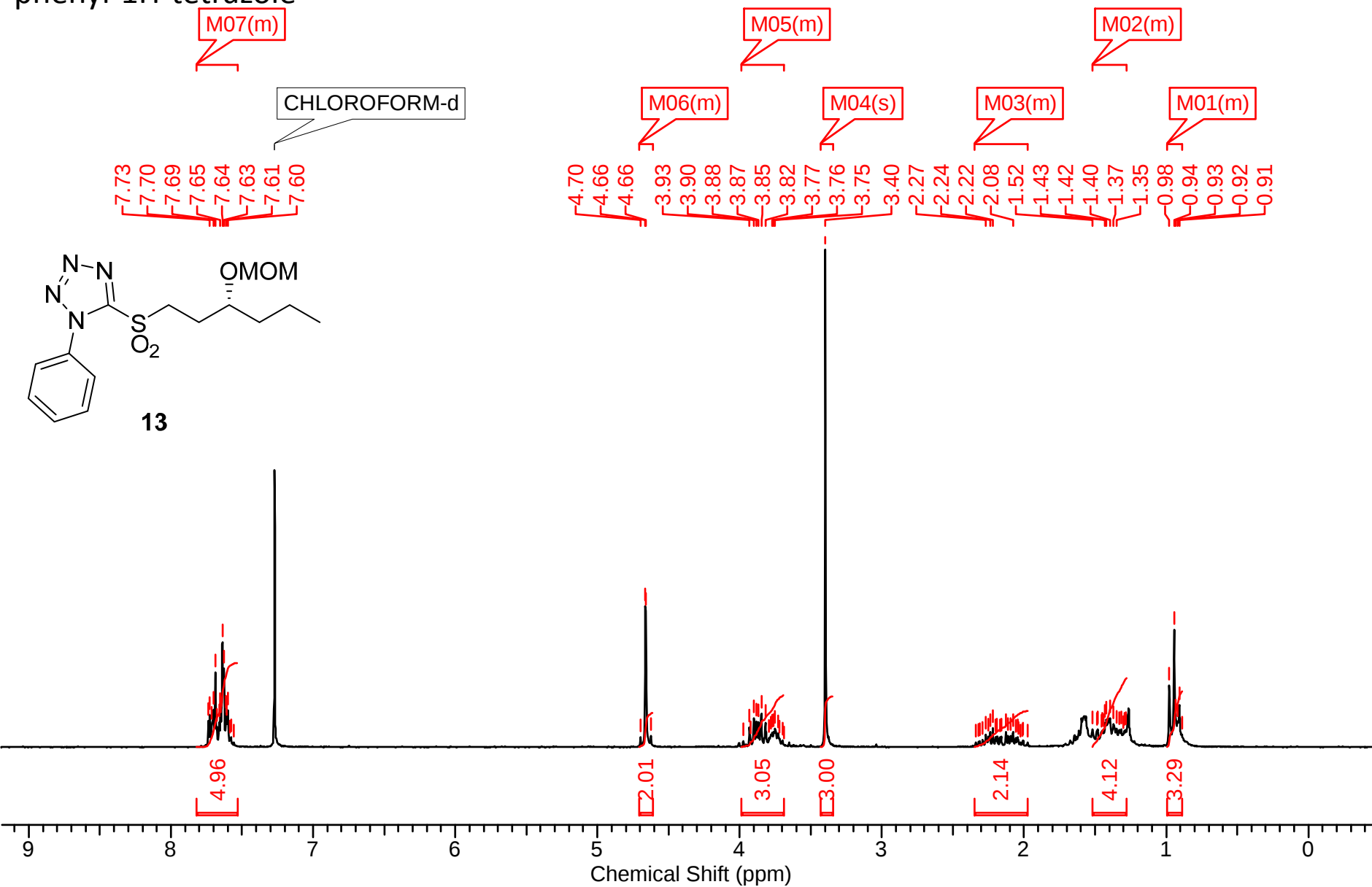
¹³C NMR (100MHz ,CDCl₃) Of the compound (R)-5-((3-(methoxymethoxy)hexyl)thio)-1-phenyl-1H-tetrazole



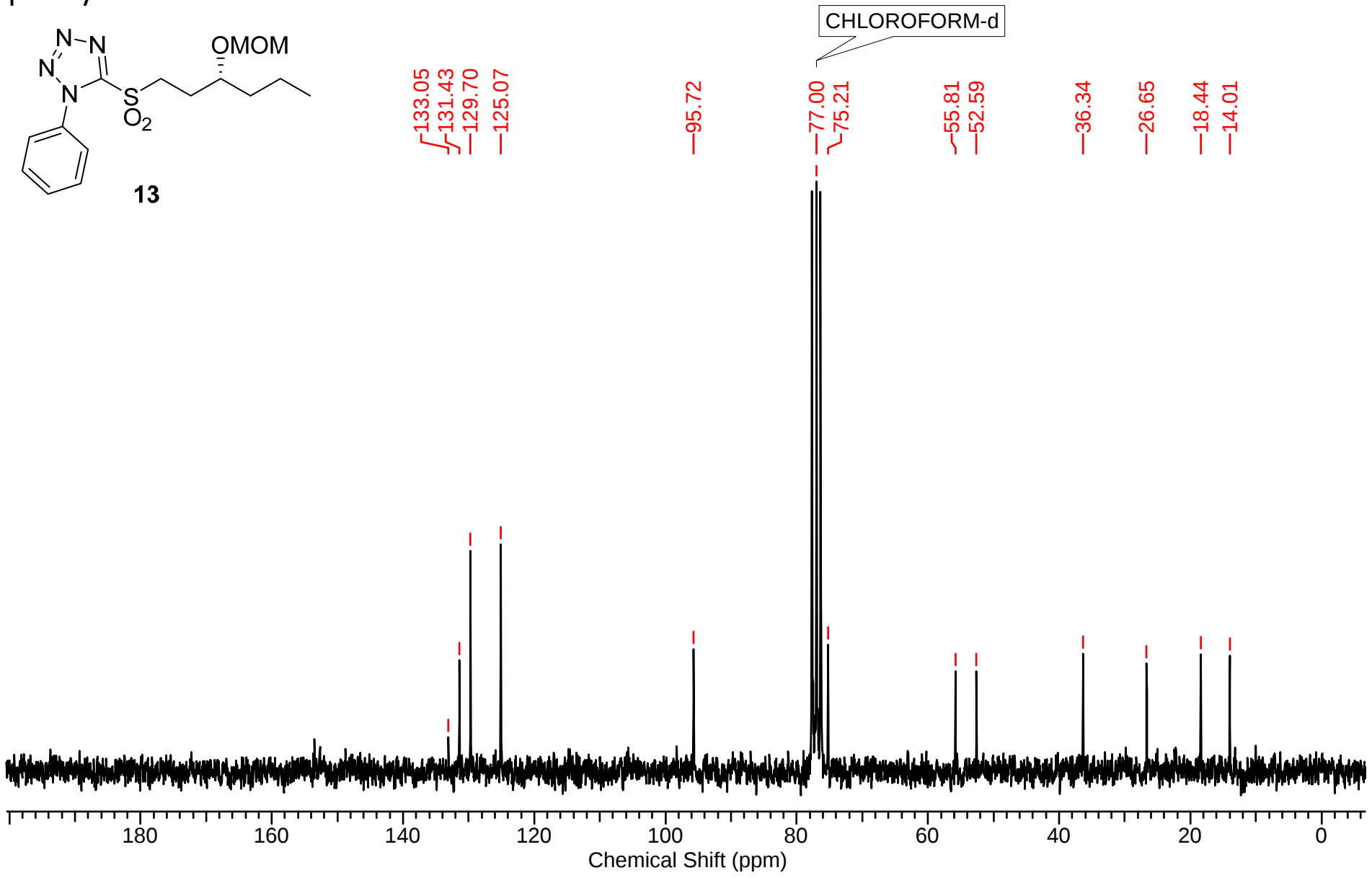
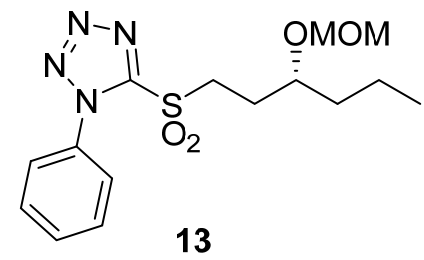
T: FTMS + p ESI Full ms [100.00-700.00]



¹H NMR (500MHz, CDCl₃) Of the compound (*R*)-5-((3-(methoxymethoxy)hexyl)sulfonyl)-1-phenyl-1H-tetrazole



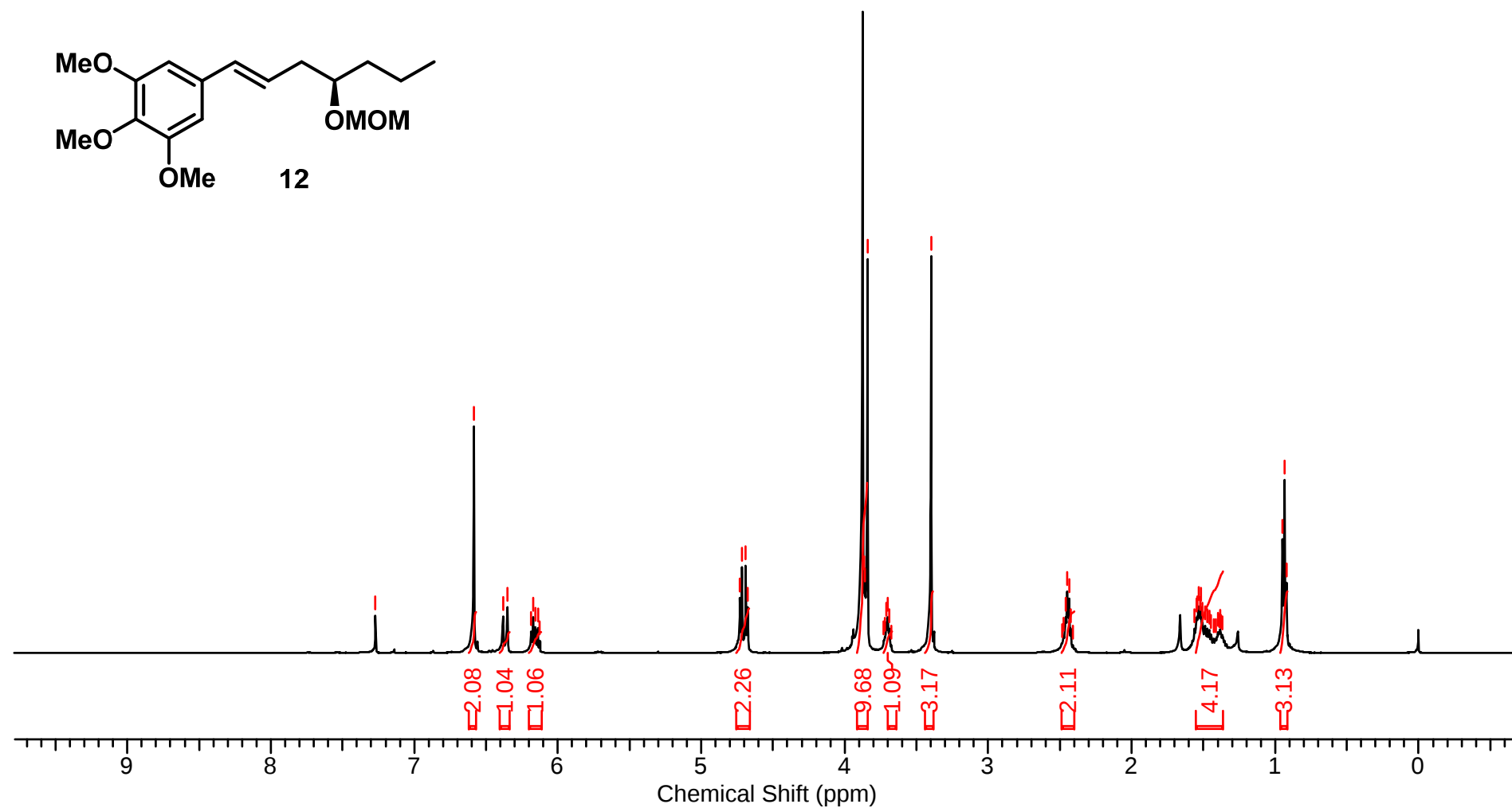
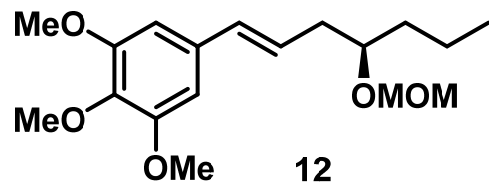
¹³C NMR (50MHz ,CDCl₃) Of the compound (R)-5-((3-(methoxymethoxy)hexyl)sulfonyl)-1-phenyl-1H-tetrazole



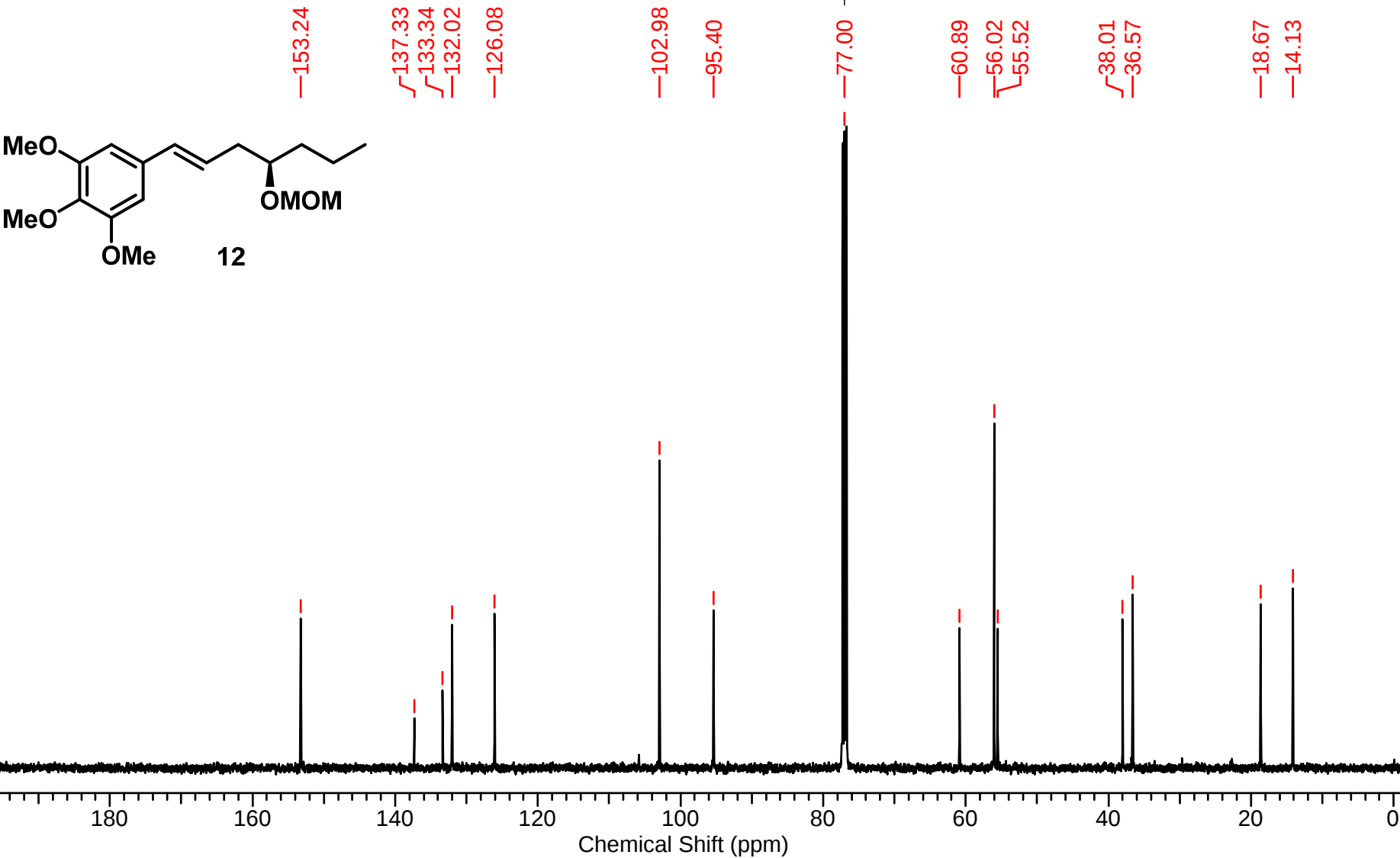
^1H NMR (500MHz, CDCl_3) Of the compound (*R,E*)-1,2,3-trimethoxy-5-(4-(methoxymethoxy)hept-1-en-1-yl)benzene

CHLOROFORM-d

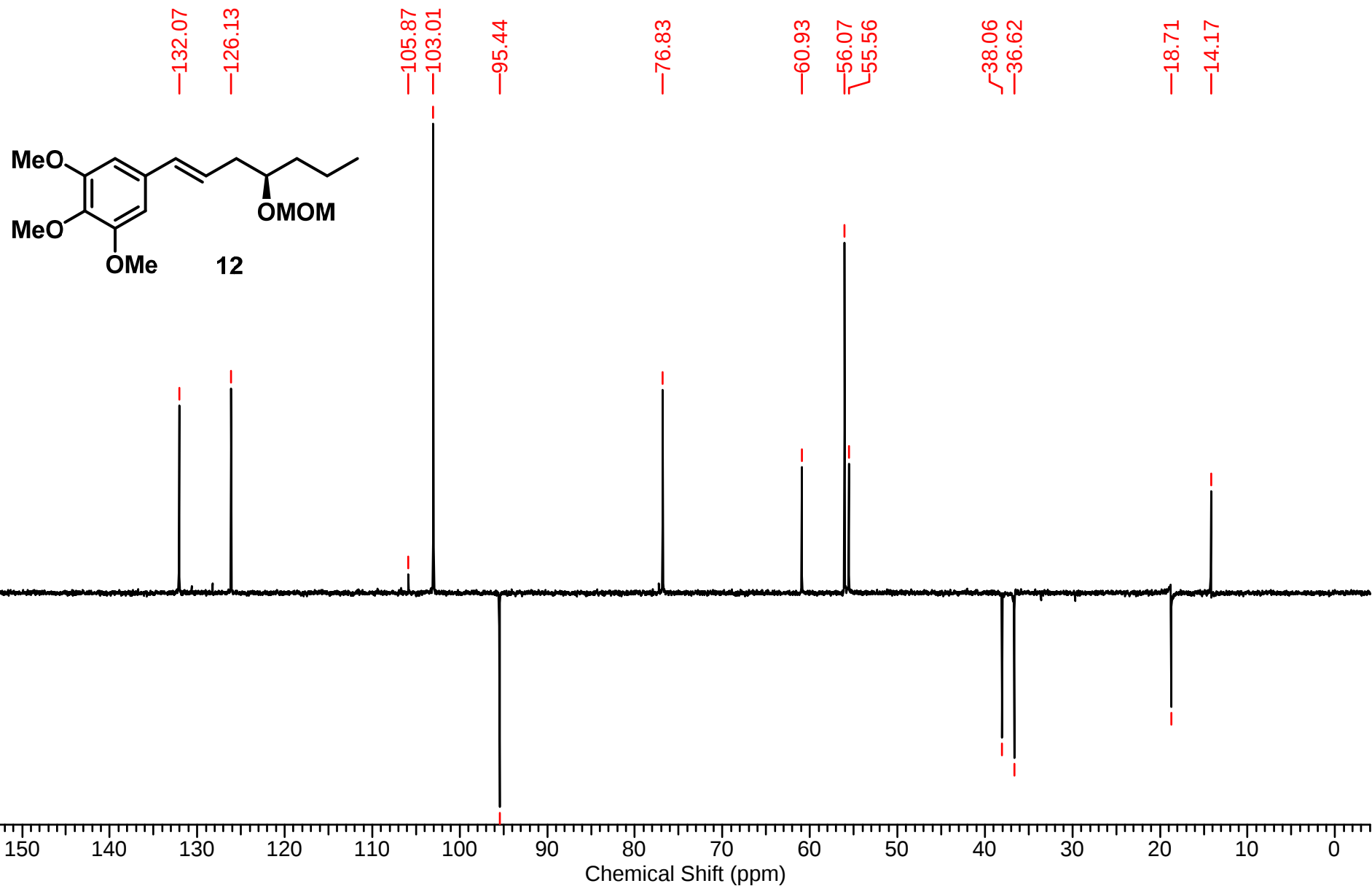
7.27, 6.58, 6.38, 6.35, 6.18, 6.17, 6.16, 6.14, 4.73, 4.72, 4.69, 4.67, 3.88, 3.86, 3.86, 3.84, 3.72, 3.71, 3.70, 3.40, 2.46, 2.45, 2.43, 2.42, 1.56, 1.55, 1.54, 1.53, 1.52, 1.52, 1.51, 1.49, 0.95, 0.93, 0.92



¹³C NMR (125MHz ,CDCl₃) Of the compound (*R,E*)-1,2,3-trimethoxy-5-(4-(methoxymethoxy)hept-1-en-1-yl)benzene

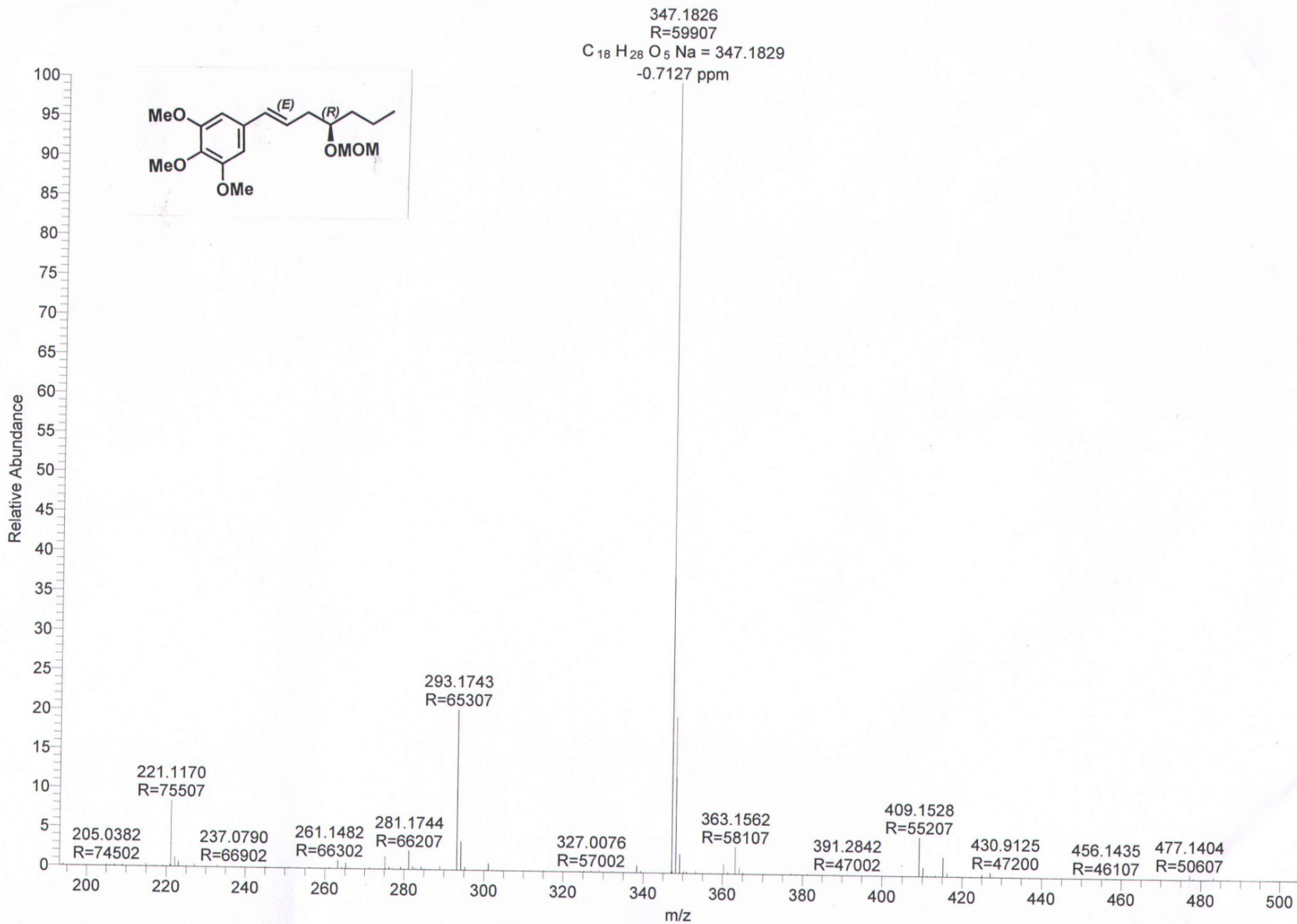


^{13}C NMR (125MHz, CDCl_3) Of the compound (*R,E*)-1,2,3-trimethoxy-5-(4-(methoxymethoxy)hept-1-en-1-yl)benzene

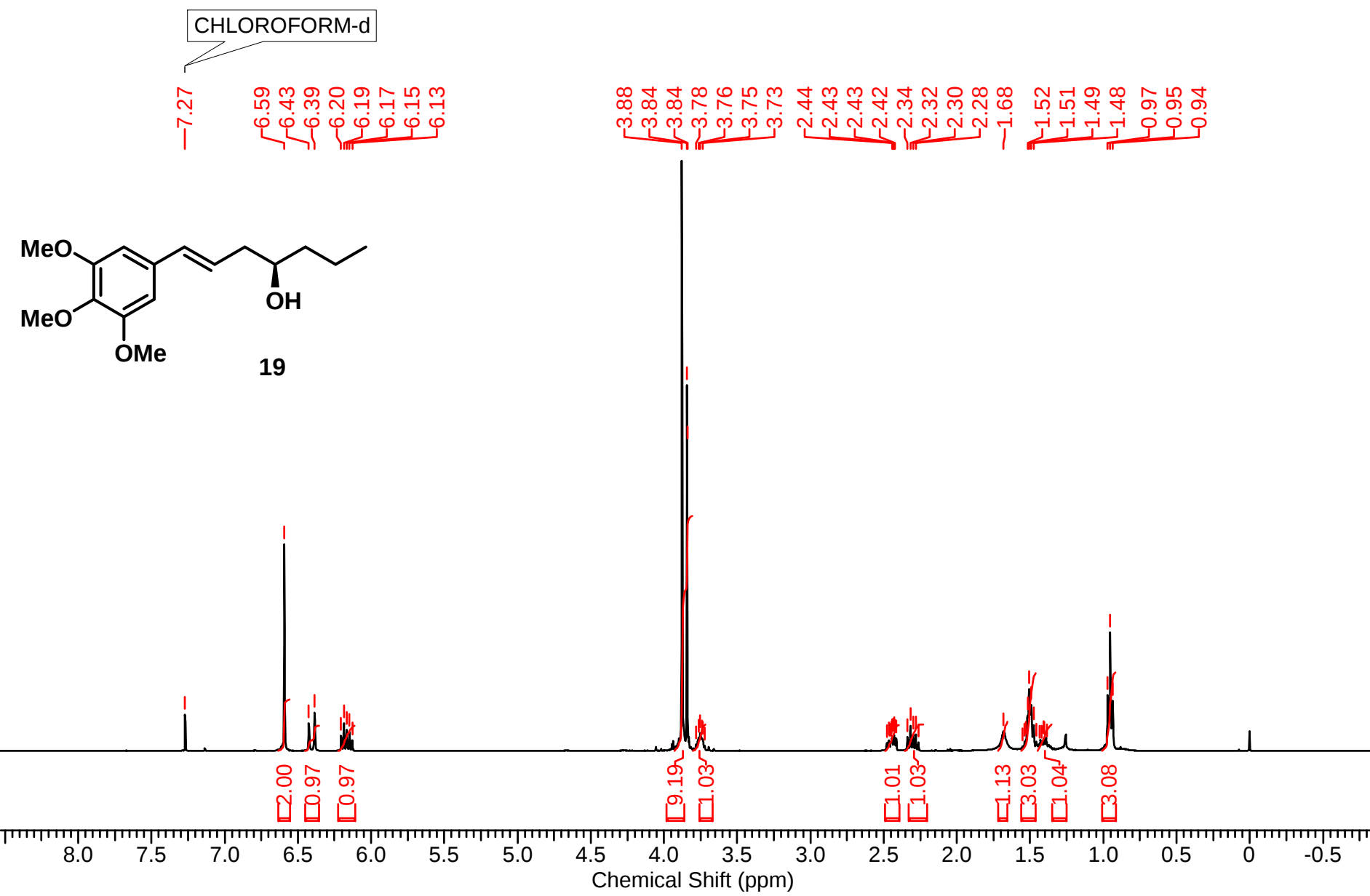


UNR-A-7 #1162 RT: 5.21 AV: 1 NL: 9.20E8

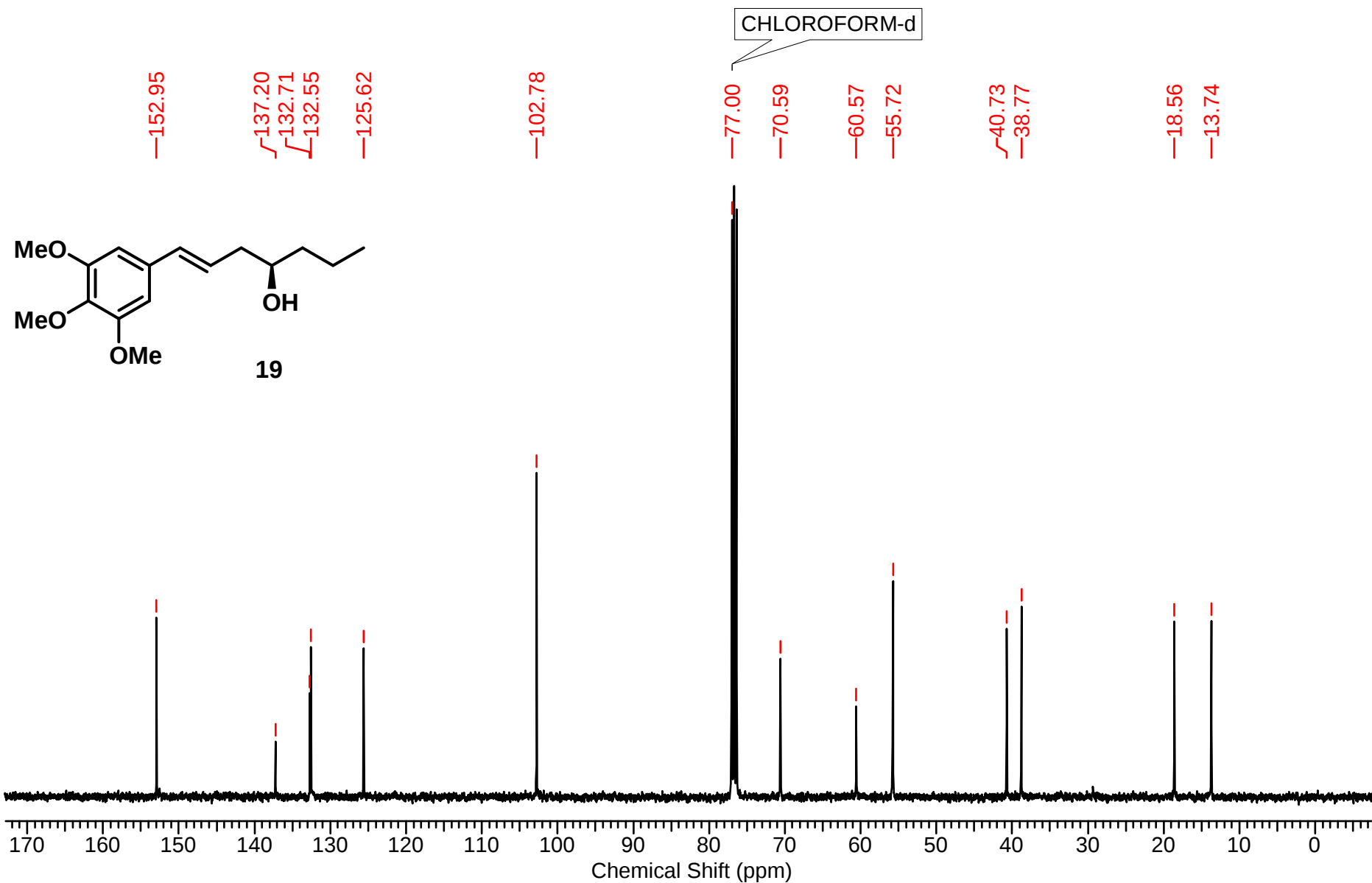
T: FTMS + p ESI Full ms [100.00-700.00]



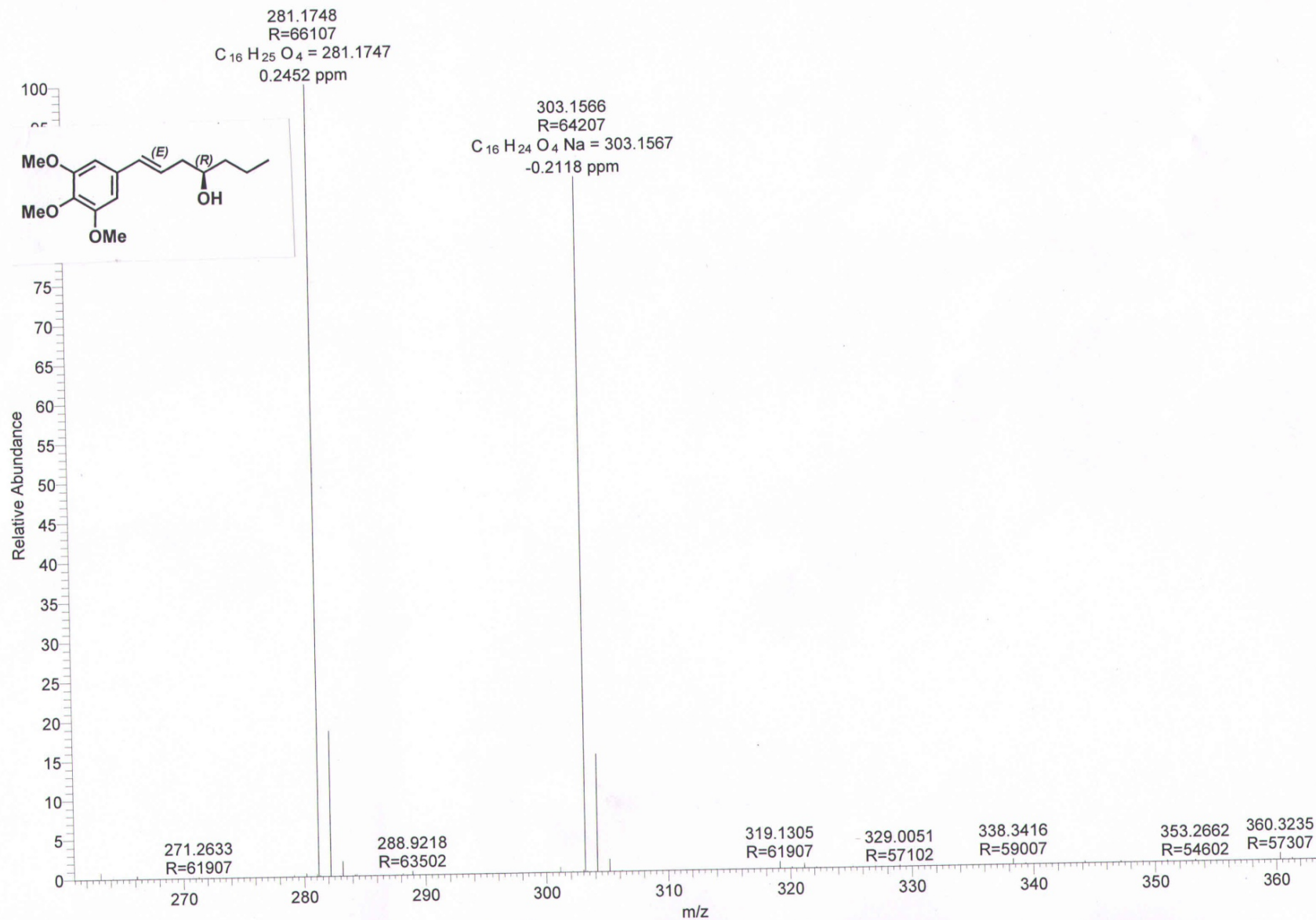
¹H NMR (400MHz ,CDCl₃) Of the compound (R,E)-1-(3,4,5-trimethoxyphenyl)hept-1-en-4-ol



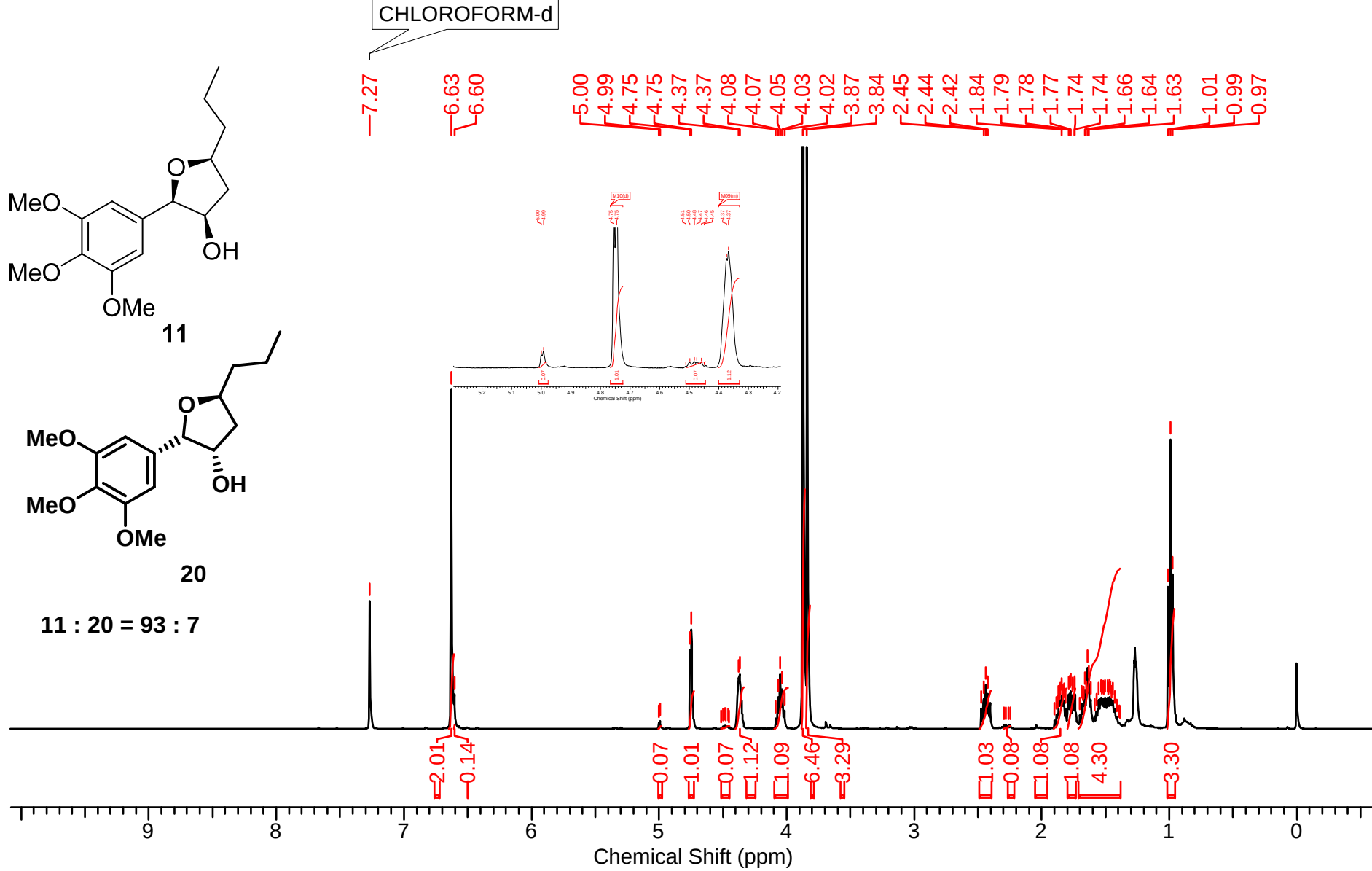
^{13}C NMR (100MHz, CDCl_3) Of the compound (*R,E*)-1-(3,4,5-trimethoxyphenyl)hept-1-en-4-ol



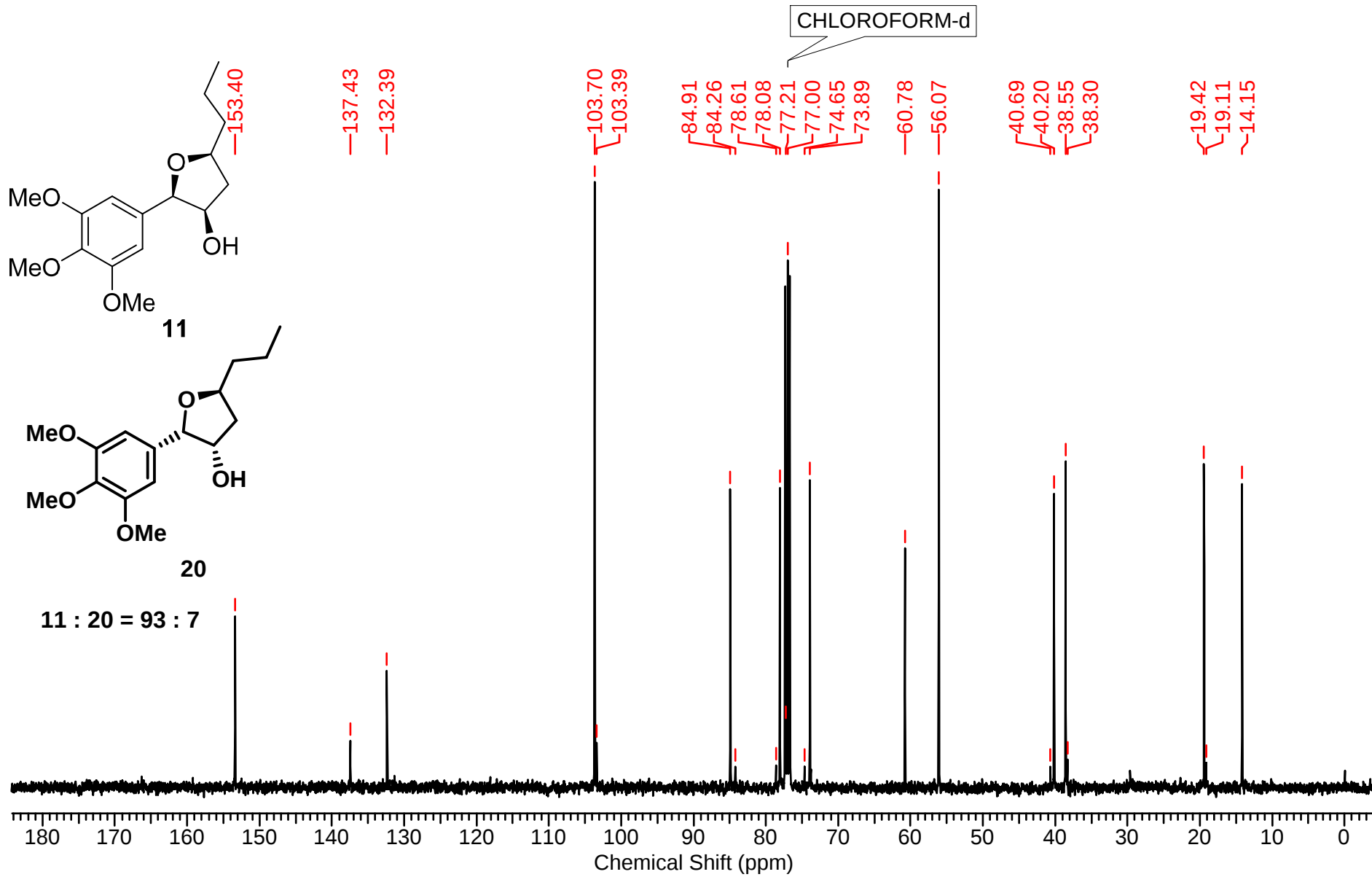
UNR-A-8 #977 RT: 4.38 AV: 1 NL: 8.63E8
T: FTMS + p ESI Full ms [100.00-700.00]



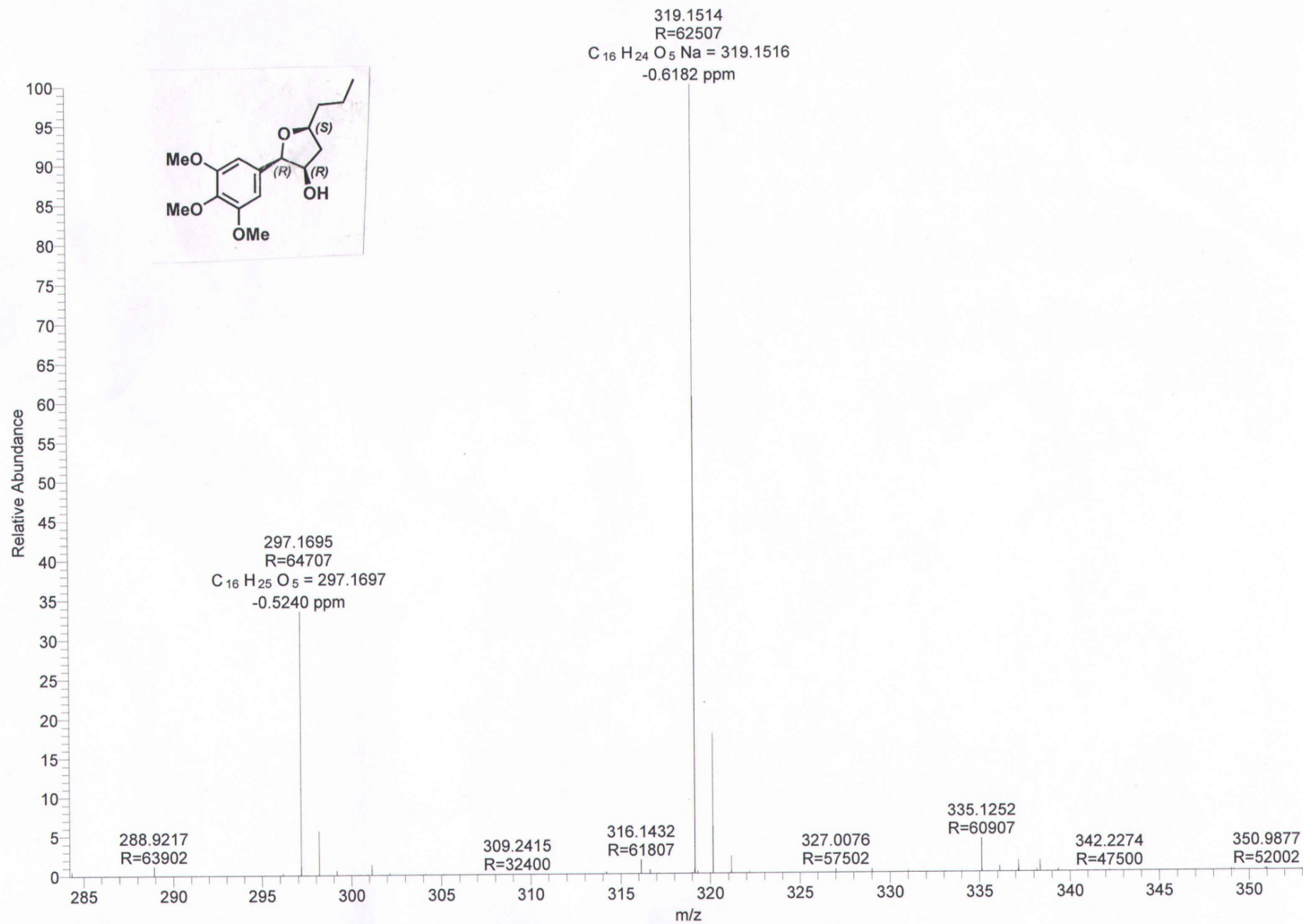
¹H NMR (400MHz ,CDCl₃) Of the compound (2R,3R,5S)-5-propyl-2-(3,4,5-trimethoxyphenyl)tetrahydrofuran-3-ol



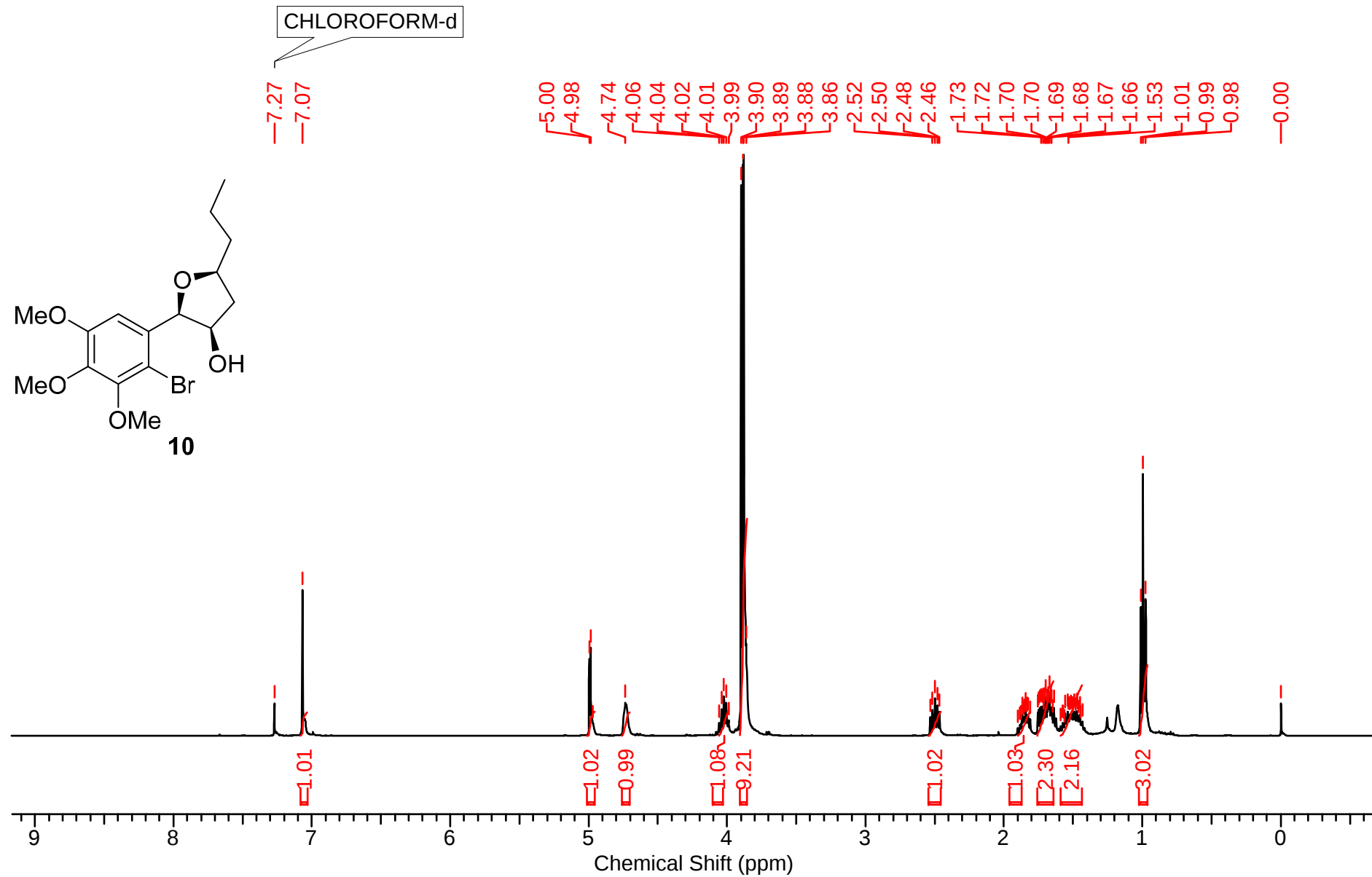
¹³C NMR (100MHz ,CDCl₃) Of the compound (2*R*,3*R*,5*S*)-5-propyl-2-(3,4,5-trimethoxyphenyl)tetrahydrofuran-3-ol



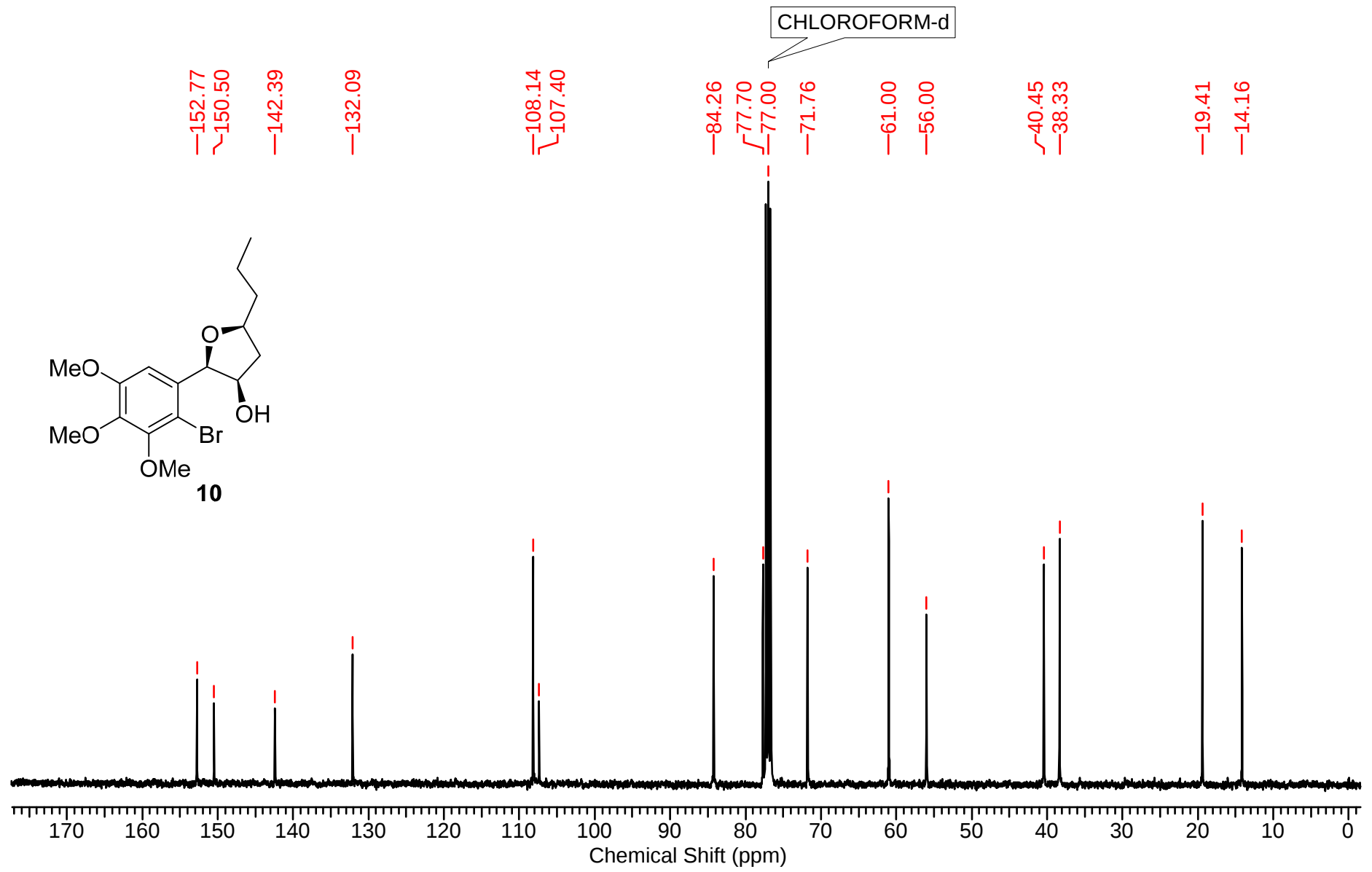
UNR-A-9 #917 RT: 4.11 AV: 1 NL: 3.35E8
T: FTMS + p ESI Full ms [100.00-700.00]



¹H NMR (400MHz ,CDCl₃) Of the compound (2*R*,3*R*,5*S*)-2-(2-bromo-3,4,5-trimethoxyphenyl)-5-propyltetrahydrofuran-3-ol

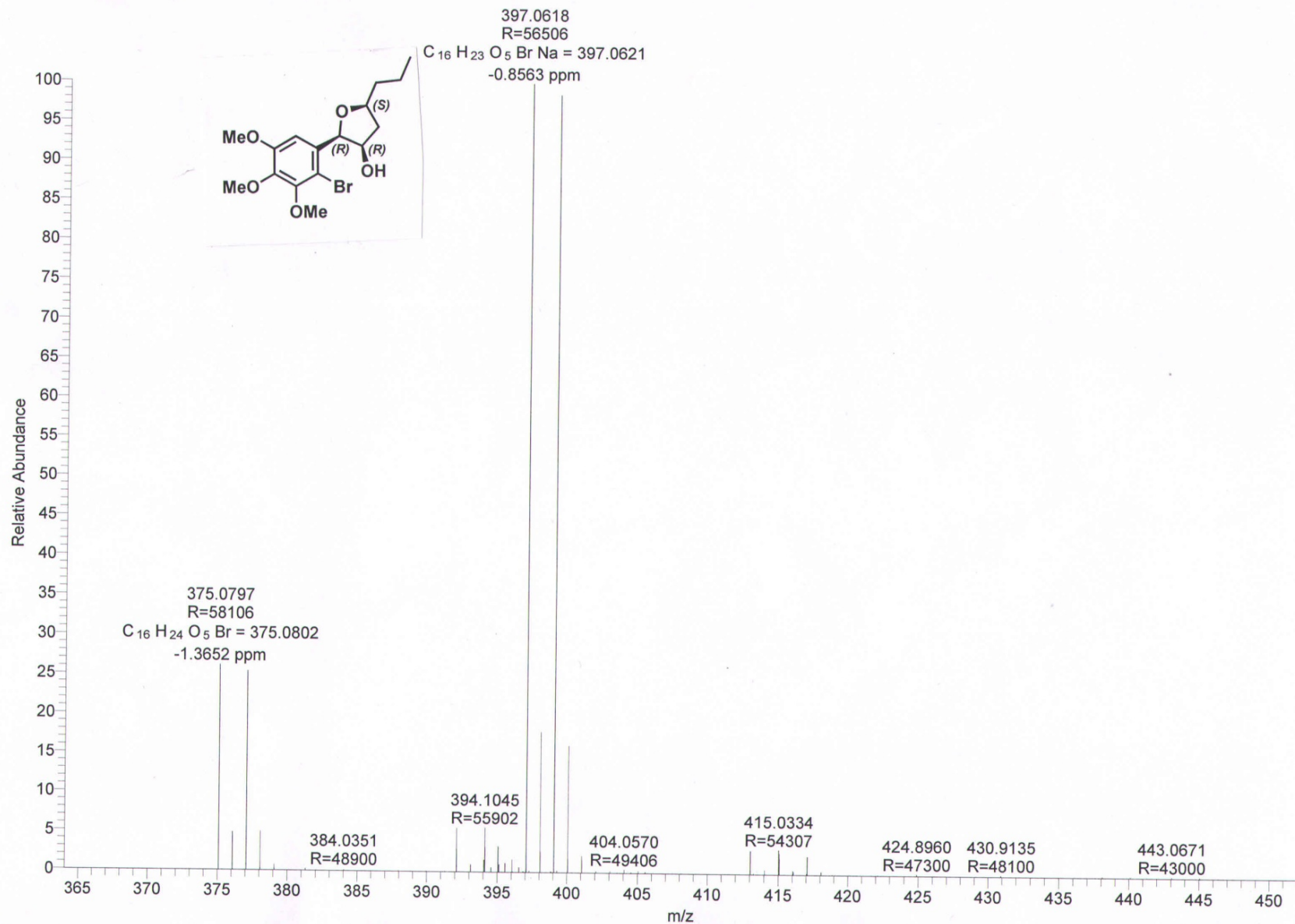


¹³C NMR (100MHz ,CDCl₃) Of the compound (2*R*,3*R*,5*S*)-2-(2-bromo-3,4,5-trimethoxyphenyl)-5-propyltetrahydrofuran-3-ol

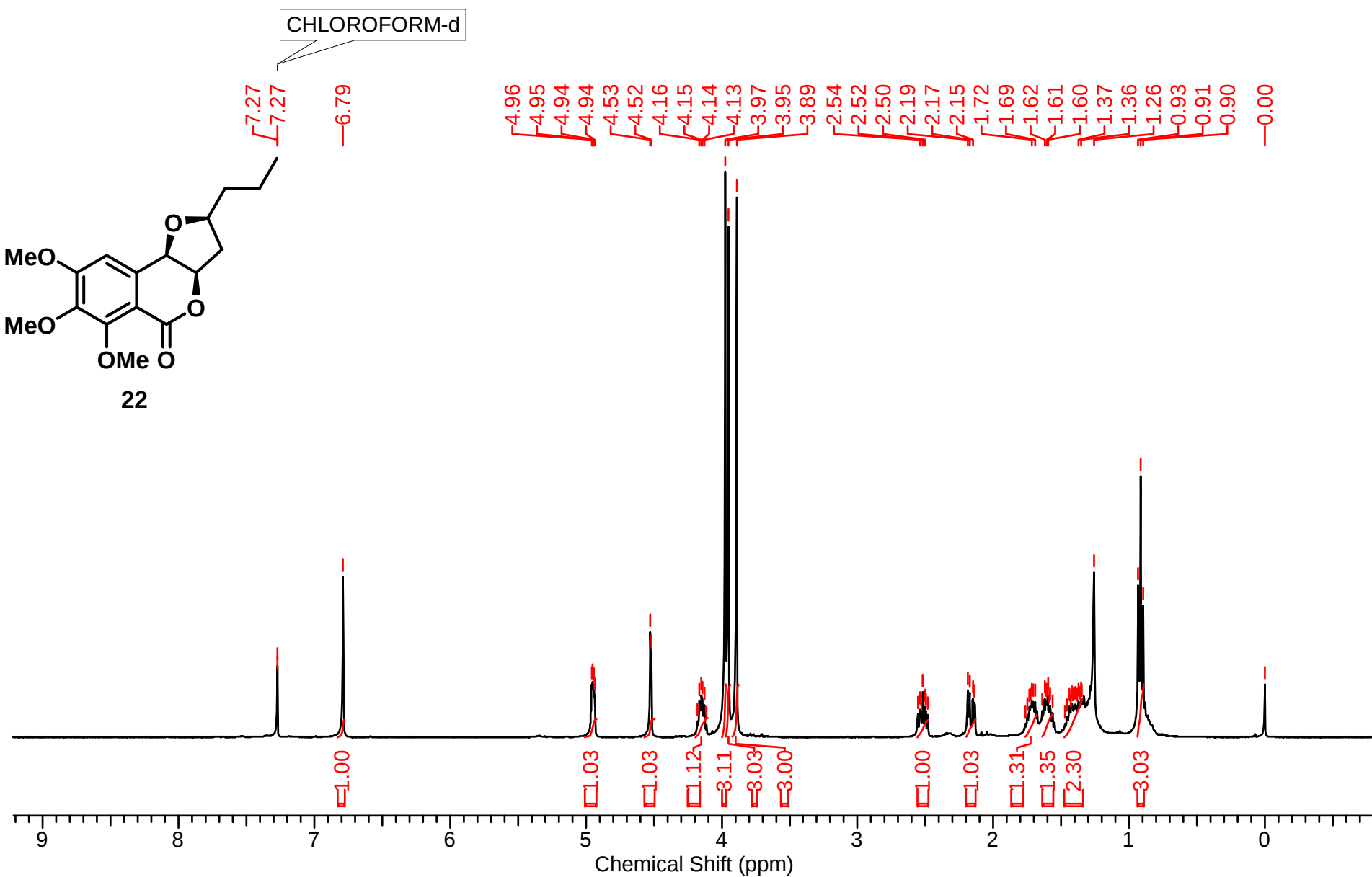


UNR-A-10 #1099 RT: 4.92 AV: 1 NL: 5.87E8

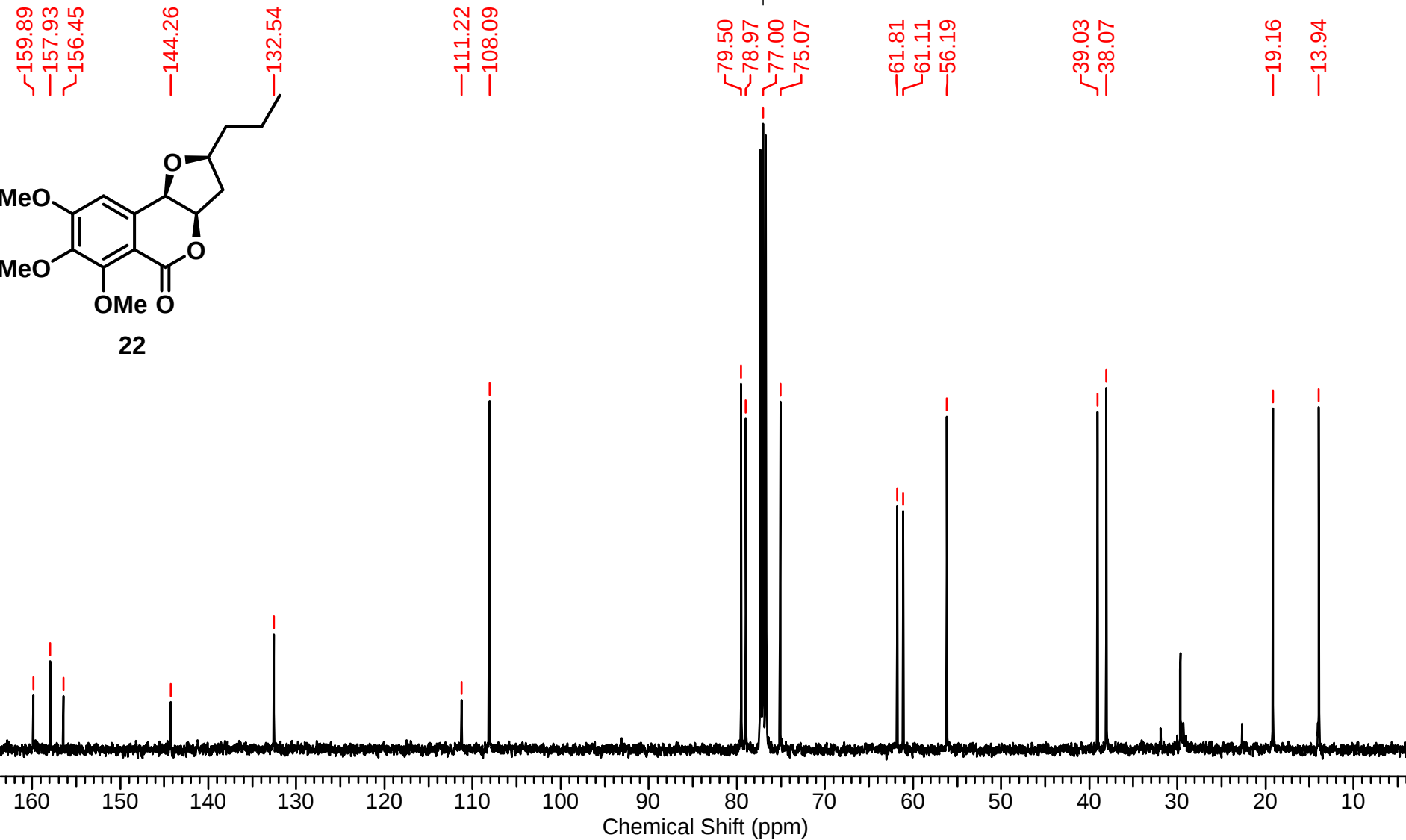
T: FTMS + p ESI Full ms [100.00-700.00]



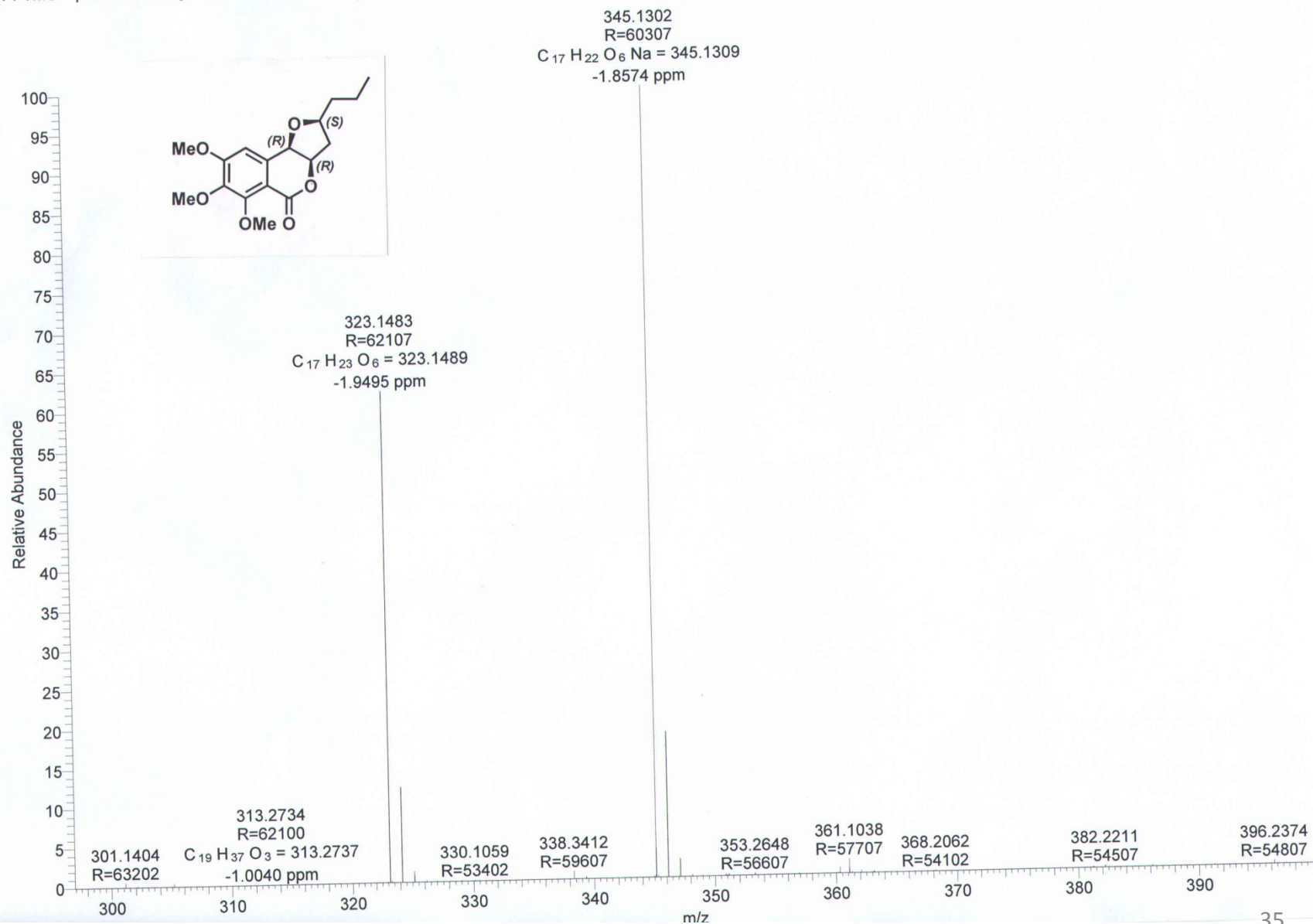
^1H NMR (400MHz, CDCl_3) Of the compound (2*S*,3*aR*,9*bR*)-6,7,8-trimethoxy-2-propyl-2,3,3*a*,9*b*-tetrahydro-5*H*-furo[3,2-*c*]isochromen-5-one



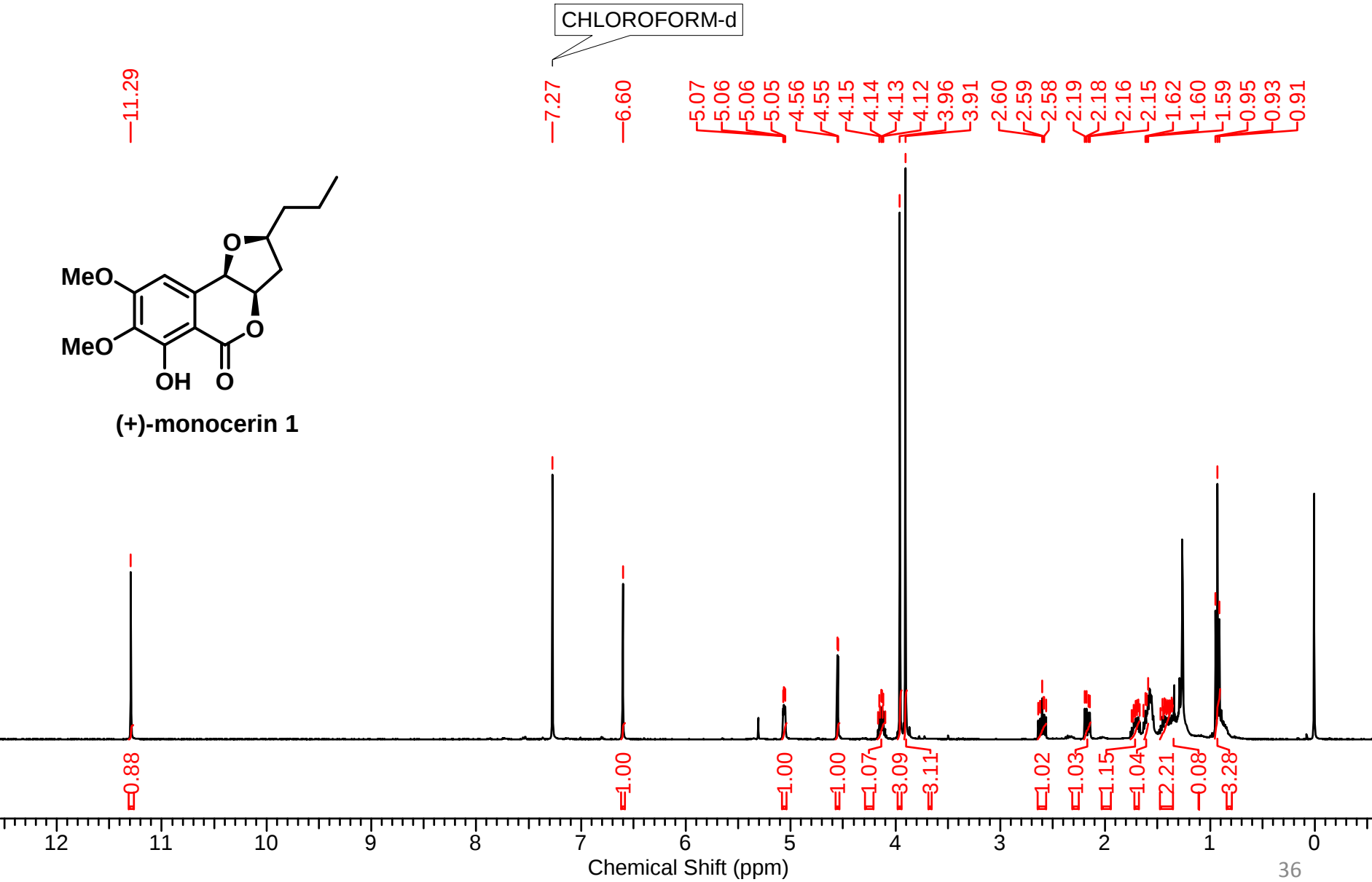
¹³C NMR (100MHz ,CDCl₃) Of the compound(2*S*,3*aR*,9*bR*)-6,7,8-trimethoxy-2-propyl-2,3,3*a*,9*b*-tetrahydro-5*H*-furo[3,2-*c*]isochromen-5-one



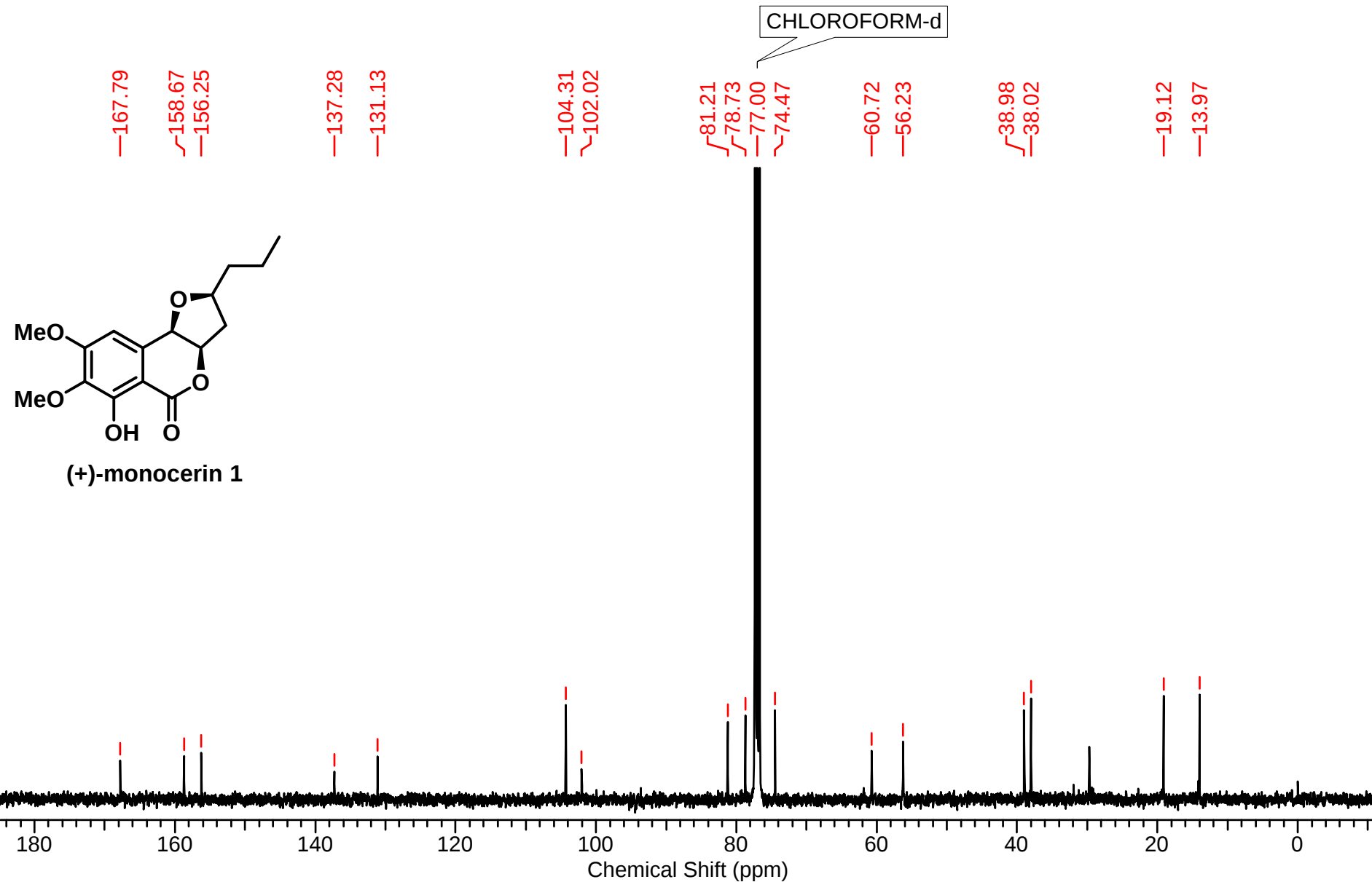
UNR-A-11 #902 RT: 4.05 AV: 1 NL: 7.61E8
T: FTMS + p ESI Full ms [100.00-700.00]



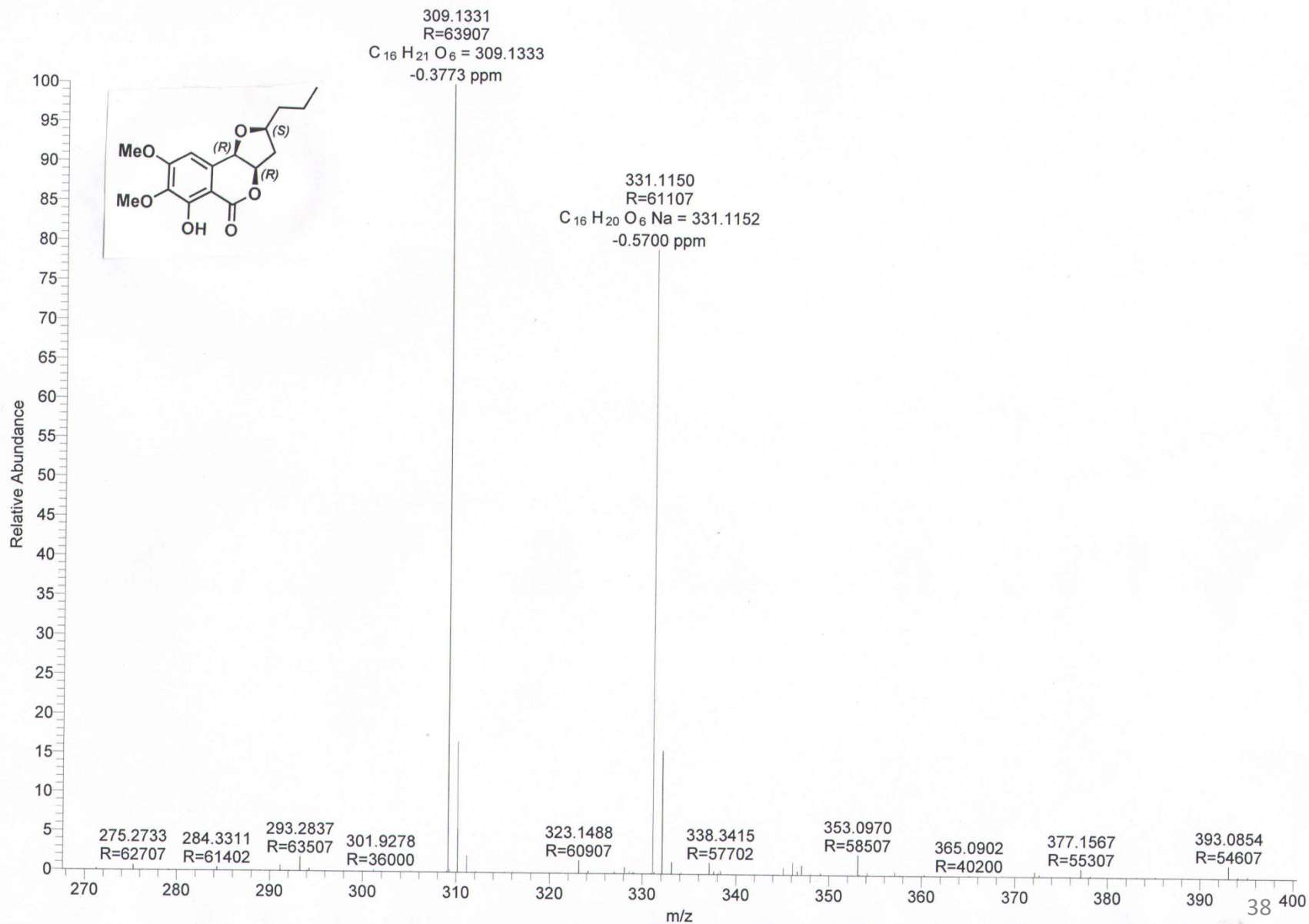
¹H NMR(400 MHz,CDCl₃) of the compound (2S,3aR,9bR)-6-hydroxy-7,8-dimethoxy-2-propyl-2,3,3a,9b-tetrahydro-5H-furo[3,2-c]isochromen-5-one :



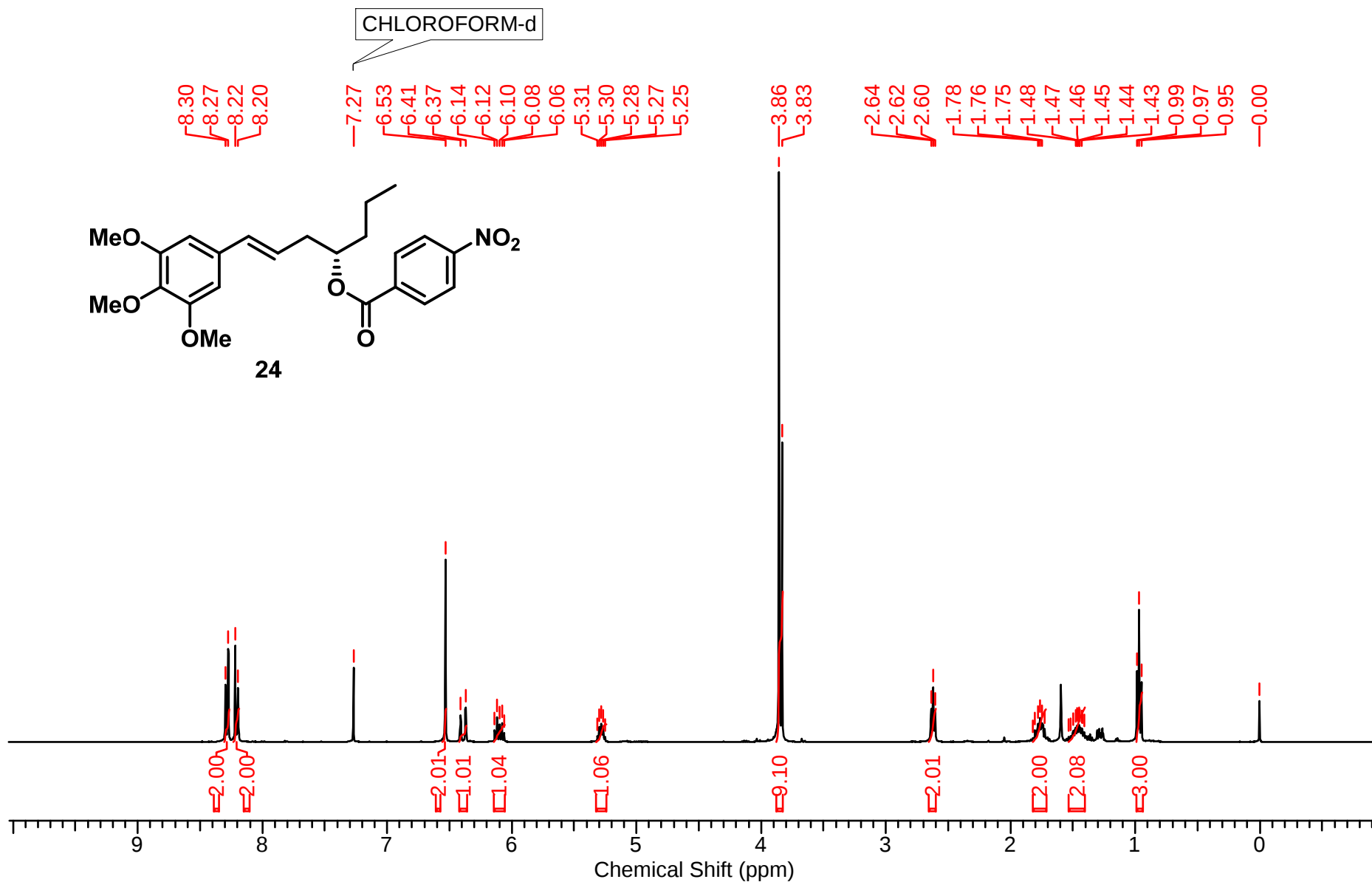
¹³CNMR(400 MHz,CDCl₃) of the compound (2S,3aR,9bR)-6-hydroxy-7,8-dimethoxy-2-propyl-2,3,3a,9b-tetrahydro-5H-furo[3,2-c]isochromen-5-one :



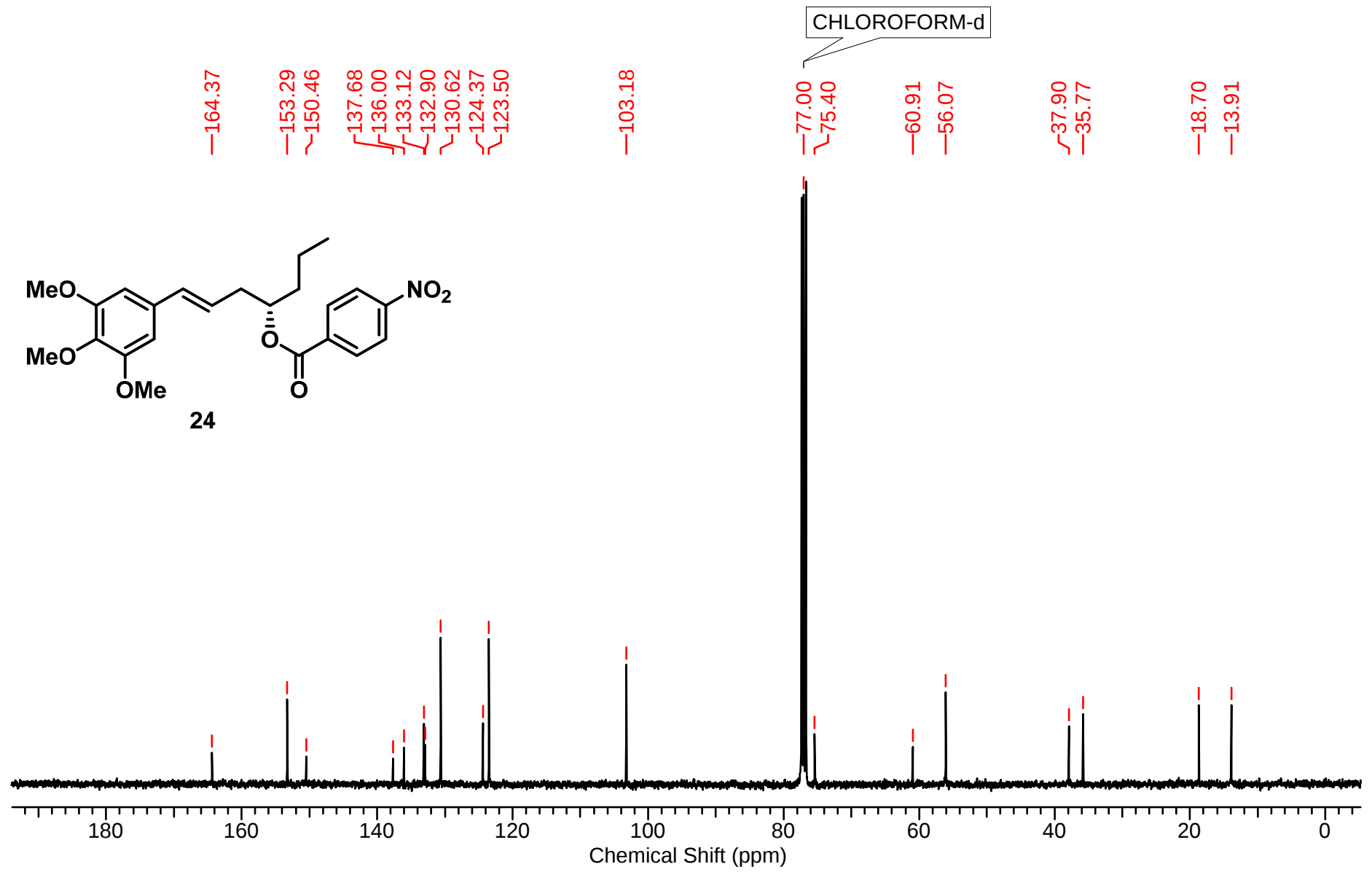
RRS-M #1112 RT: 4.96 AV: 1 NL: 4.02E8
T: FTMS + p ESI Full ms [100.00-700.00]



^1H NMR (400MHz, CDCl_3) Of the compound (*S,E*)-1-(3,4,5-trimethoxyphenyl)hept-1-en-4-yl 4-nitrobenzoate :

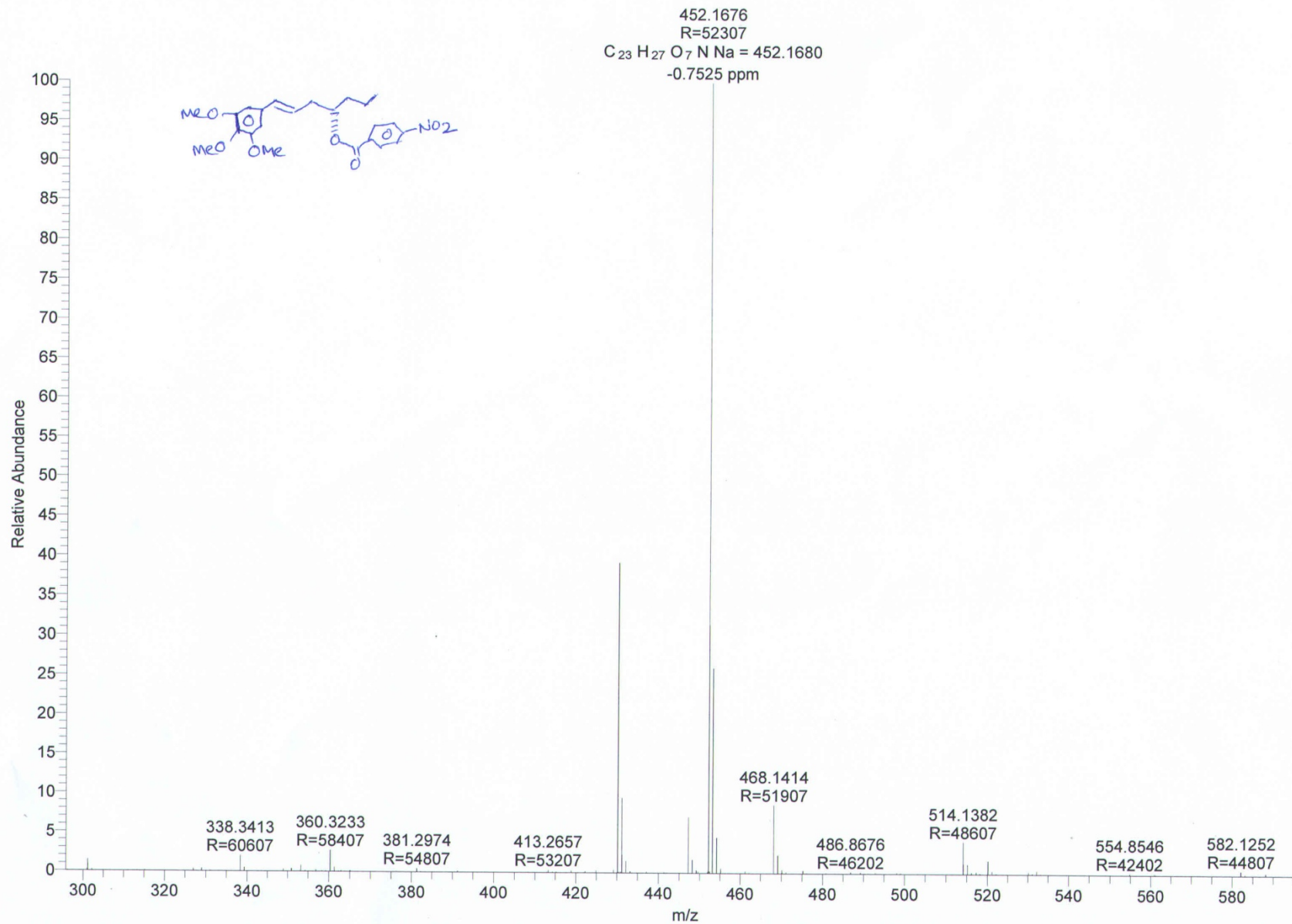


¹³C NMR (100MHz ,CDCl₃) Of the compound (S,E)-1-(3,4,5-trimethoxyphenyl)hept-1-en-4-yl 4-nitrobenzoate



UNR-A-13 #1401 RT: 6.28 AV: 1 NL: 3.58E8

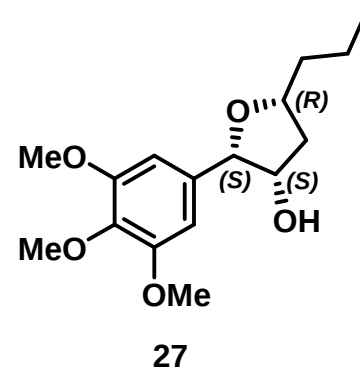
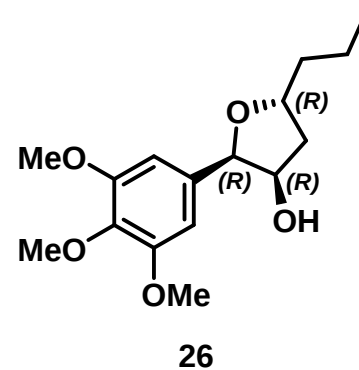
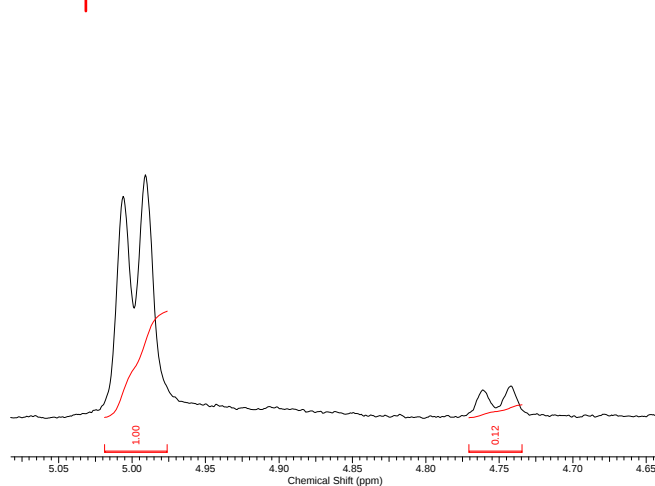
T: FTMS + p ESI Full ms [100.00-700.00]



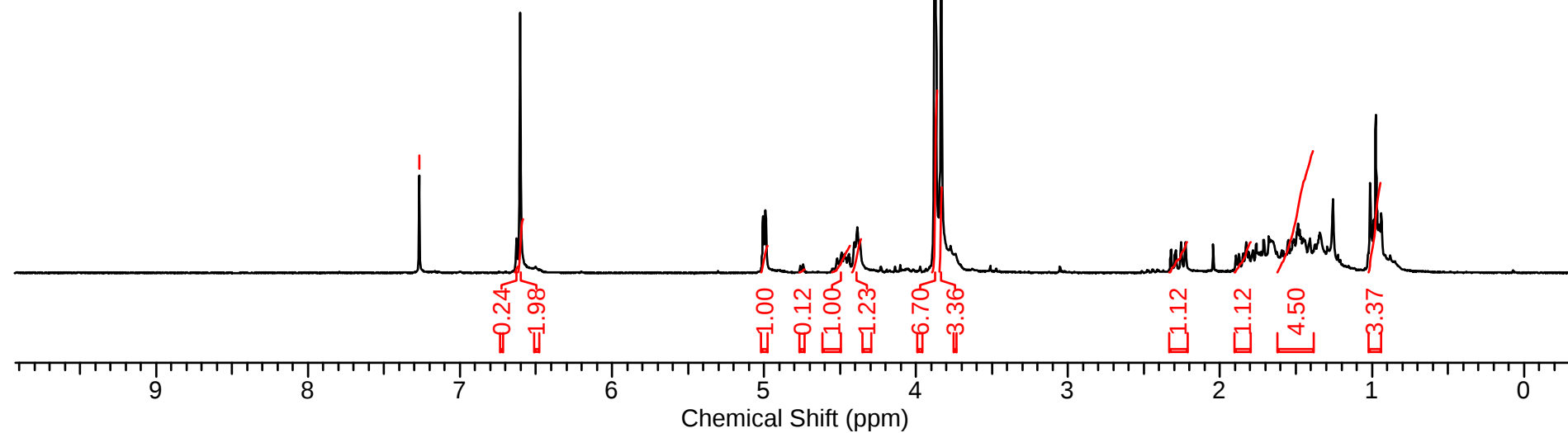
^1H NMR (200 MHz, CDCl_3) of the compound (2*R*,3*R*,5*R*)-5-propyl-2-(3,4,5-trimethoxyphenyl)tetrahydrofuran-3-ol

CHLOROFORM-d

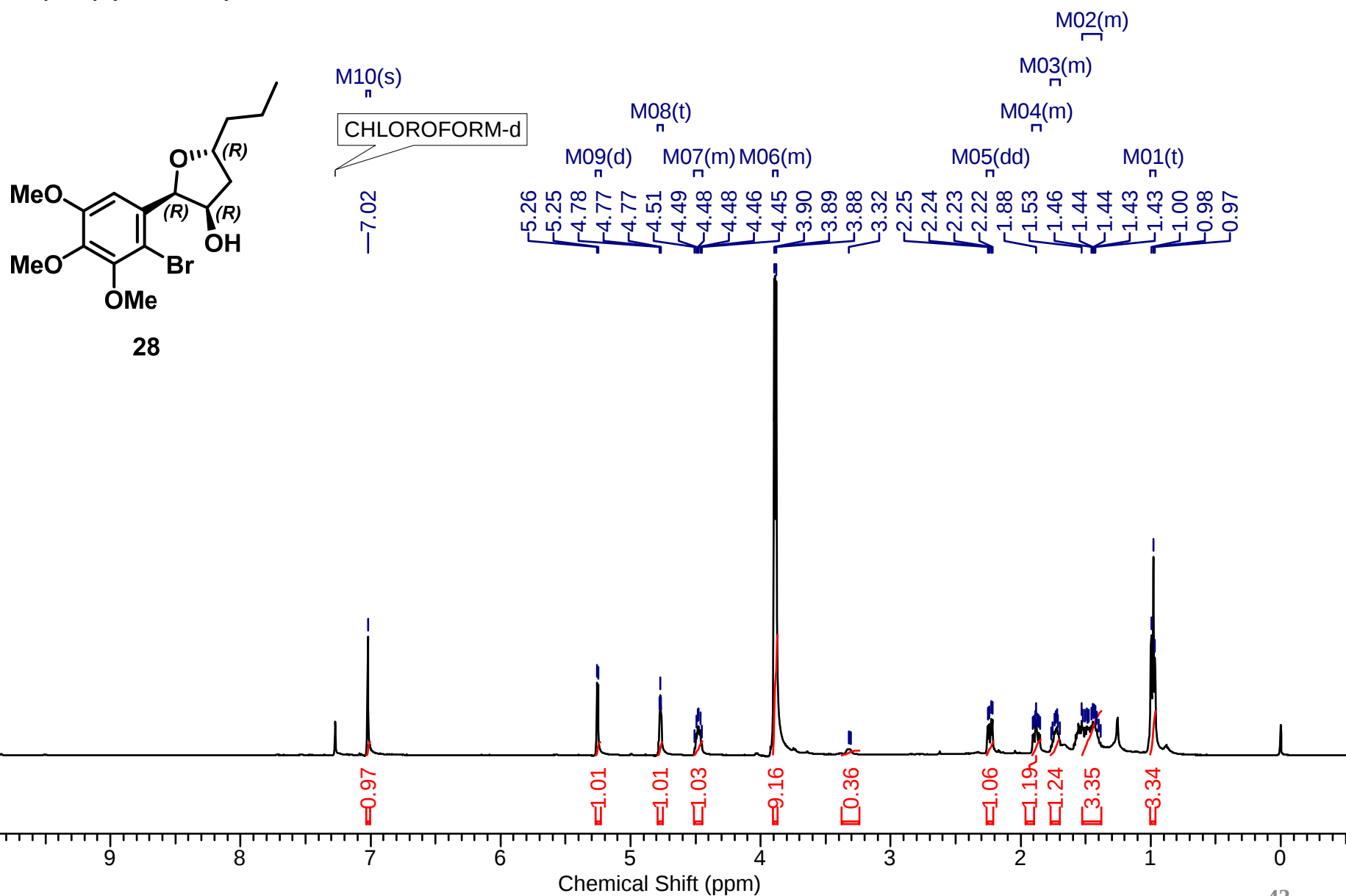
-7.27



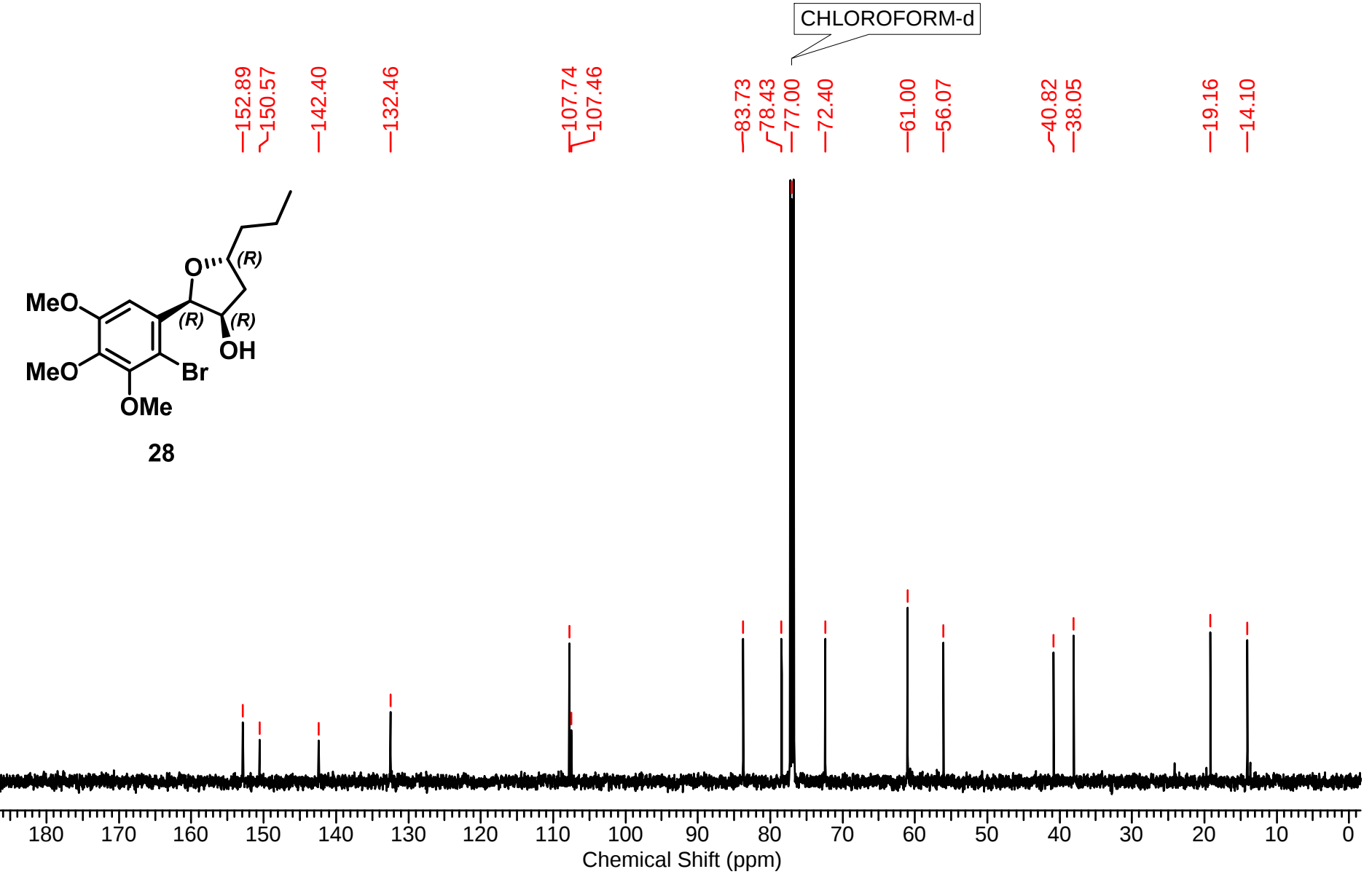
26:27 = 88:12



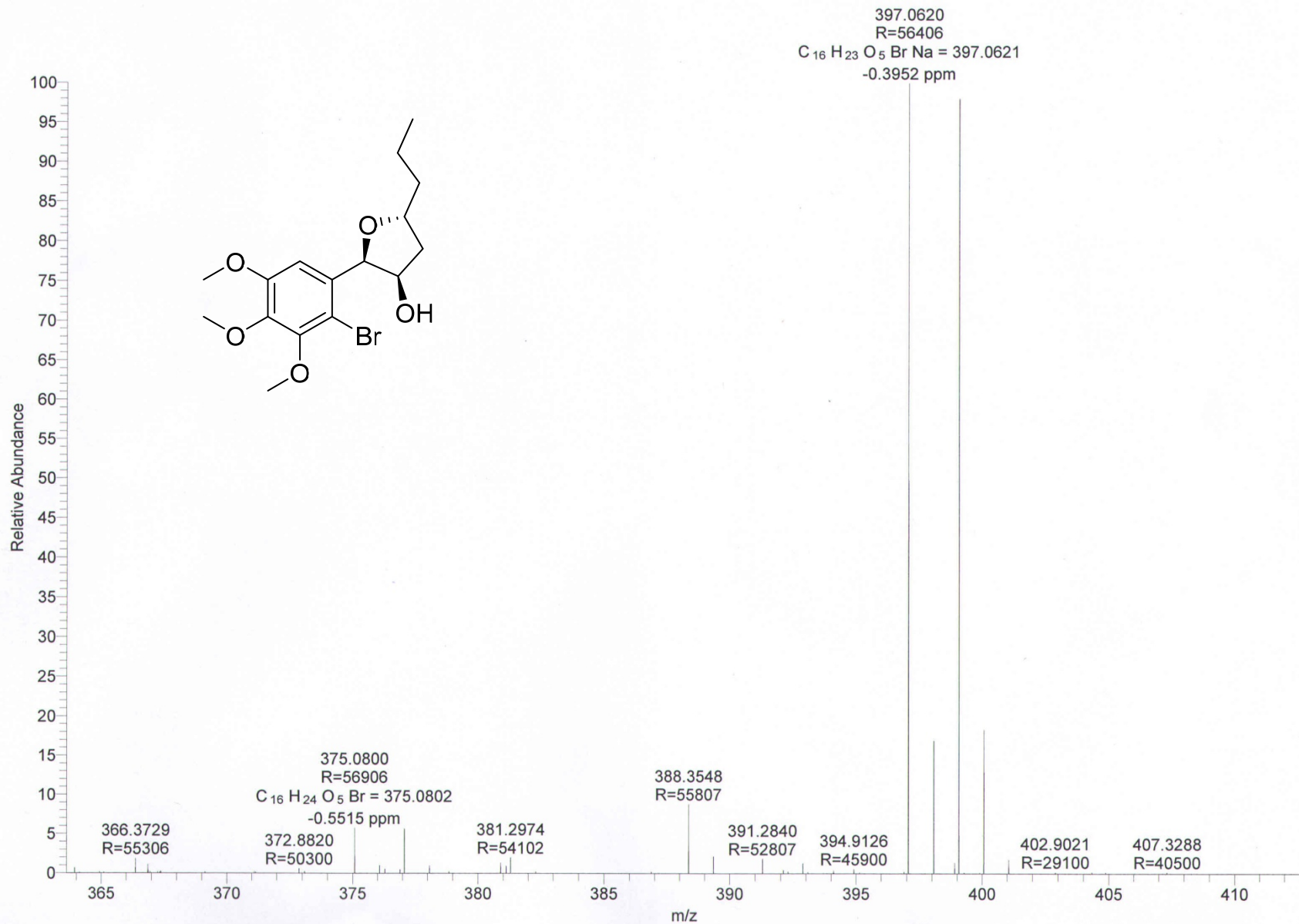
^1H NMR (400 MHz, CDCl_3) Of the compound (2*R*,3*R*,5*R*)-2-(2-bromo-3,4,5-trimethoxyphenyl)-5-propyltetrahydrofuran-3-ol



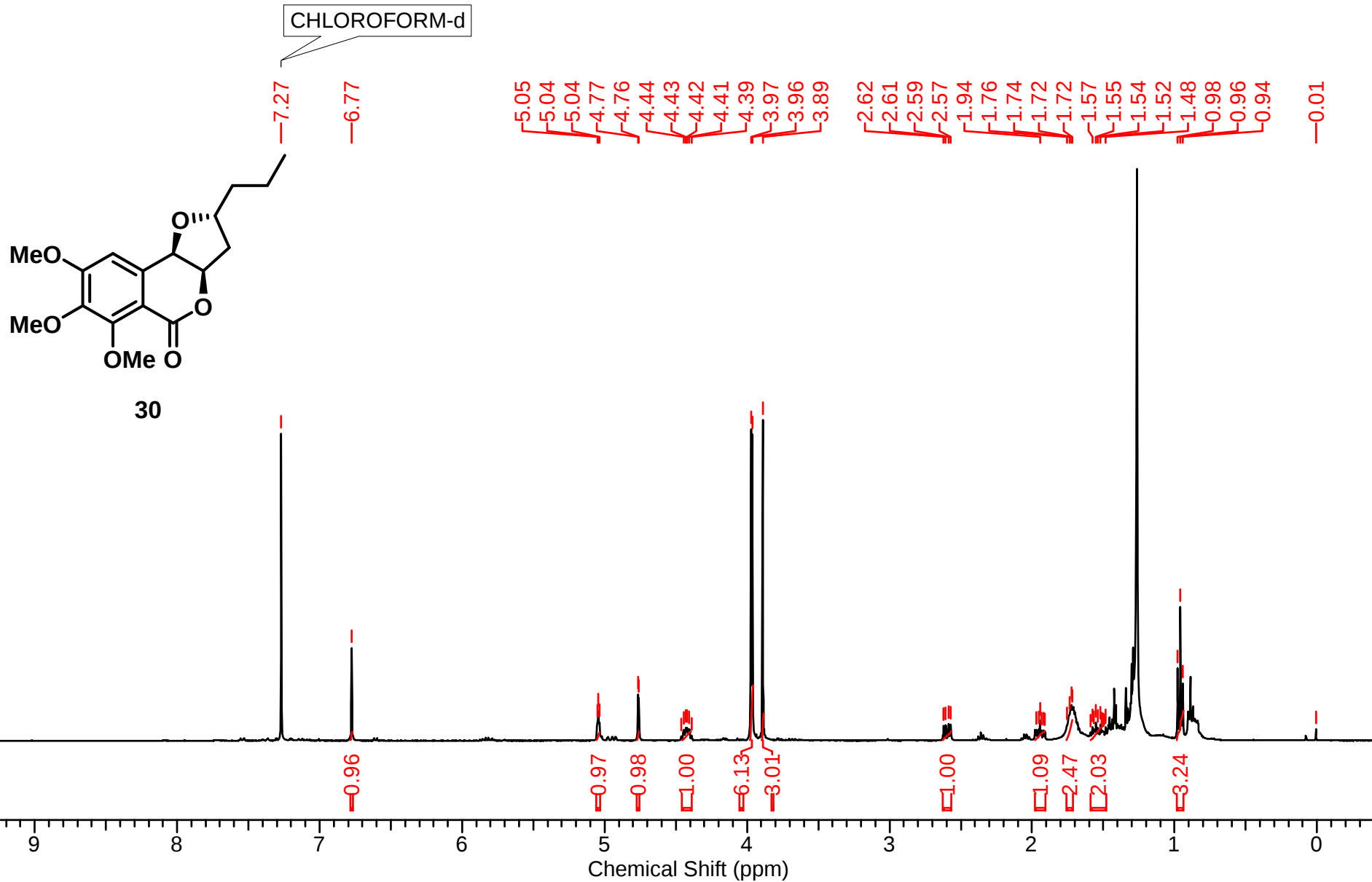
¹³C NMR (100 MHz, CDCl₃) Of the compound (2*R*,3*R*,5*R*)-2-(2-bromo-3,4,5-trimethoxyphenyl)-5-propyltetrahydrofuran-3-ol



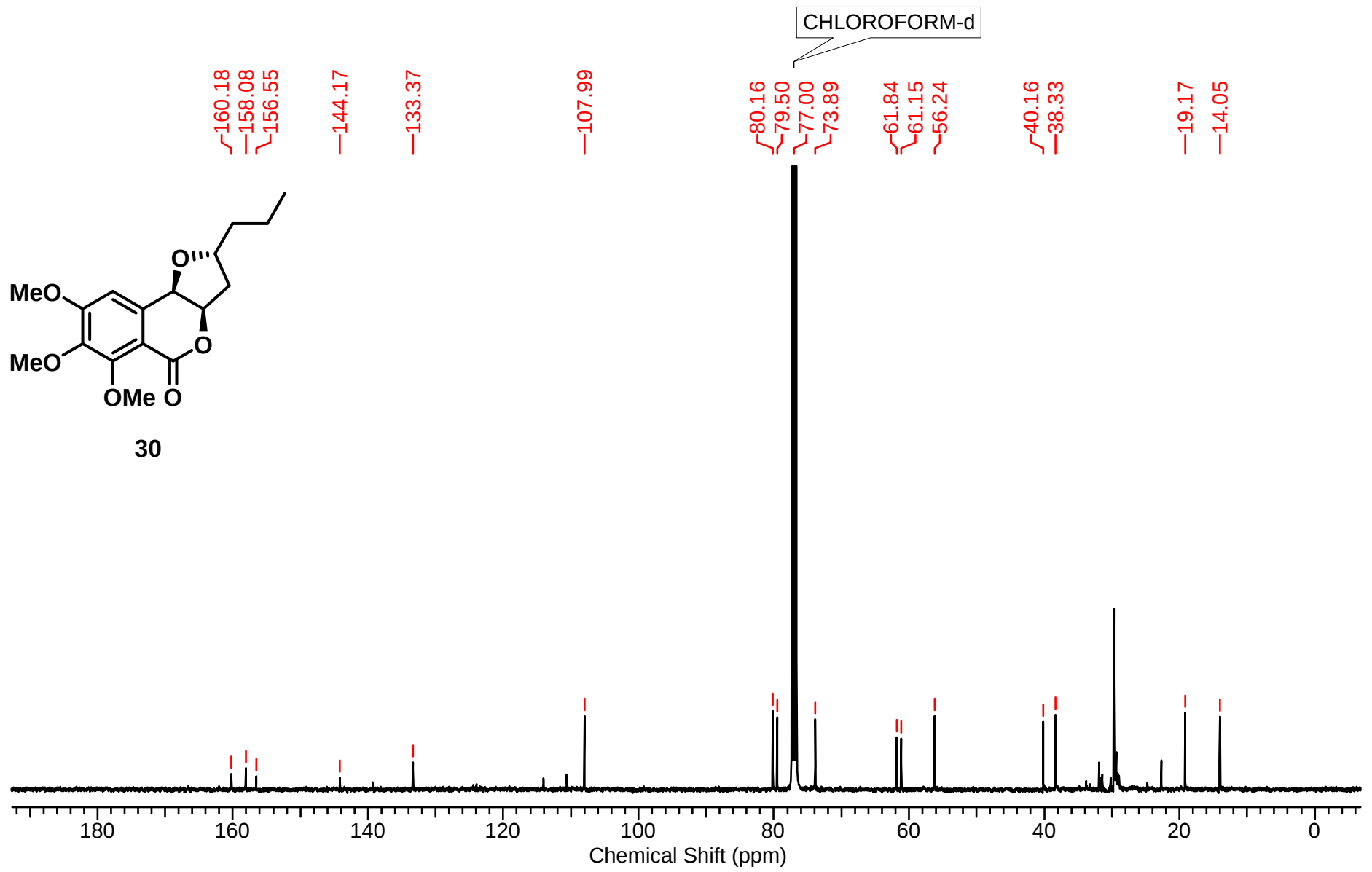
RRR-BROMO #1034 RT: 4.63 AV: 1 NL: 1.72E7
T: FTMS + p ESI Full ms [100.00-700.00]



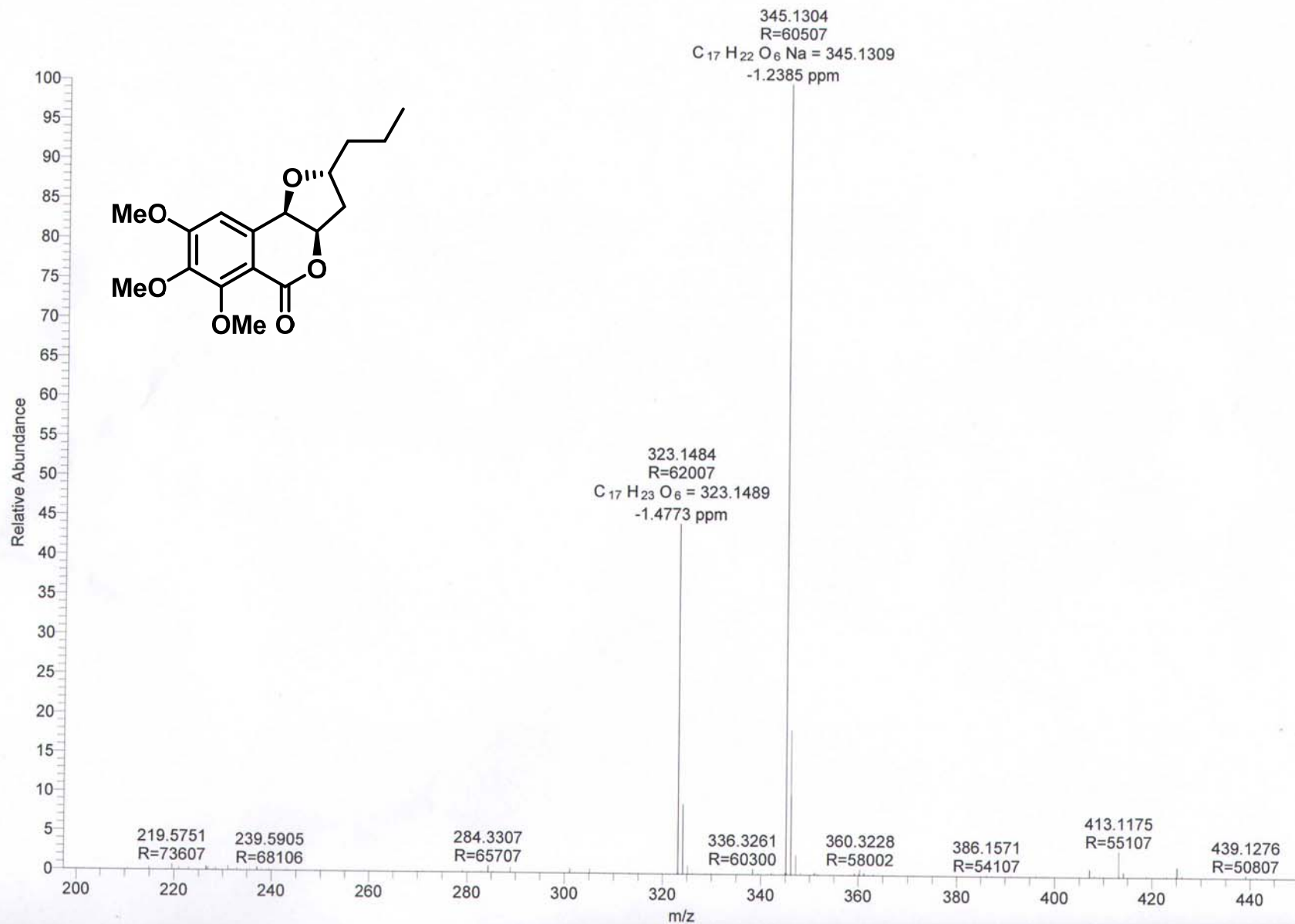
¹H NMR (400 MHz, CDCl₃) Of the compound (2*R*,3*aR*,9*bR*)-6,7,8-trimethoxy-2-propyl-2,3,3*a*,9*b*-tetrahydro-5*H*-furo[3,2-*c*]isochromen-5-one :



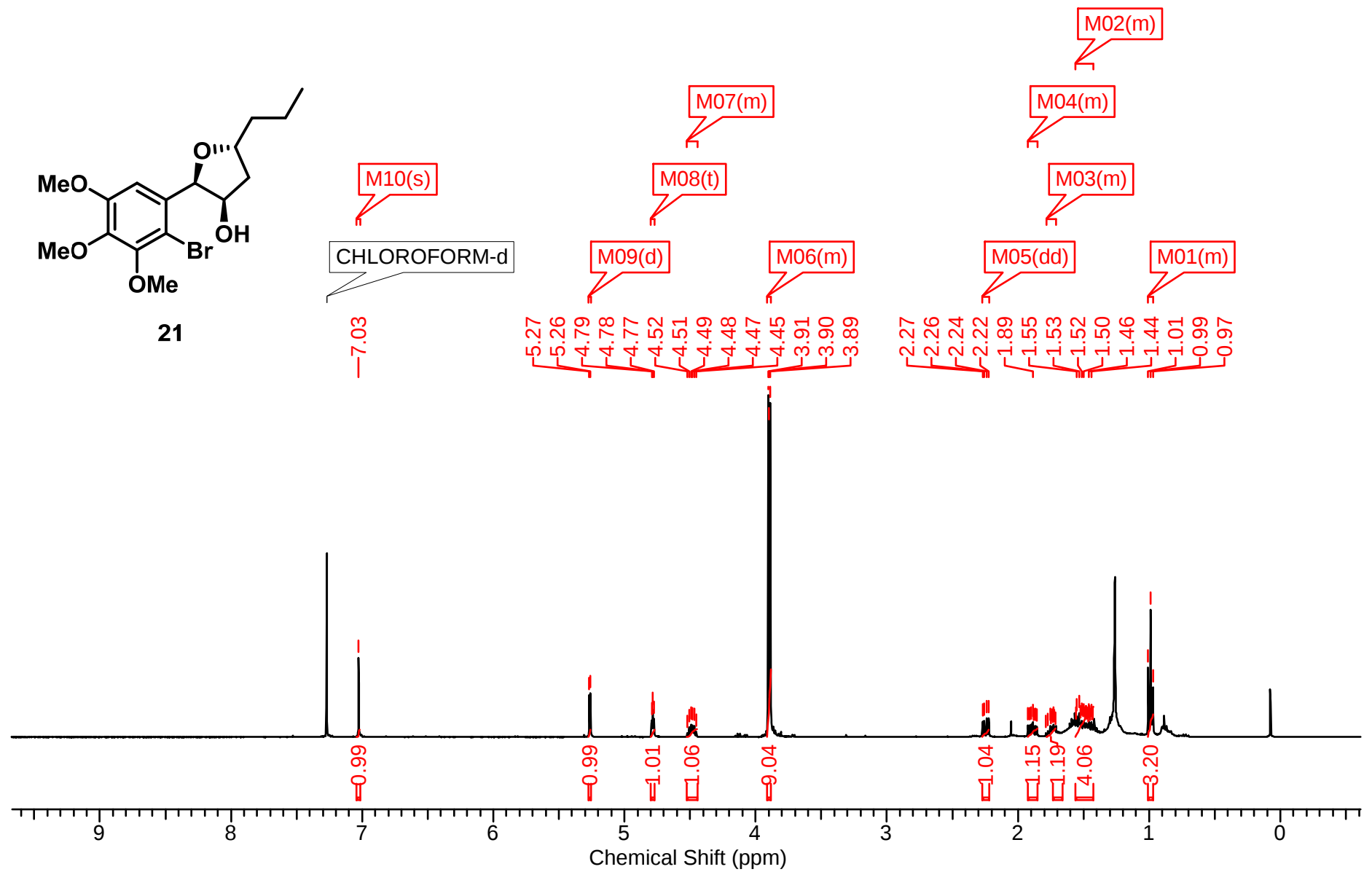
¹³ C NMR (100 MHz, CDCl₃) Of the compound (2*R*,3*aR*,9*bR*)-6,7,8-trimethoxy-2-propyl-2,3,3*a*,9*b*-tetrahydro-5*H*-furo[3,2-*c*]isochromen-5-one :



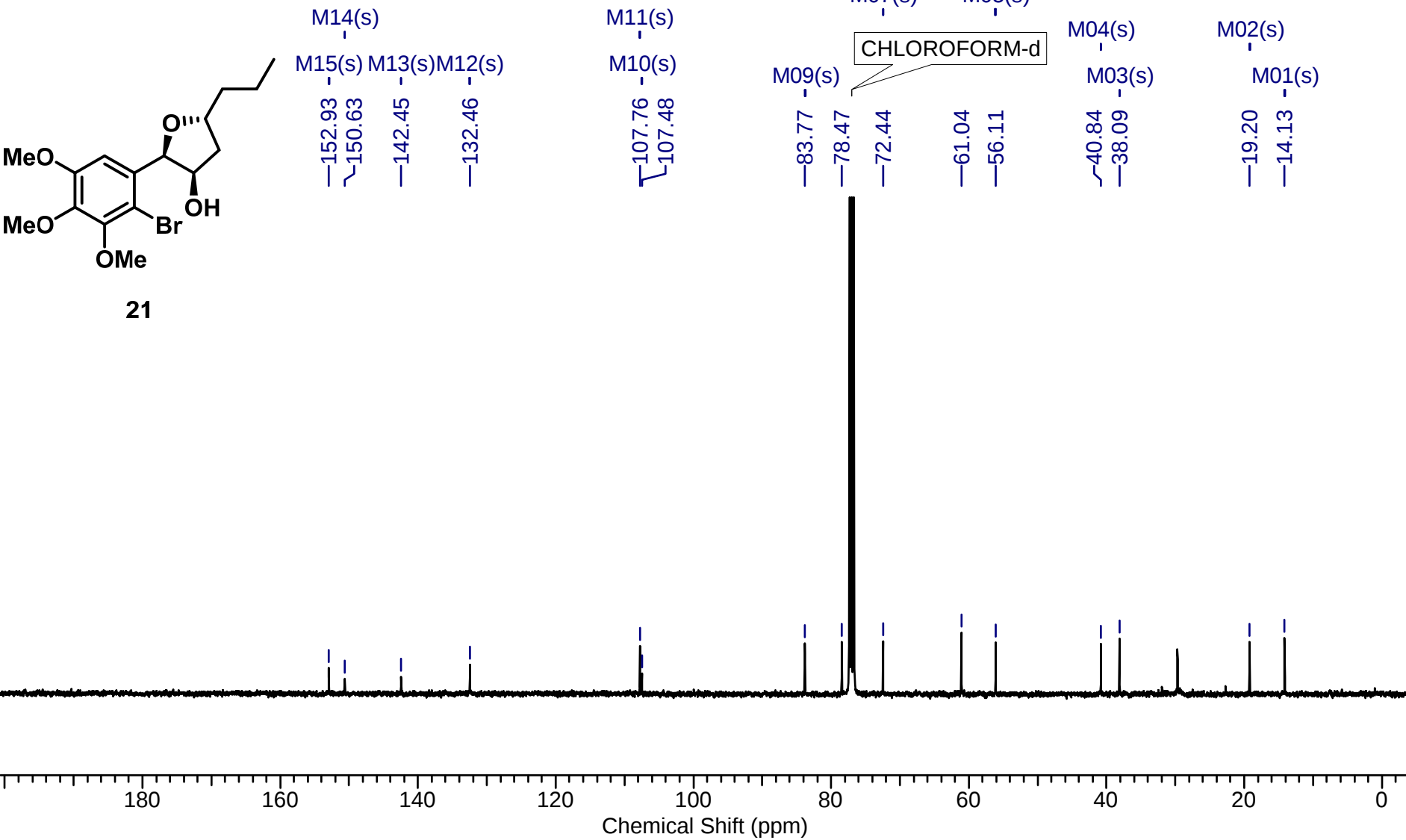
RRR-CYANO #936 RT: 4.19 AV: 1 NL: 1.05E9
T: FTMS + p ESI Full ms [100.00-700.00]



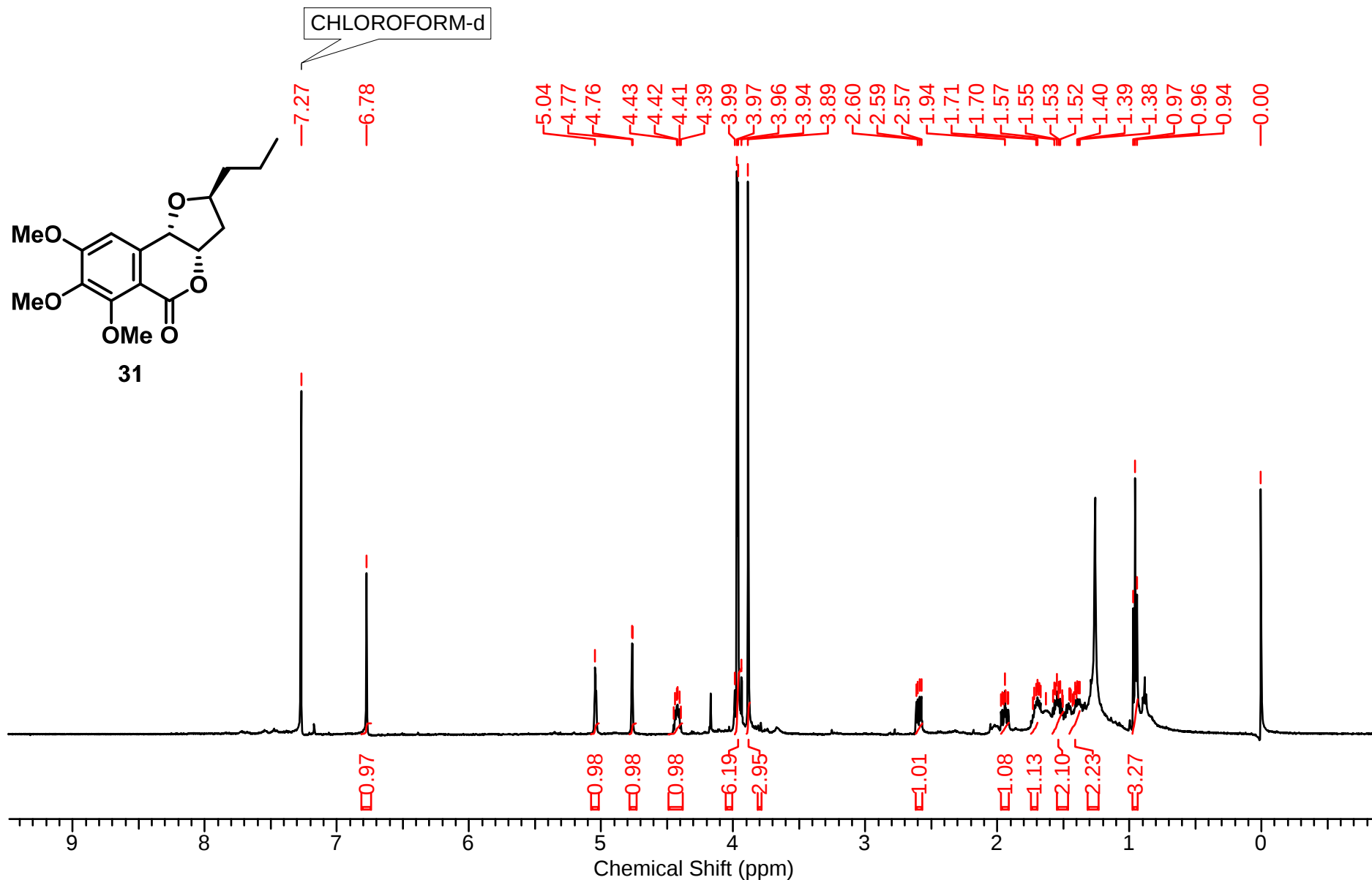
¹H NMR (400 MHz, CDCl₃) Of the compound (2S,3S,5S)-2-(2-bromo-3,4,5-trimethoxyphenyl)-5-propyltetrahydrofuran-3-ol



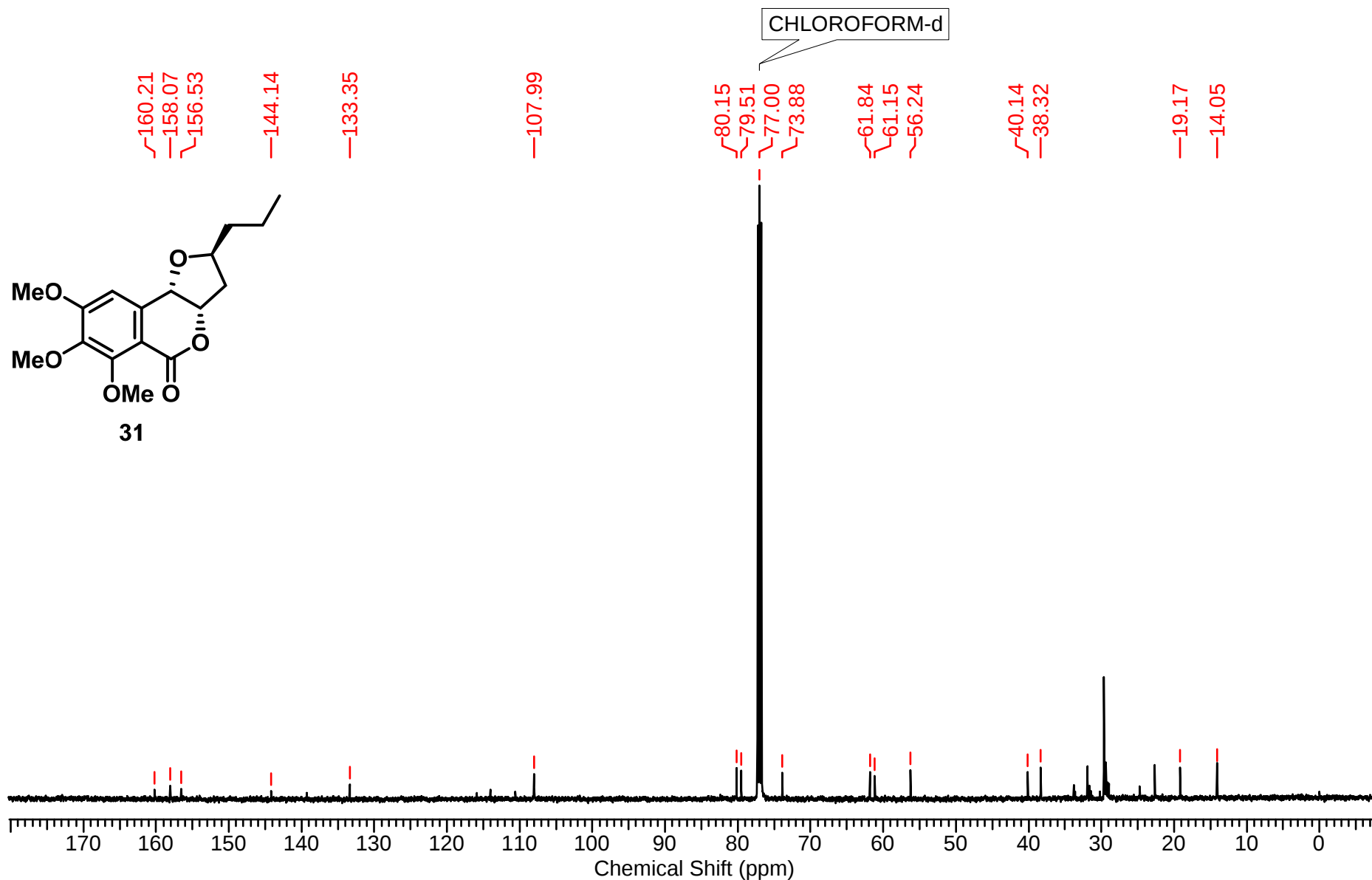
¹³ C NMR (100 MHz, CDCl₃) Of the compound (2*S*,3*S*,5*S*)-2-(2-bromo-3,4,5-trimethoxyphenyl)-5-propyltetrahydrofuran-3-ol



^1H NMR (500 MHz, CDCl_3) of the compound (2*S*,3*aS*,9*bS*)-6,7,8-trimethoxy-2-propyl-2,3,3*a*,9*b*-tetrahydro-5*H*-furo[3,2-*c*]isochromen-5-one



^{13}C NMR (125 MHz, CDCl_3) of the compound (2*S*,3*aS*,9*bS*)-6,7,8-trimethoxy-2-propyl-2,3,3*a*,9*b*-tetrahydro-5*H*-furo[3,2-*c*]isochromen-5-one



SSS-CYANO #895 RT: 4.02 AV: 1 NL: 3.65E8
T: FTMS + p ESI Full ms [100.00-700.00]

