

Supplementary Information

Cyclization reaction of amines with dialkyl carbonates to yield 1,3-oxazinan-2-ones*

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General Experimental Data

dimethyl propane-1,3-diyl dicarbonate **1** ^1H

dimethyl propane-1,3-diyl dicarbonate **1** ^{13}C

dimethyl propane-1,3-diyl dicarbonate **1** Exact Mass

dimethyl 2-methyl-propane-1,3-diyl dicarbonate **2** ^1H

dimethyl 2-methyl-propane-1,3-diyl dicarbonate **2** ^{13}C

dimethyl 2-methyl-propane-1,3-diyl dicarbonate **2** Exact Mass

3-benzyl-1,3-oxazinan-2-one **3** ^1H

3-benzyl-1,3-oxazinan-2-one **3** ^{13}C

3-benzyl-5-methyl-1,3-oxazinan-2-one **4** ^1H

3-benzyl-5-methyl-1,3-oxazinan-2-one **4** ^{13}C

3-benzyl-5-methyl-1,3-oxazinan-2-one **4** Exact Mass

methyl *N*-benzyl carbamate **5** ^1H

methyl *N*-benzyl carbamate **5** ^{13}C

3-hydroxy-2-methylpropyl *N*-benzyl carbamate **6** ^1H

3-hydroxy-2-methylpropyl *N*-benzyl carbamate **6** ^{13}C

3-hydroxy-2-methylpropyl *N*-benzyl carbamate **6** Exact Mass

1,3-dibenzylurea **7** ^1H

1,3-dibenzylurea **7** ^{13}C

methyl 2-methyl-3-(benzylcarbamoyl)propyl carbonate **8** ^1H

methyl 2-methyl-3-(benzylcarbamoyl)propyl carbonate **8** ^{13}C

methyl 2-methyl-3-(benzylcarbamoyl)propyl carbonate **8** Exact Mass

2-methylpropyl-1,3-dibenzylcarbamoyl **9** ¹H

2-methylpropyl-1,3-dibenzylcarbamoyl **9** ¹³C

2-methylpropyl-1,3-dibenzylcarbamoyl **9** Exact Mass

dimethyl-butane-1,4-diyl dicarbonate **11** ¹H

dimethyl-butane-1,4-diyl dicarbonate **11** ¹³C

dimethyl-pentane-1,5-diyl dicarbonate **12** ¹H

dimethyl-pentane-1,5-diyl dicarbonate **12** ¹³C

dimethyl-pentane-1,5-diyl dicarbonate **12** Exact Mass

3-phenylamino-1,3-oxazinan-2-one **13** ¹H

3-phenylamino-1,3-oxazinan-2-one **13** ¹³C

3-phenylamino-1,3-oxazinan-2-one **13** Exact Mass

5-methyl-3-phenylamino-1,3-oxazinan-2-one **14** ¹H

5-methyl-3-phenylamino-1,3-oxazinan-2-one **14** ¹³C

5-methyl-3-phenylamino-1,3-oxazinan-2-one **14** Exact Mass

3-phenyl-1,3-oxazinan-2-one **15** ¹H

3-phenyl-1,3-oxazinan-2-one **15** ¹³C

5-methyl-3-phenyl-1,3-oxazinan-2-one **16** ¹H

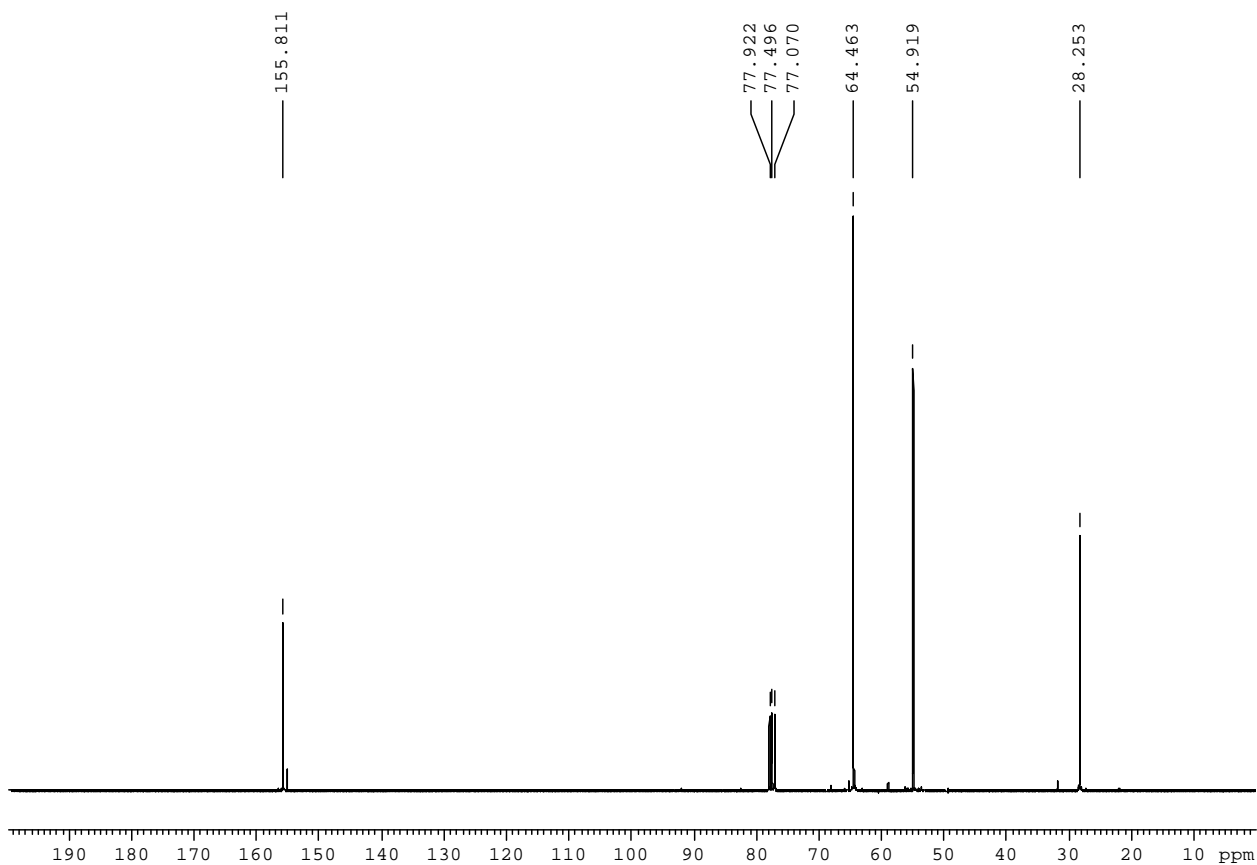
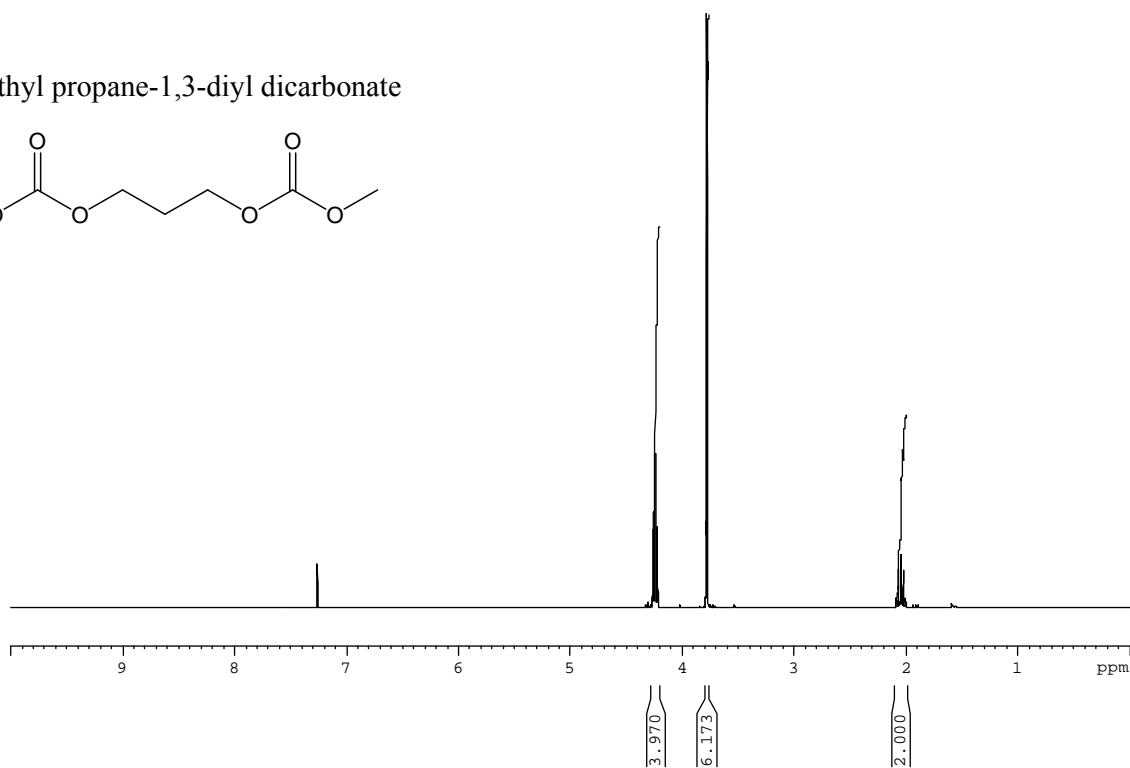
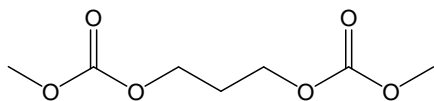
5-methyl-3-phenyl-1,3-oxazinan-2-one **16** ¹³C

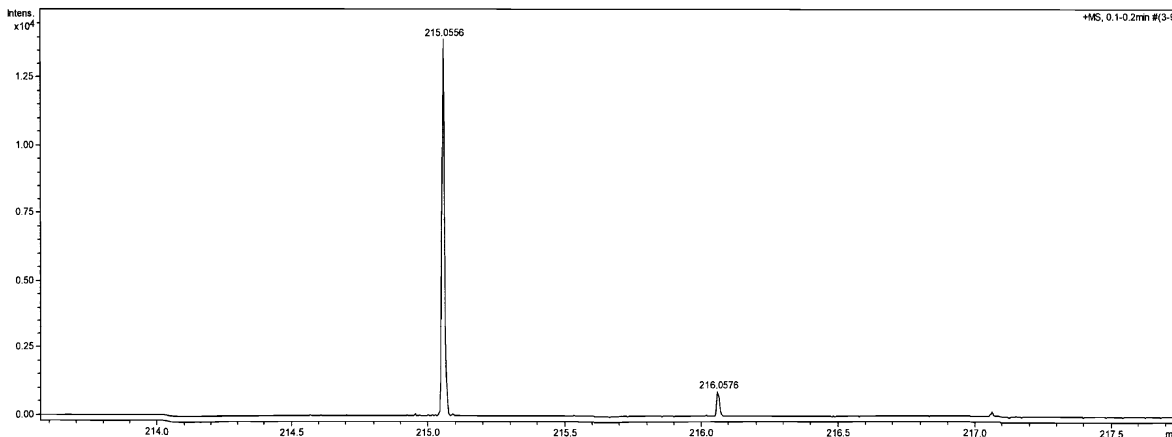
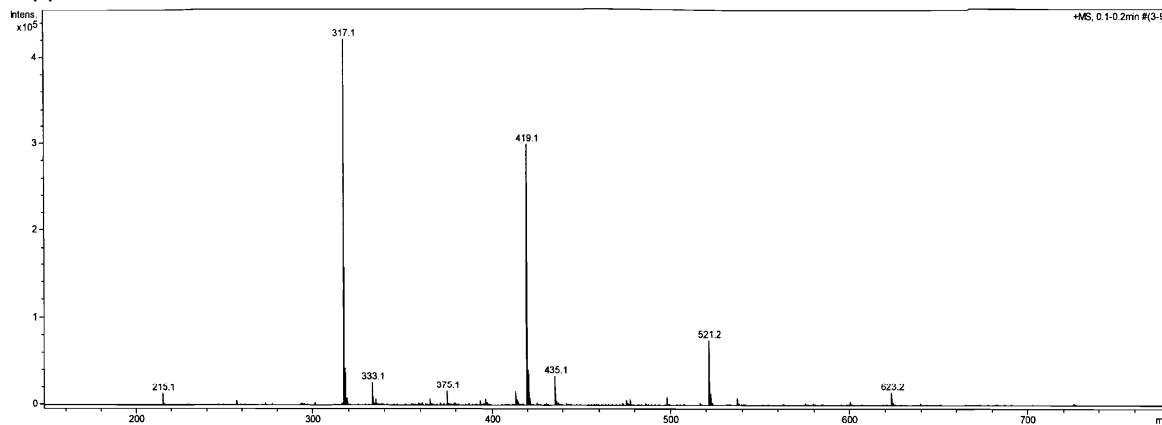
Table 3 - Crystal Data and Structure Parameters for Compounds **13-16**

General Experimental Data. All experimental procedures described were carried out under air and in oven dried glassware. All reagents were ASC grade and were employed without further purification. ¹H and ¹³C NMR spectra were obtained on an 300 MHz spectrometer at 25 °C and referred to deuterated chloroform as internal standard. Melting point analyses carried out are uncorrected. HPLC analyses were carried out at 20 °C on a C18 5 μm column (4.6 x 150 mm) using an eluting system of 65/35 acidified water/acetonitrile at 0.8 mL/min and detecting at λ = 210. A typical sample size was 100 μL of a solution comprising of 0.1 g of compound, 5 mL of 18.8 mM

solution of nitrobenzene in acetonitrile as standard and 20 mL of 50/50 acetonitrile/water. GC-MS analyses were performed at 70 eV (MS) with a 30 m HP 5 capillary GC column. A typical sample size was 0.2 μ L of a solution of 1 mg of compound in 1 mL of dichloromethane.

1 dimethyl propane-1,3-diyl dicarbonate





Elemental Composition Report

Single Mass Analysis (displaying only valid results)

Tolerance = 50.0 PPM / DBE: min = -1.5, max = 50.0
Isotope matching not enabled

Monoisotopic Mass, Even Electron Ions
1139 formula(e) evaluated with 2 results within limits (up to 20 closest results for each mass)

Mass	Calc. Mass	mDa	PPM	DBE	Formula
216.0576	216.0565	1.1	5.0	1.5	¹² C ₆ ¹³ C ¹ H ₁₂ ¹⁶ O ₆ ²³ Na
	216.0506	7.0	32.2	10.5	¹² C ₁₃ ¹³ C ¹ H ₈ ¹⁶ O ²³ Na

Elemental Composition Report

Single Mass Analysis (displaying only valid results)

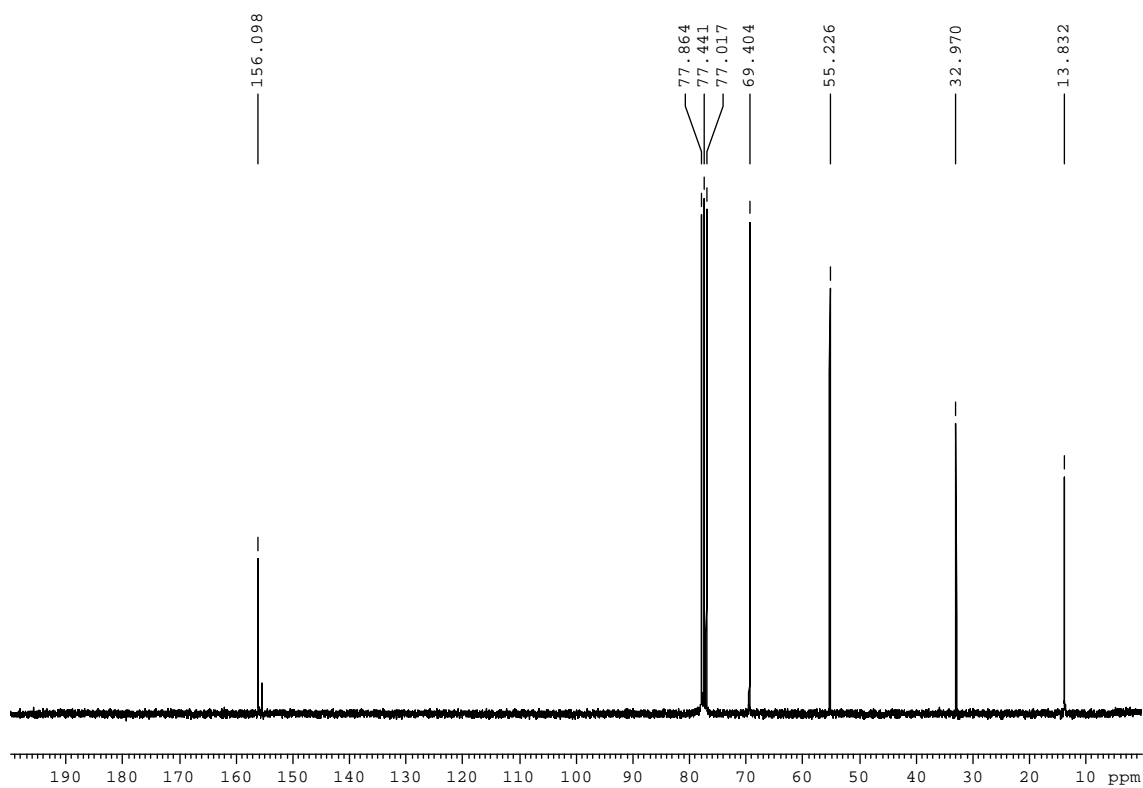
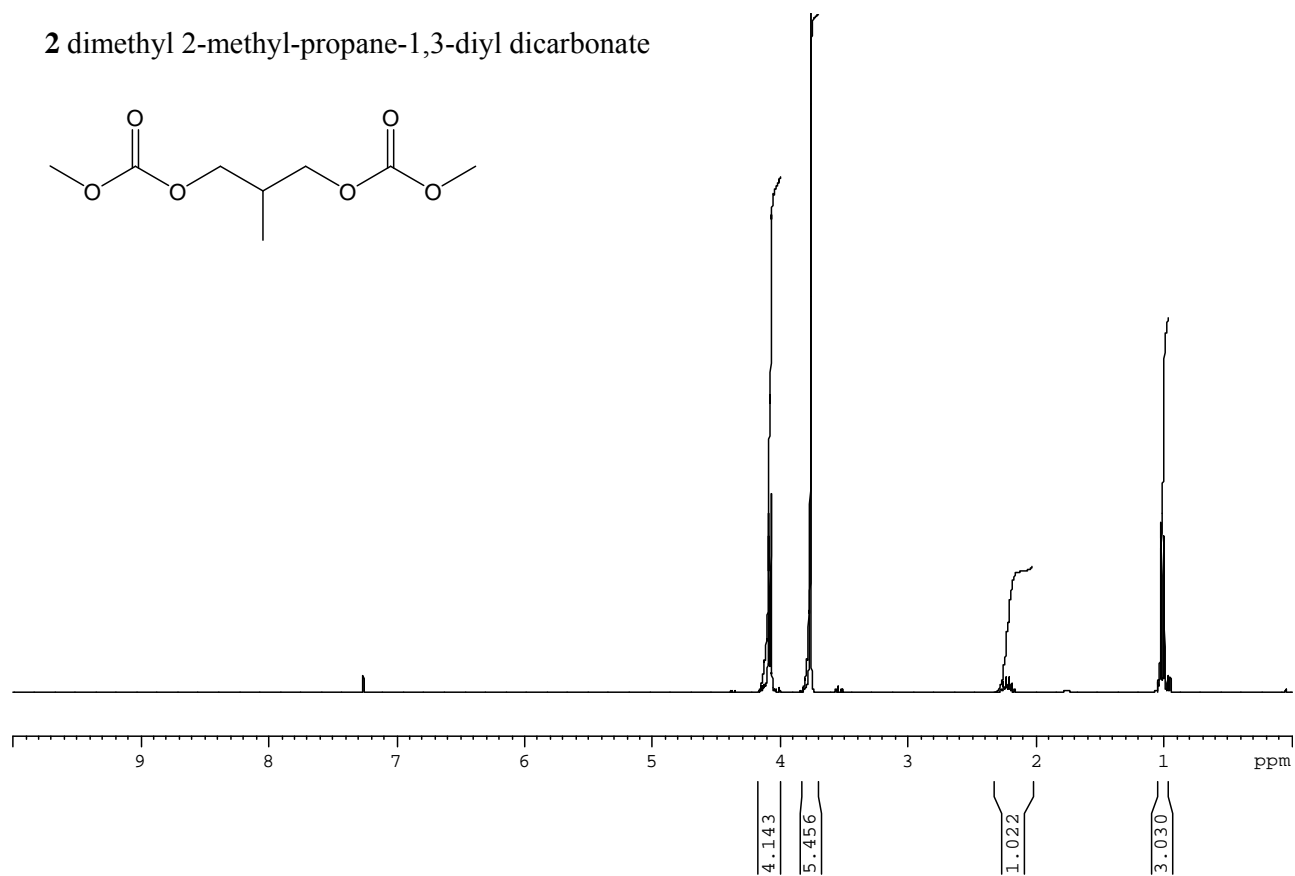
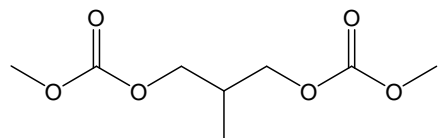
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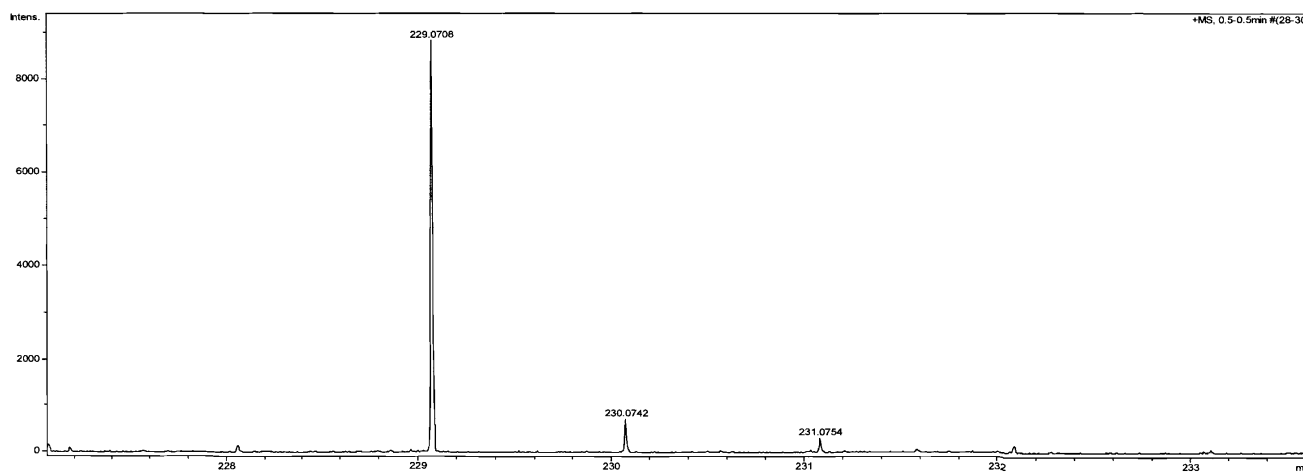
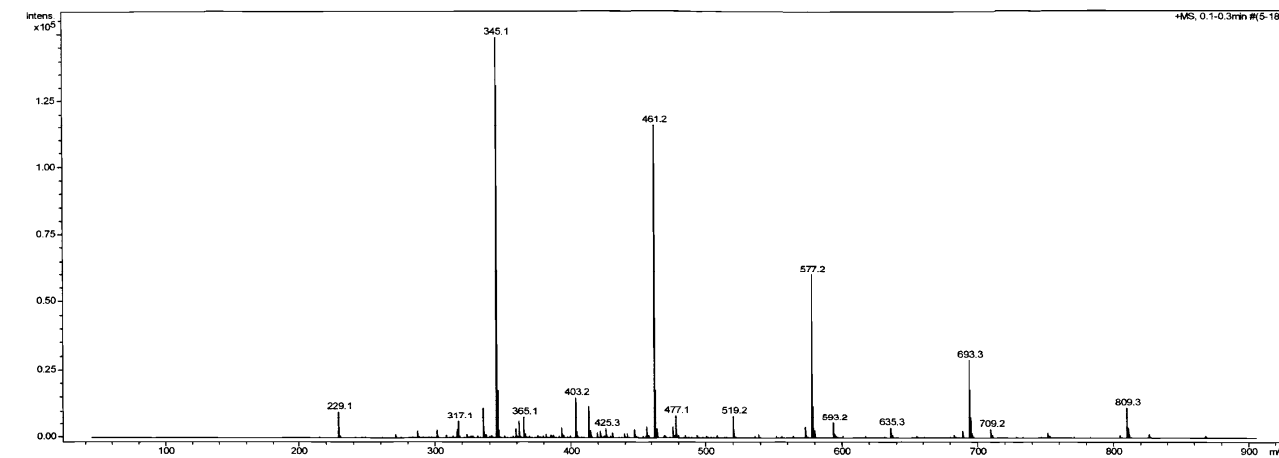
Monoisotopic Mass, Even Electron Ions
1501 formula(e) evaluated with 2 results within limits (up to 20 closest results for each mass)

Mass	Calc. Mass	mDa	PPM	DBE	Formula
215.0556	215.0532	2.4	11.4	1.5	¹² C ₇ ¹ H ₁₂ ¹⁶ O ₆ ²³ Na
	215.0473	8.3	38.7	10.5	¹² C ₁₄ ¹ H ₈ ¹⁶ O ²³ Na

Sample was dissolved in methanol and was diluted in 50% methanol, 50% water and 0.1% formic acid. Then it was directly injected into the Bruker maXis mass spectrometer for analysis in the positive ESI mode. The observed peak at m/z of 215.0556 was within 11 ppm of the theoretical [M+Na]⁺ value (215.0532), and the observed C¹³ peak at 216.0576 was within 5 ppm of the expected value (216.0565)

2 dimethyl 2-methyl-propane-1,3-diyl dicarbonate





Elemental Composition Report

Single Mass Analysis (displaying only valid results)
 Tolerance = 50.0 PPM / DBE: min = -1.5, max = 50.0
 Isotope matching not enabled

Monoisotopic Mass, Even Electron Ions
 1567 formula(e) evaluated with 2 results within limits (up to 20 closest results for each mass)

Mass	Calc. Mass	mDa	PPM	DBE	Formula
230.0742	230.0722	2.0	8.9	1.5	12C7 13C 1H14 16O6 23Na
	230.0663	7.9	34.4	10.5	12C14 13C 1H10 16O 23Na

Elemental Composition Report

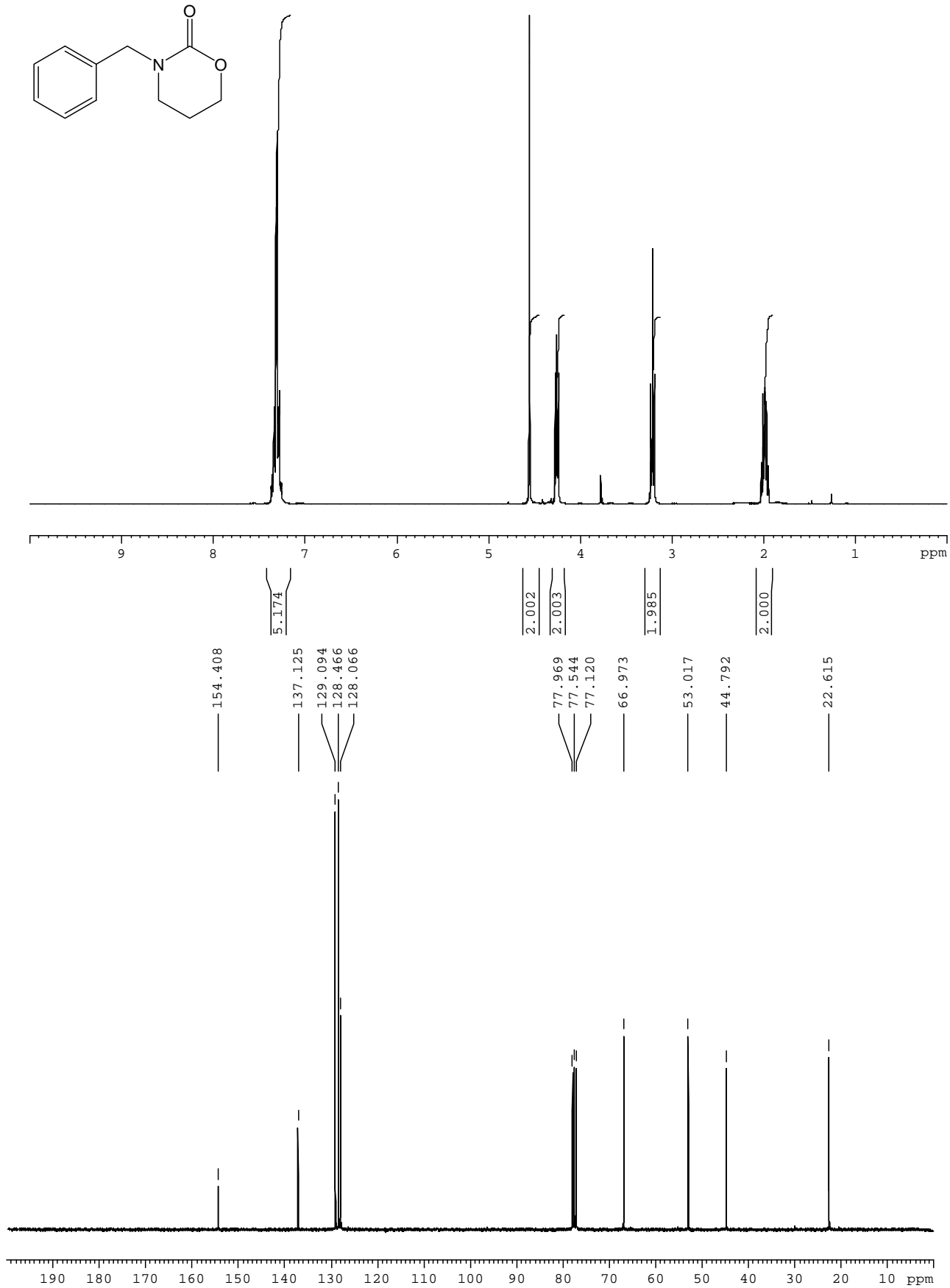
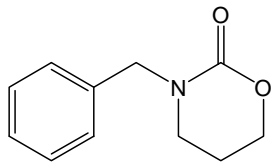
Single Mass Analysis (displaying only valid results)
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 Isotope matching not enabled

Monoisotopic Mass, Even Electron Ions
 1993 formula(e) evaluated with 2 results within limits (up to 20 closest results for each mass)

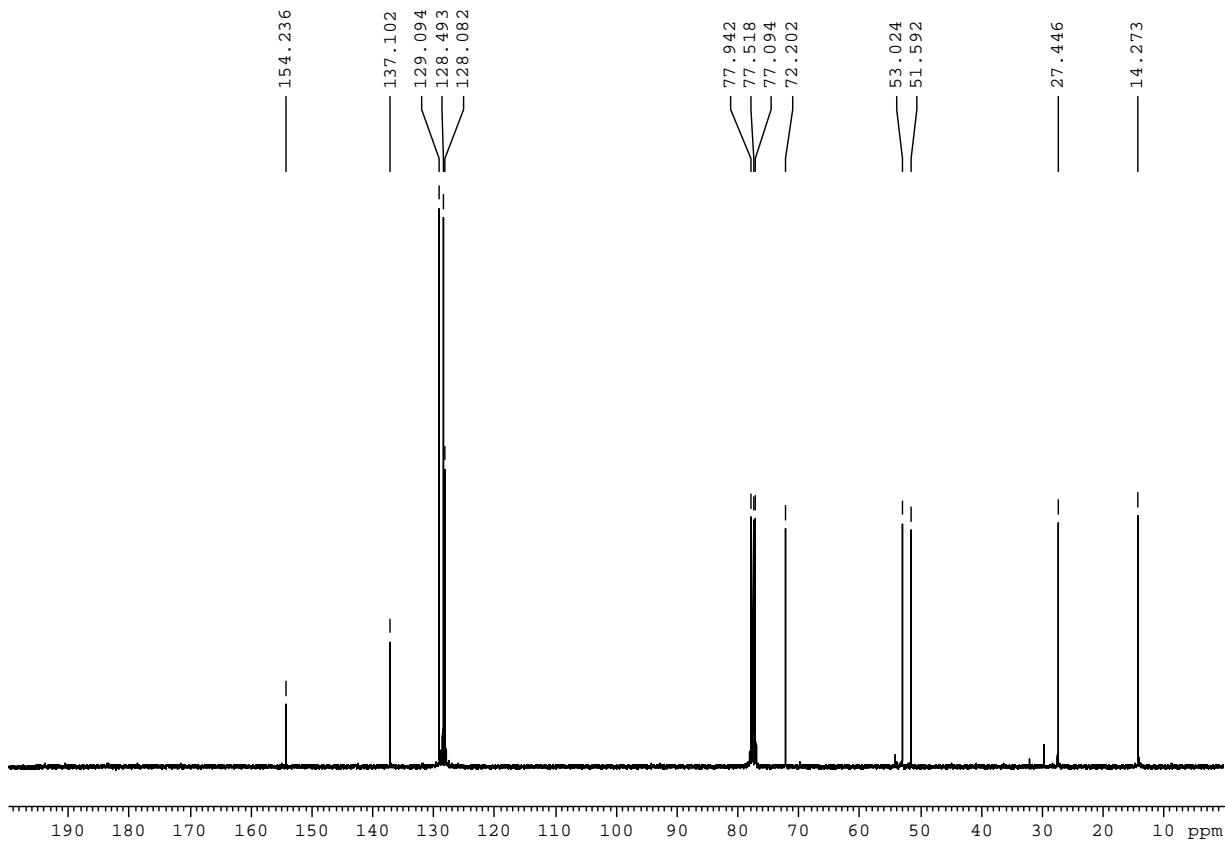
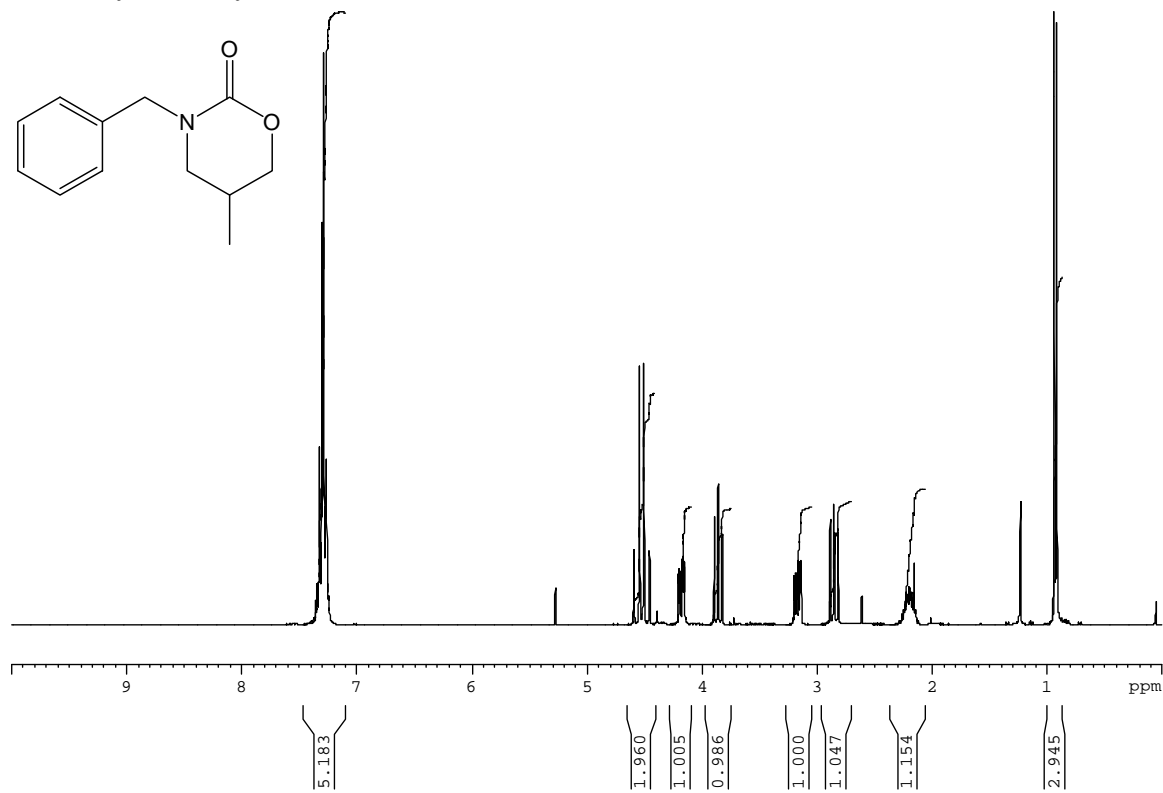
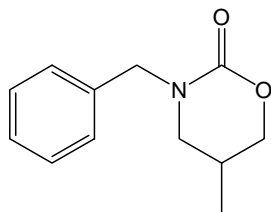
Mass	Calc. Mass	mDa	PPM	DBE	Formula
229.0708	229.0688	2.0	8.7	1.5	12C8 1H14 16O6 23Na
	229.0629	7.9	34.3	10.5	12C15 1H10 16O 23Na

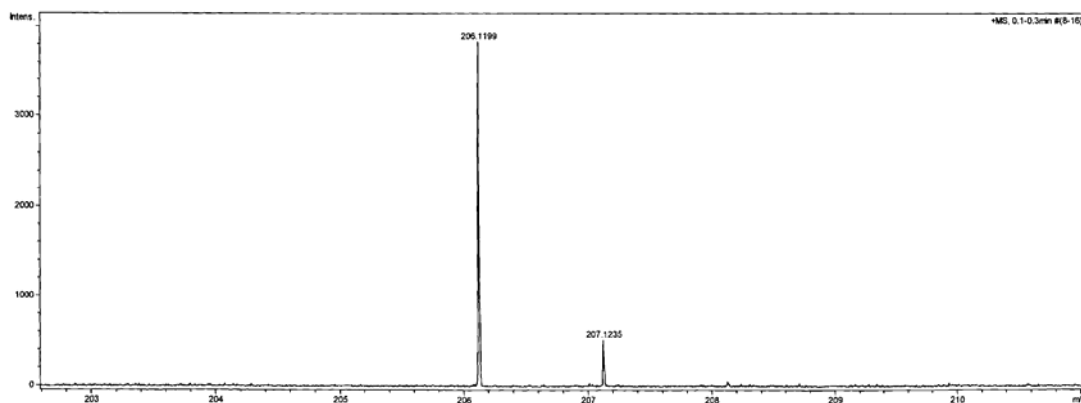
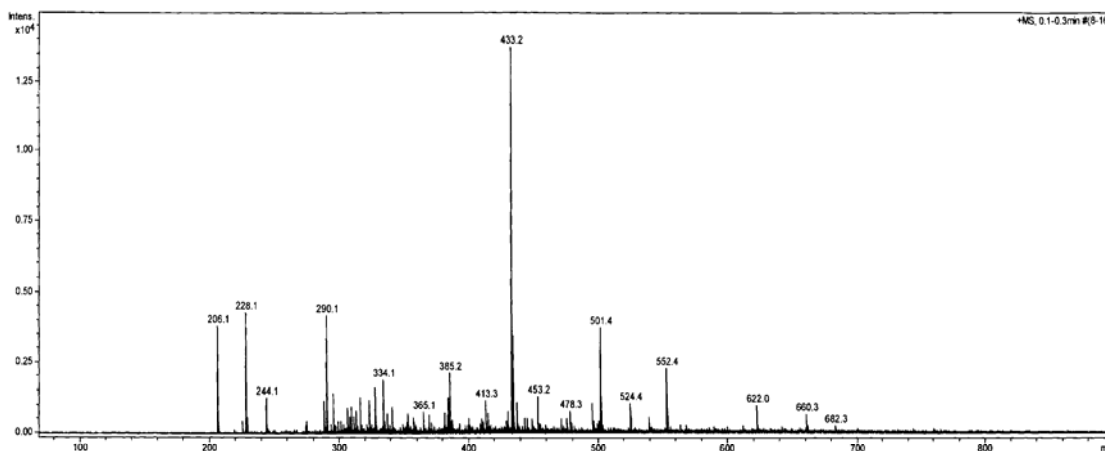
Sample was dissolved in methanol and was diluted in 50% methanol, 50% water and 0.1% formic acid. Then it was directly injected into the Bruker maXis mass spectrometer for analysis in the positive ESI mode. The observed peak at m/z of 229.0708 was within 9 ppm of the theoretical [M+Na]⁺ value (229.0688), and the observed C13 peak at 230.0742 was within 9 ppm of the expected value (230.0722)

3 3-benzyl-1,3-oxazinan-2-one



4 3-benzyl-5-methyl-1,3-oxazinan-2-one





Elemental Composition Report

Single Mass Analysis

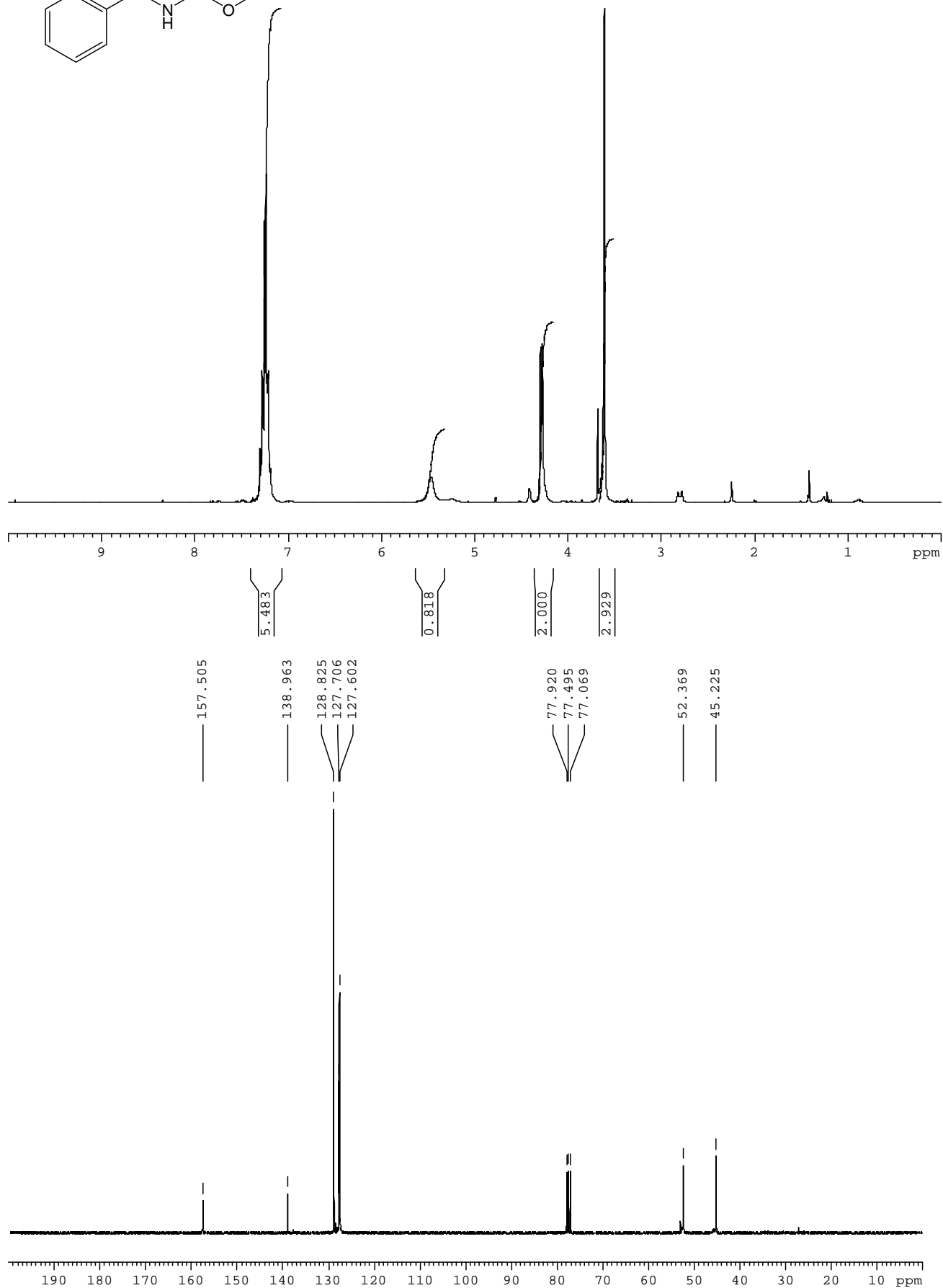
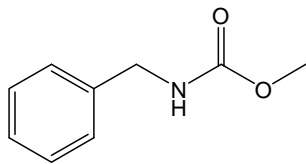
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 Isotope matching not enabled

Monoisotopic Mass, Even Electron Ions
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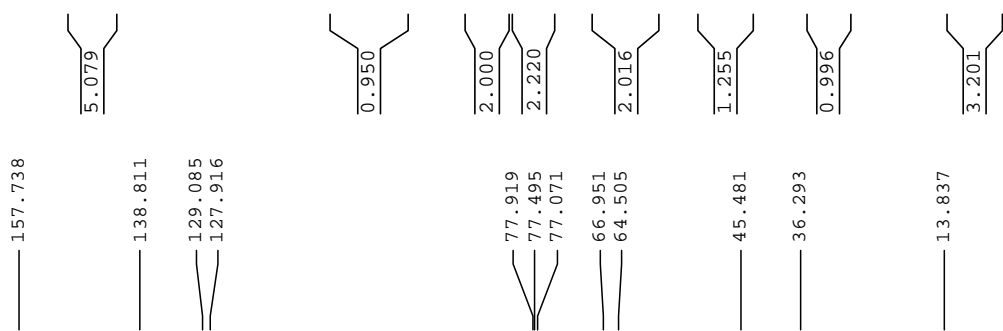
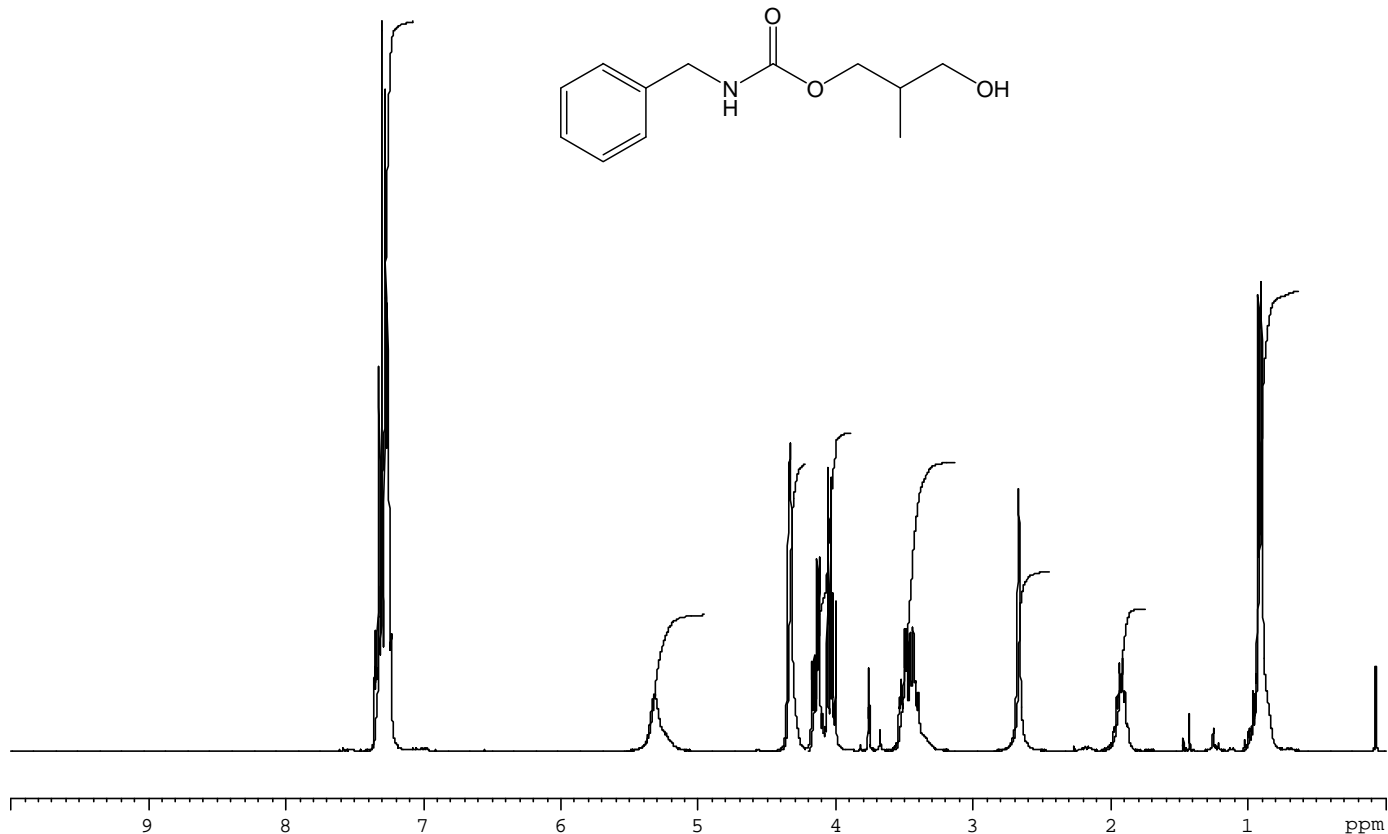
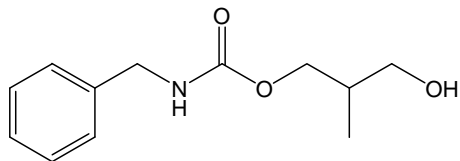
Mass	Calc. Mass	mDa	PPM	DBE	Formula
207.1225	207.1215	1.0	5.0	5.5	12C11 13C 1H16 14N 16O2
	207.1174	5.1	24.4	1.5	12C6 13C 1H16 14N3 16O4
	207.1287	-6.2	-29.8	1.5	12C5 13C 1H16 14N5 16O3
	207.1327	-10.2	-49.2	5.5	12C10 13C 1H16 14N3 16O

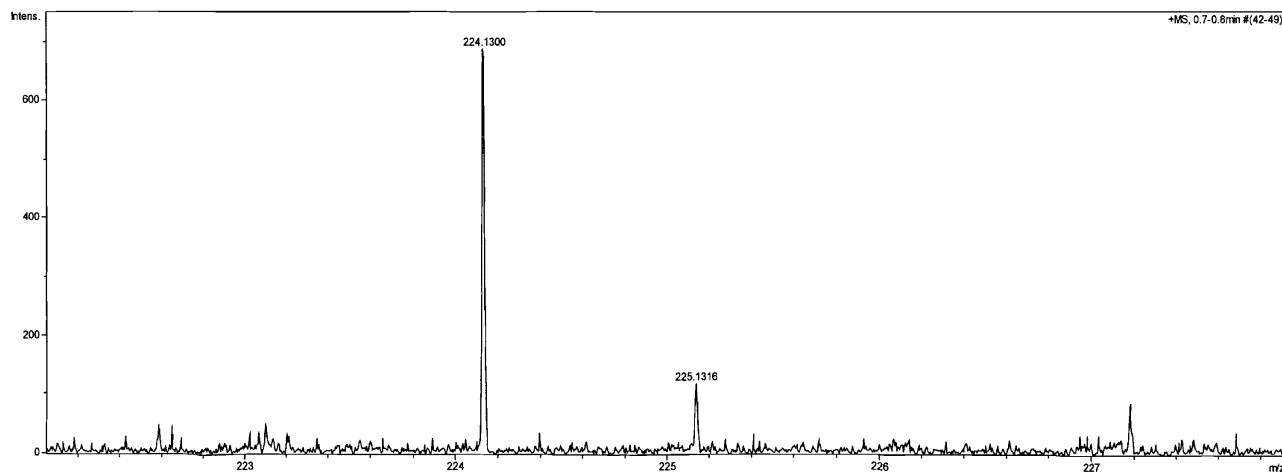
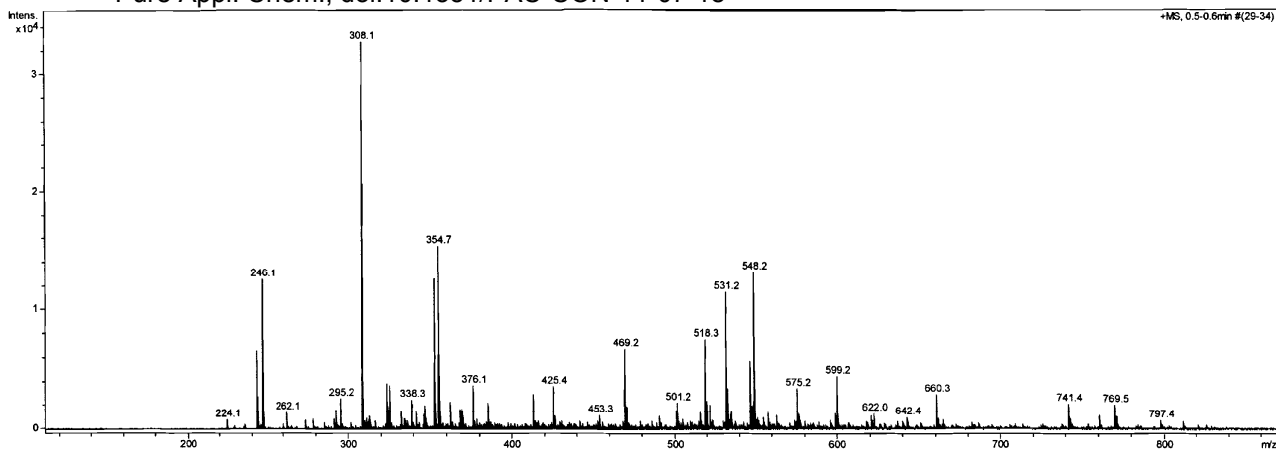
Sample was dissolved in methanol and was diluted in 50% methanol, 50% water and 0.1% formic acid. Then it was directly injected into the Bruker maXis mass spectrometer for analysis in the positive ESI mode. The observed peak at m/z of 206.1199 was within 9 ppm of the theoretical [M+H]⁺ value (206.1182), and the observed C13 peak at 207.1225 was within 5 ppm of the expected value (207.1215).

5 methyl N-benzyl carbamate



6 3-hydroxy-2-methylpropyl N-benzyl carbamate





Elemental Composition Report

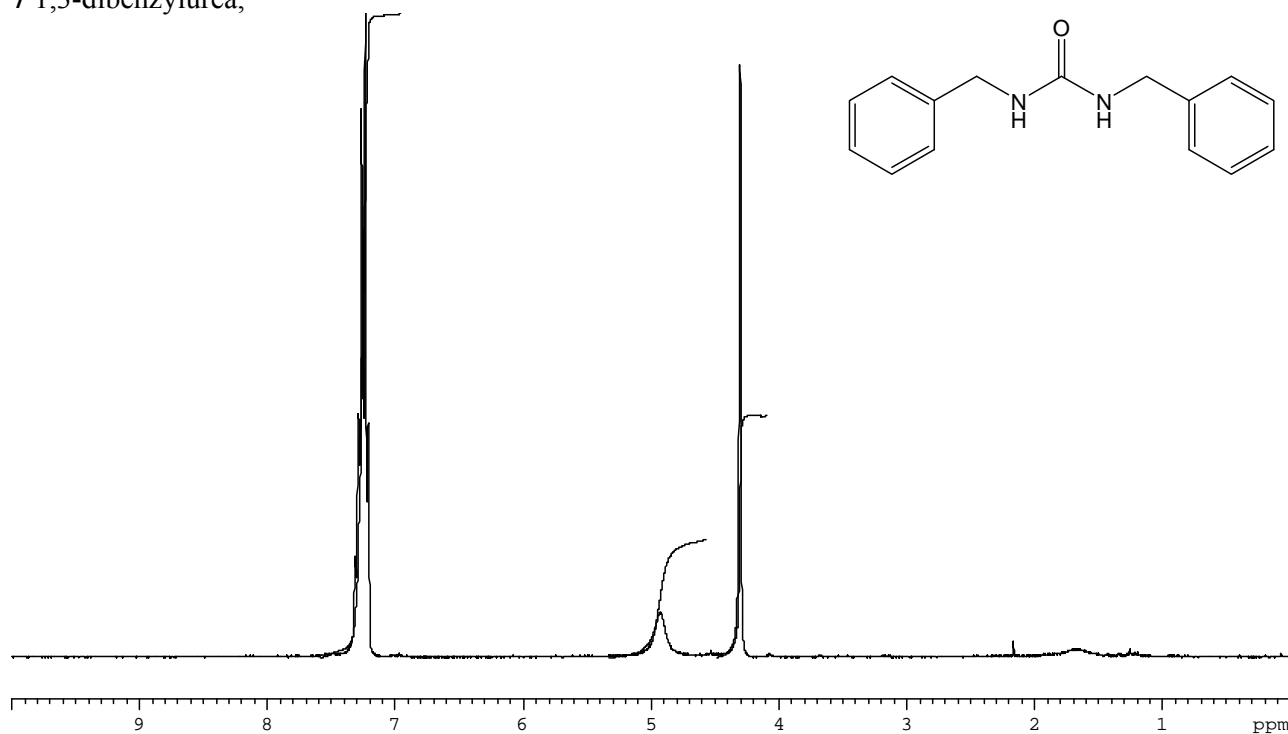
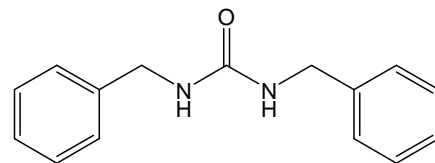
Single Mass Analysis (displaying only valid results)
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 Isotope matching not enabled

Monoisotopic Mass, Even Electron Ions
 1687 formula(e) evaluated with 3 results within limits (up to 50 closest results for each mass)

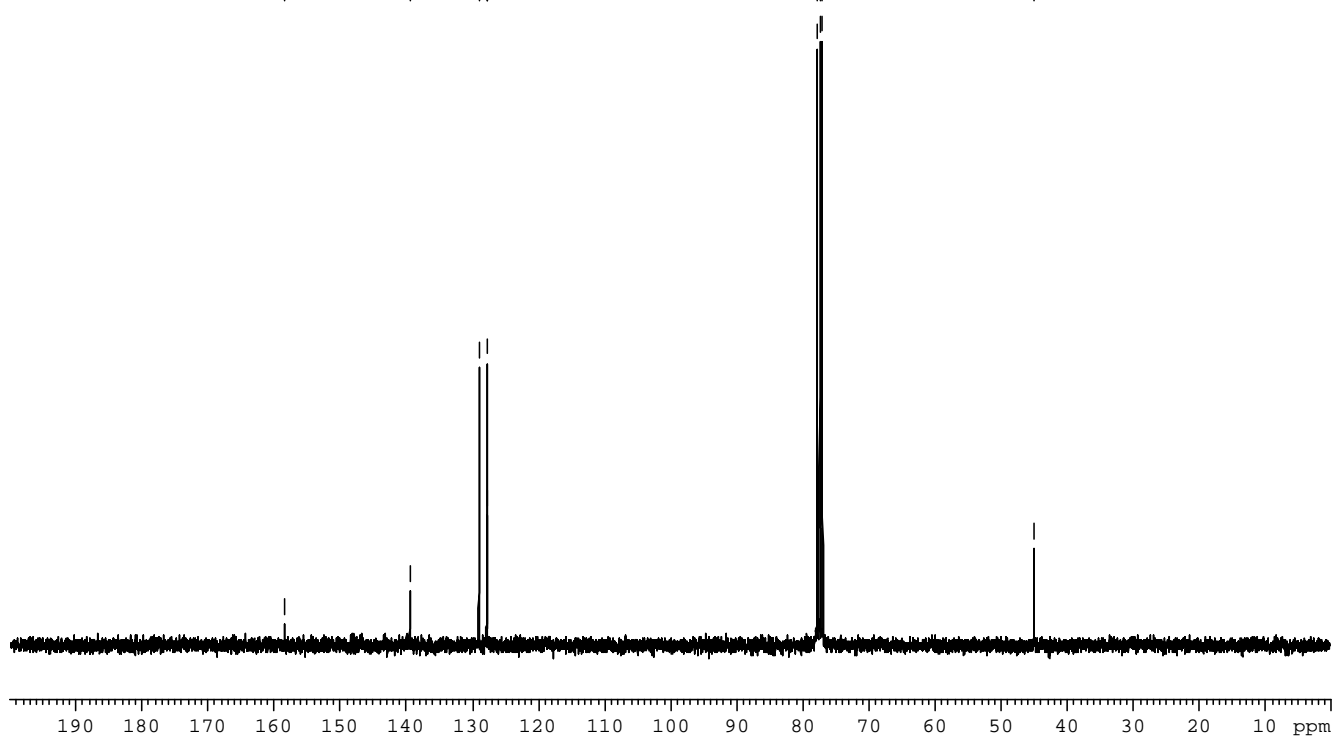
Mass	Calc. Mass	mDa	PPM	DBE	Formula
224.1300	224.1287	1.3	5.9	4.5	12C12 1H18 14N 16O3
	224.1399	-9.9	-44.2	4.5	12C11 1H18 14N3 16O2
	224.1511	-21.1	-94.3	4.5	12C10 1H18 14N5 16O

Sample was dissolved in methanol and was diluted in 50% methanol, 50% water and 0.1% formic acid. Then it was directly injected into the Bruker maXis mass spectrometer for analysis in the positive ESI mode. The observed peak at m/z of 224.1300 was within 6 ppm of the theoretical [M+H]⁺ value (224.1287), and the observed C13 peak at 225.1316 was within 2 ppm of the expected value (225.1320)

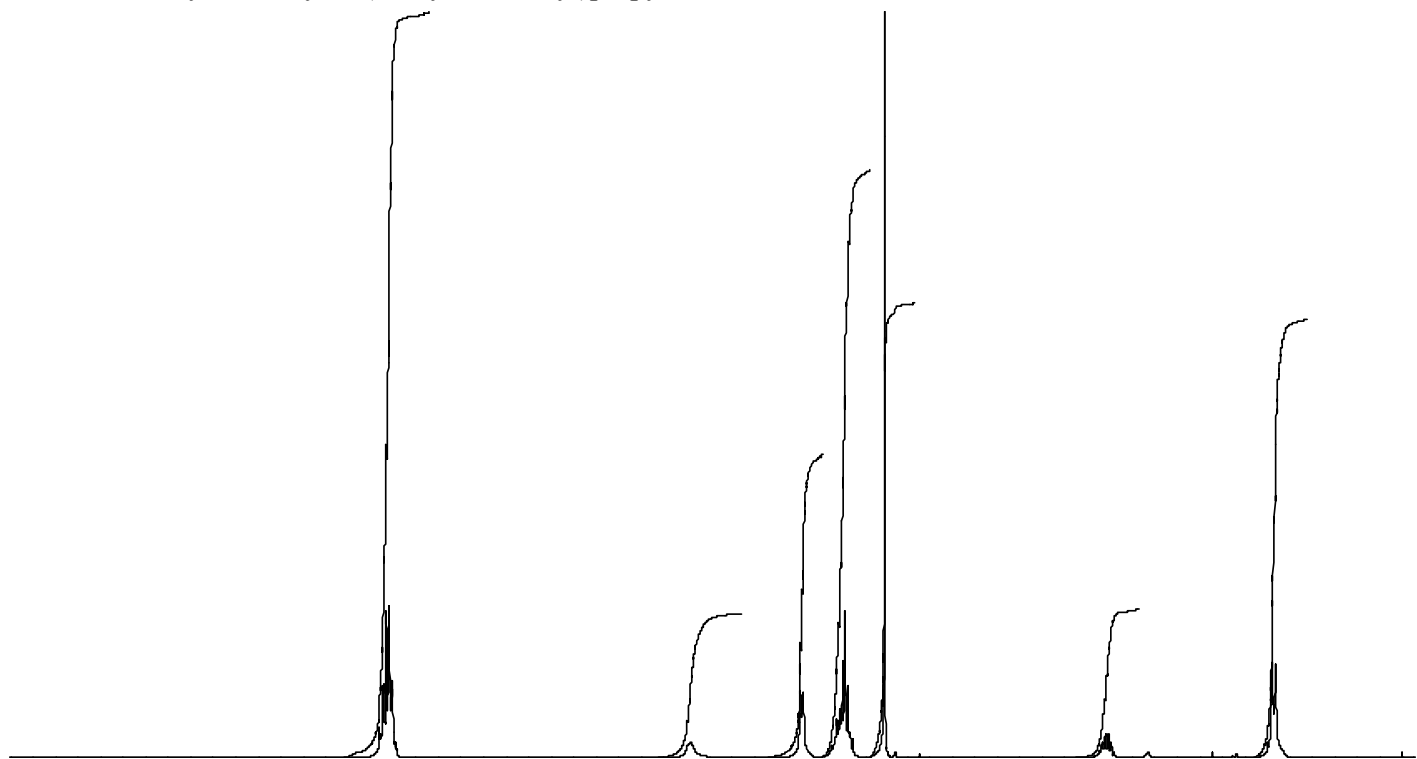
7 1,3-dibenzylurea,



158.475
139.477
129.037
127.825
127.724
77.857
77.434
77.011
44.953



8 methyl 2-methyl-3-(benzylcarbamoyl)propyl carbonat



9 8 7 6 5 4 3 2 1 ppm

156.847
156.192
5.098

138.872

129.061
127.934
127.884

0.987

2.082

77.4523
77.497
77.3198

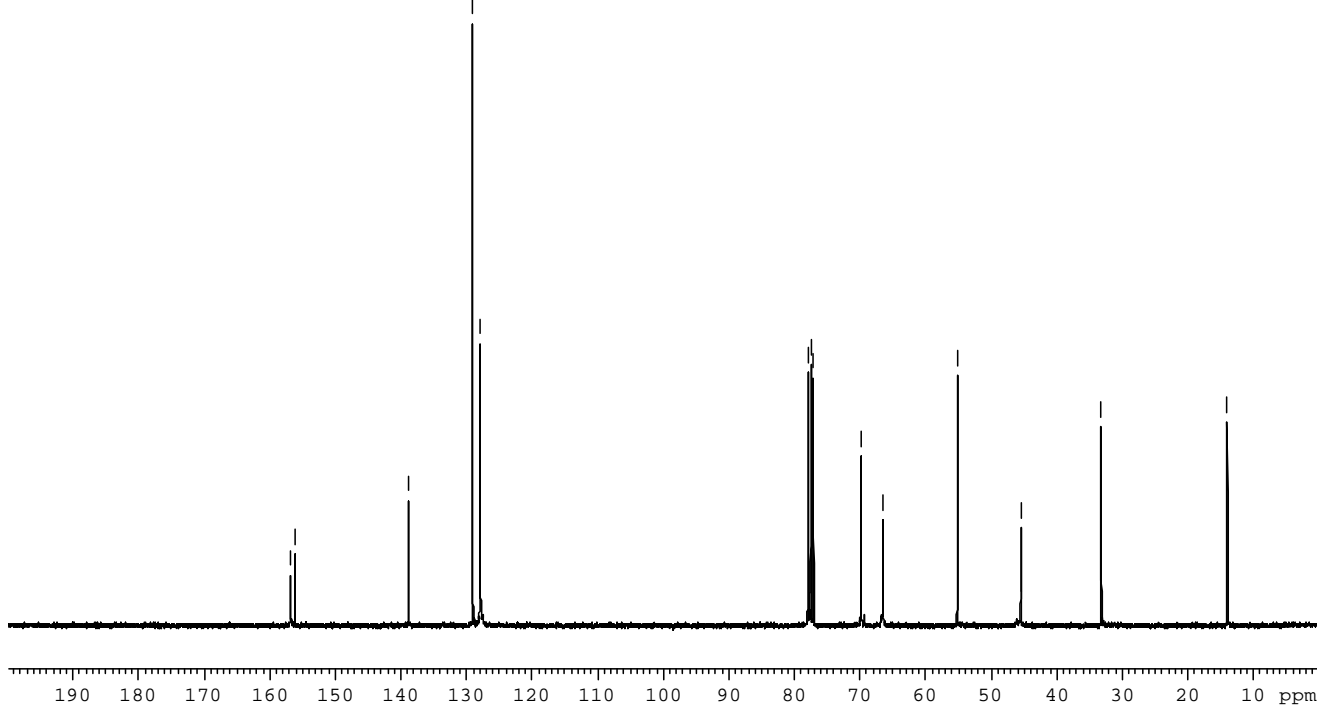
69.862
66.469

55.167

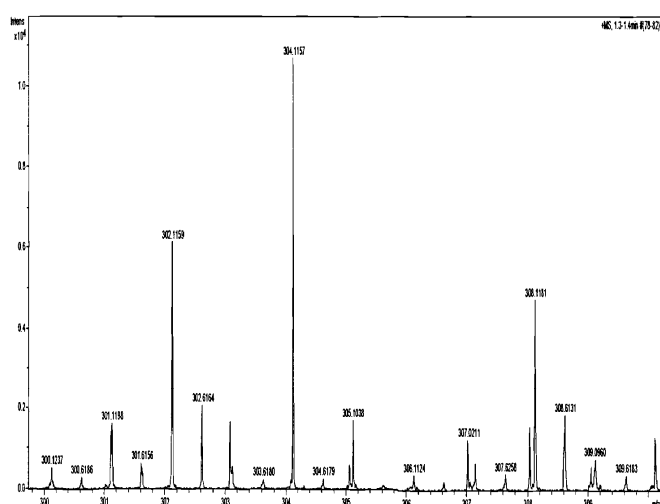
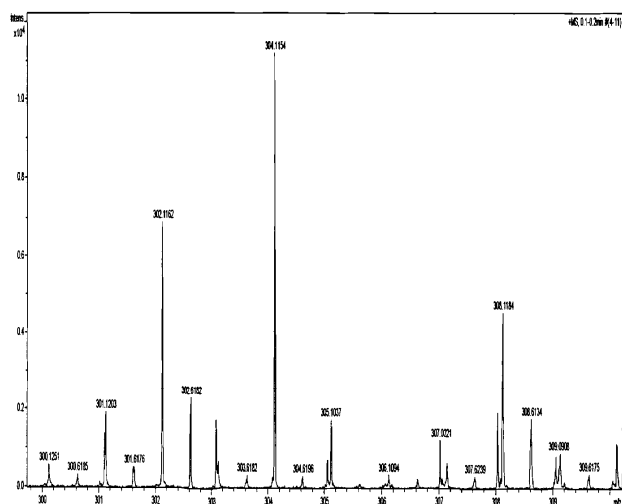
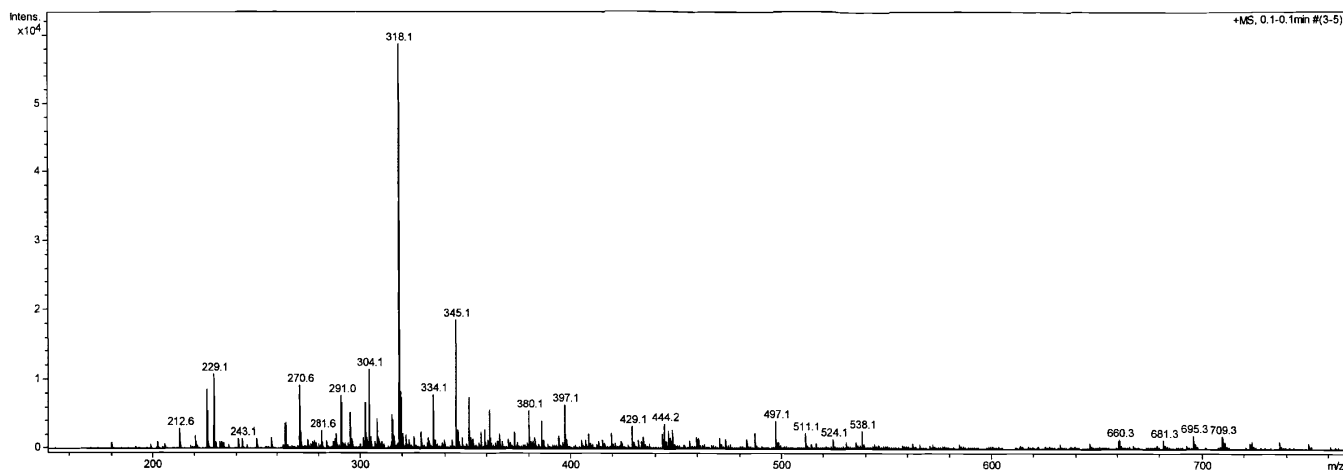
45.459
1.018

33.218

3.000
13.572



190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



Elemental Composition Report

Single Mass Analysis (displaying only valid results)

Tolerance = 50.0 PPM / DBE: min = -1.5, max = 50.0

Isotope matching not enabled

Monoisotopic Mass, Even Electron Ions

9053 formula(e) evaluated with 7 results within limits (up to 20 closest results for each mass)

Mass	Calc. Mass	mDa	PPM	DBE	Formula
304.1154	304.1161	-0.7	-2.3	5.5	12C14 1H19 14N 16O5 23Na
	304.1134	2.0	6.6	6.5	12C10 1H15 14N7 16O3 23Na
	304.1174	-2.0	-6.7	10.5	12C15 1H15 14N5 16O 23Na
	304.1062	9.2	30.3	10.5	12C16 1H15 14N3 16O2 23Na
	304.1273	-11.9	-39.2	5.5	12C13 1H19 14N3 16O4 23Na
	304.1022	13.2	43.5	6.5	12C11 1H15 14N5 16O4 23Na
	304.1008	14.6	47.9	1.5	12C10 1H19 14N 16O8 23Na

Elemental Composition Report

Single Mass Analysis (displaying only valid results)

Tolerance = 50.0 PPM / DBE: min = -1.5, max = 50.0

Isotope matching not enabled

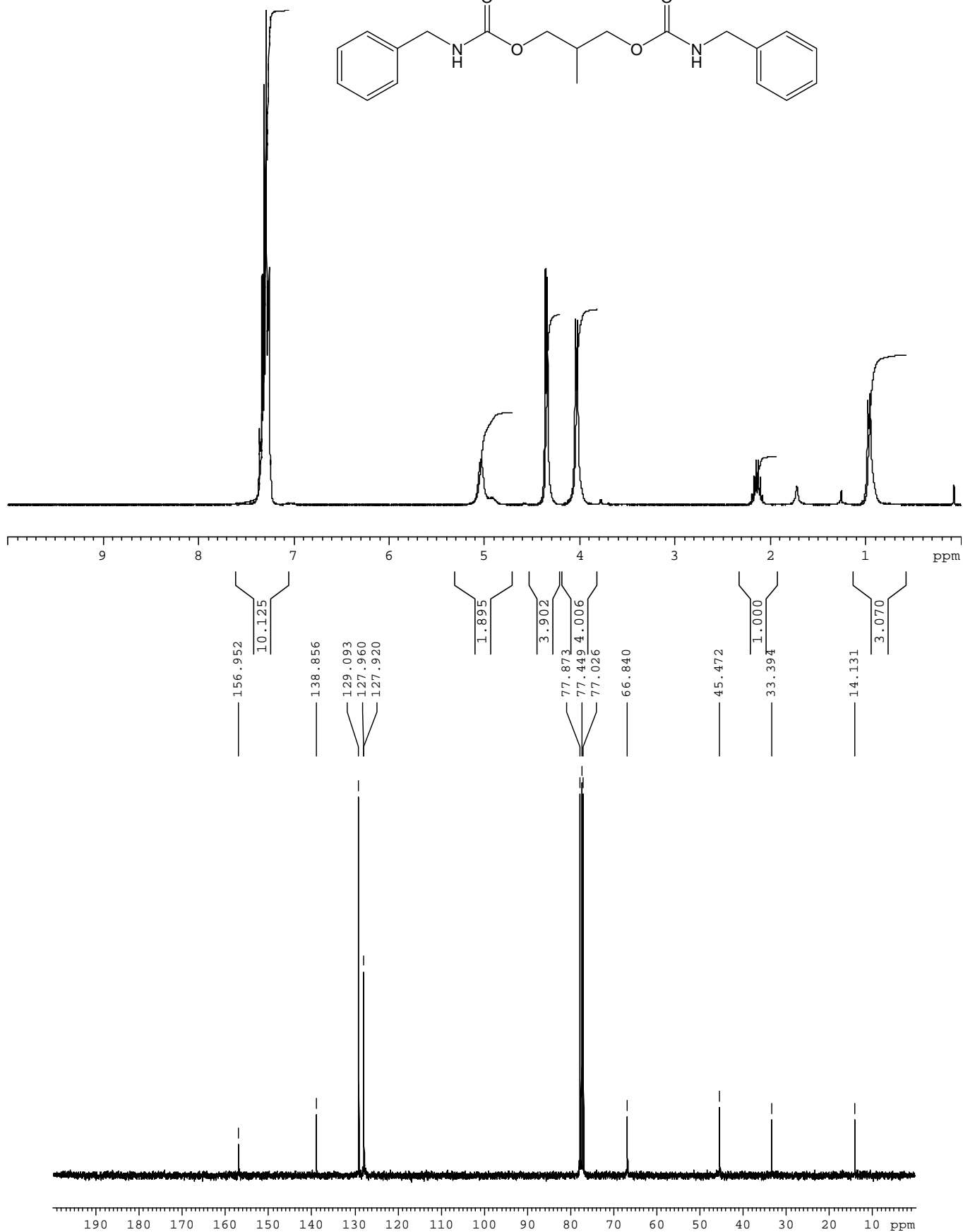
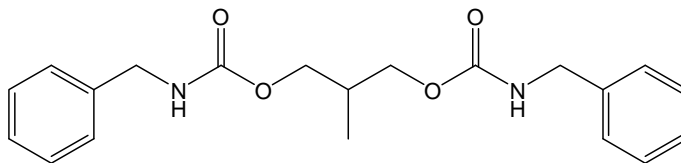
Monoisotopic Mass, Even Electron Ions

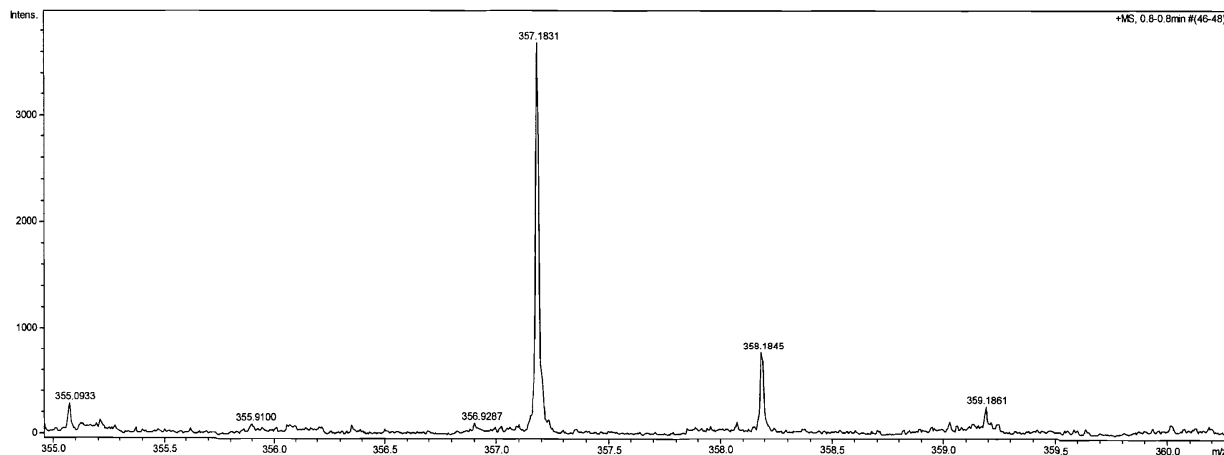
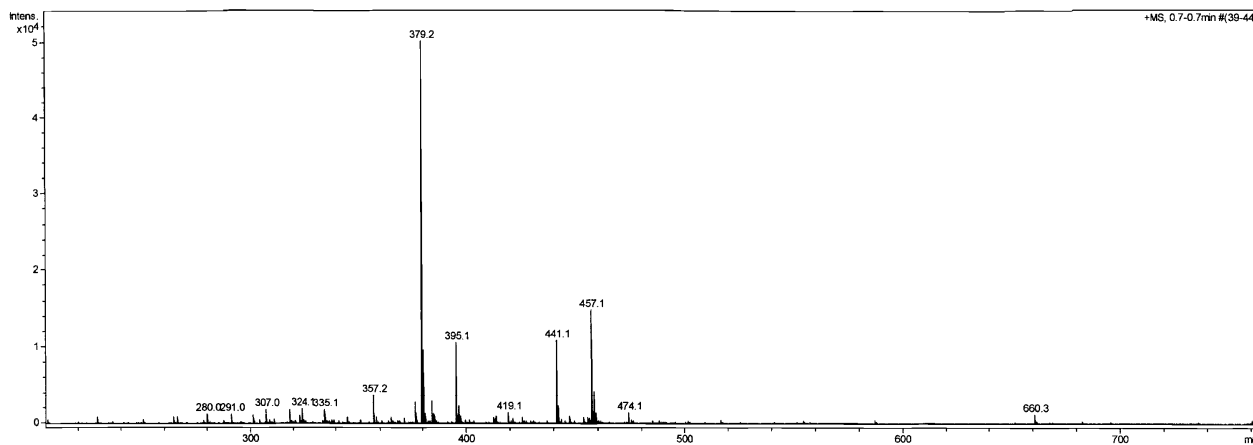
9054 formula(e) evaluated with 7 results within limits (up to 20 closest results for each mass)

Mass	Calc. Mass	mDa	PPM	DBE	Formula
304.1157	304.1161	-0.4	-1.3	5.5	12C14 1H19 14N 16O5 23Na
	304.1174	-1.7	-5.7	10.5	12C15 1H15 14N5 16O 23Na
	304.1134	2.3	7.5	6.5	12C10 1H15 14N7 16O3 23Na
	304.1062	9.5	31.2	10.5	12C16 1H15 14N3 16O2 23Na
	304.1273	-13.6	-38.2	5.5	12C13 1H19 14N3 16O4 23Na
	304.1022	13.5	44.5	6.5	12C11 1H15 14N5 16O4 23Na
	304.1008	14.9	48.9	1.5	12C10 1H19 14N 16O8 23Na

Sample was dissolved in methanol and was diluted in 50% methanol, 50% water and 0.1% formic acid. Then it was directly injected into the Bruker maXis mass spectrometer for analysis in the positive ESI mode. The observed peak at m/z of 304.1154 was within 3 ppm of the theoretical [M+Na]⁺ value (304.1161) in one run and the m/z of 304.1157 was within 2 ppm of the theoretical value in another run.

9 2-methylpropyl-1,3-dibenzylcarbamoyl





Elemental Composition Report

Single Mass Analysis (displaying only valid results)
 Tolerance = 50.0 PPM / DBE: min = -1.5, max = 50.0
 Isotope matching not enabled

Monoisotopic Mass, Even Electron Ions
 35229 formula(e) evaluated with 19 results within limits (up to 20 closest results for each mass)

Mass	Calc. Mass	mDa	PPM	DBE	Formula
357.1831	357.1814	1.7	4.7	9.5	12C20 H25 14N2 16O4
	357.1855	-2.4	-6.6	13.5	12C25 H25 16O2
	357.1873	-4.2	-11.8	0.5	12C13 H29 14N2 16O9
	357.1787	4.4	12.2	10.5	12C16 H21 14N8 16O2
	357.1886	-5.5	-15.5	5.5	12C14 H25 14N6 16O5
	357.1774	5.7	15.9	5.5	12C15 H25 14N4 16O6
	357.1900	-6.9	-19.3	10.5	12C15 H21 14N10 16O
	357.1761	7.0	19.7	0.5	12C14 H29 16O10
	357.1913	-8.2	-23.0	4.5	12C18 H29 16O7
	357.1747	8.4	23.4	6.5	12C11 H21 14N10 16O4
	357.1927	-9.6	-26.8	9.5	12C19 H25 14N4 16O3
	357.1734	9.7	27.2	1.5	12C10 H25 14N6 16O8
	357.1715	11.6	32.4	14.5	12C22 H21 14N4 16O
	357.1702	12.9	36.1	9.5	12C21 H25 16O5
	357.1967	-13.6	-38.0	13.5	12C24 H25 14N2 16O
	357.1985	-15.4	-43.2	0.5	12C12 H29 14N4 16O8
	357.1875	15.6	43.6	10.5	12C17 H21 14N6 16O3
	357.1999	-16.8	-47.0	5.5	12C13 H25 14N8 16O4
	357.1662	16.9	47.4	5.5	12C16 H25 14N2 16O7

Elemental Composition Report

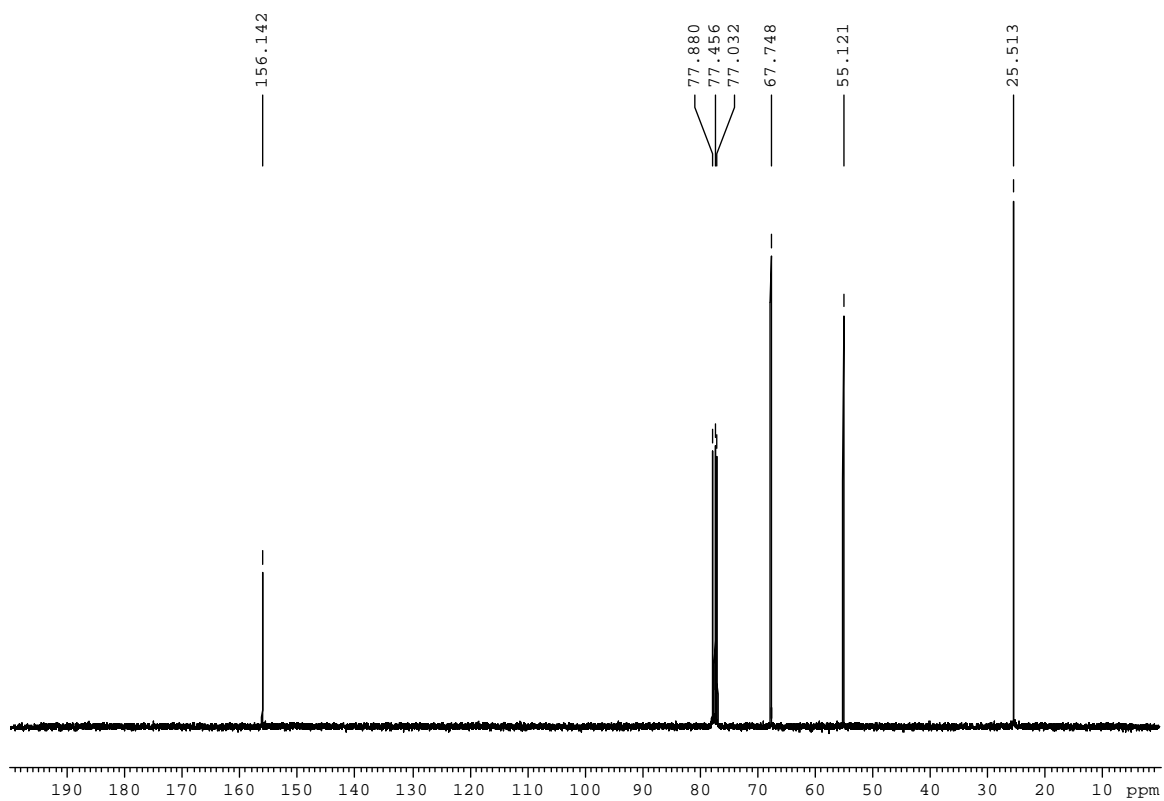
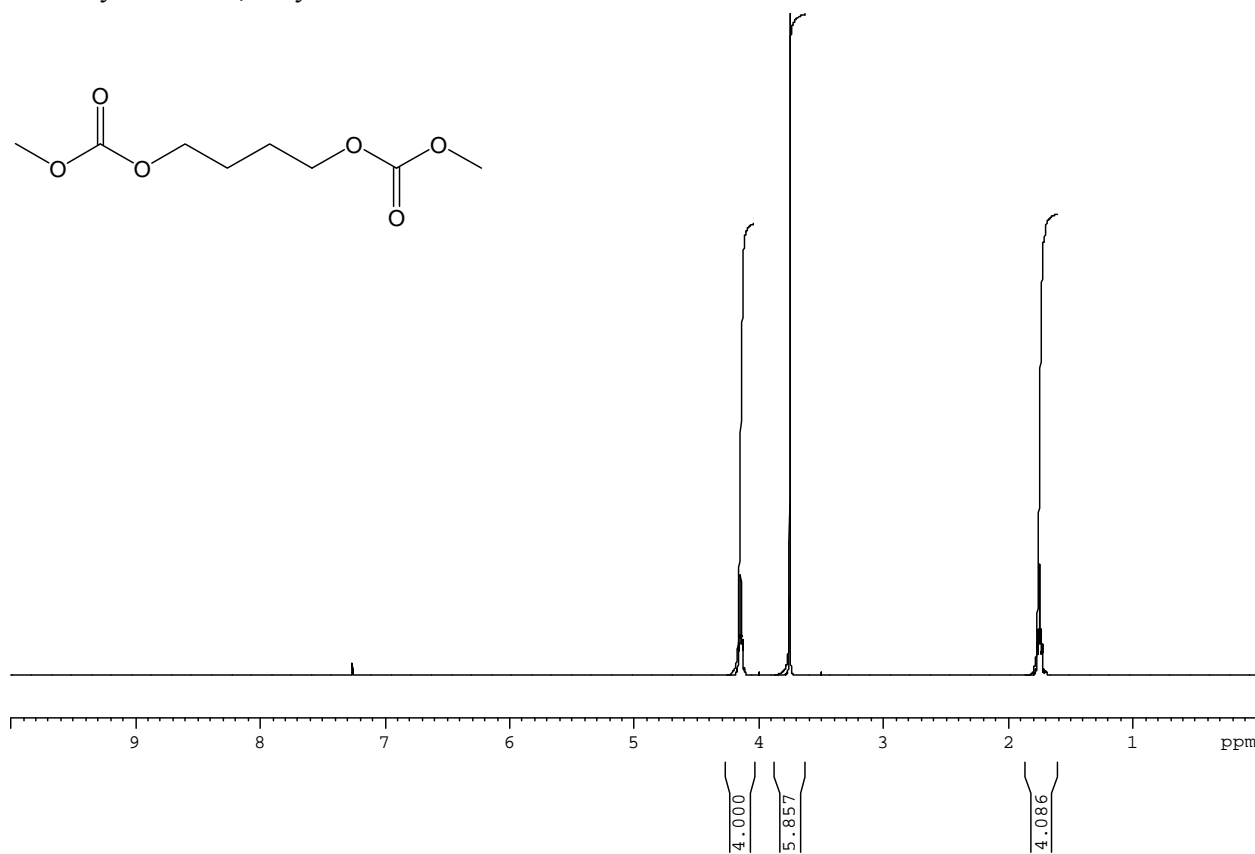
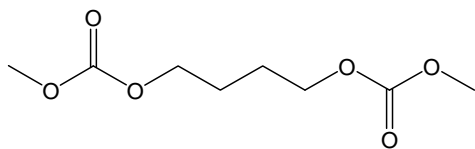
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 Isotope matching not enabled

Monoisotopic Mass, Even Electron Ions
 29754 formula(e) evaluated with 18 results within limits (up to 20 closest results for each mass)

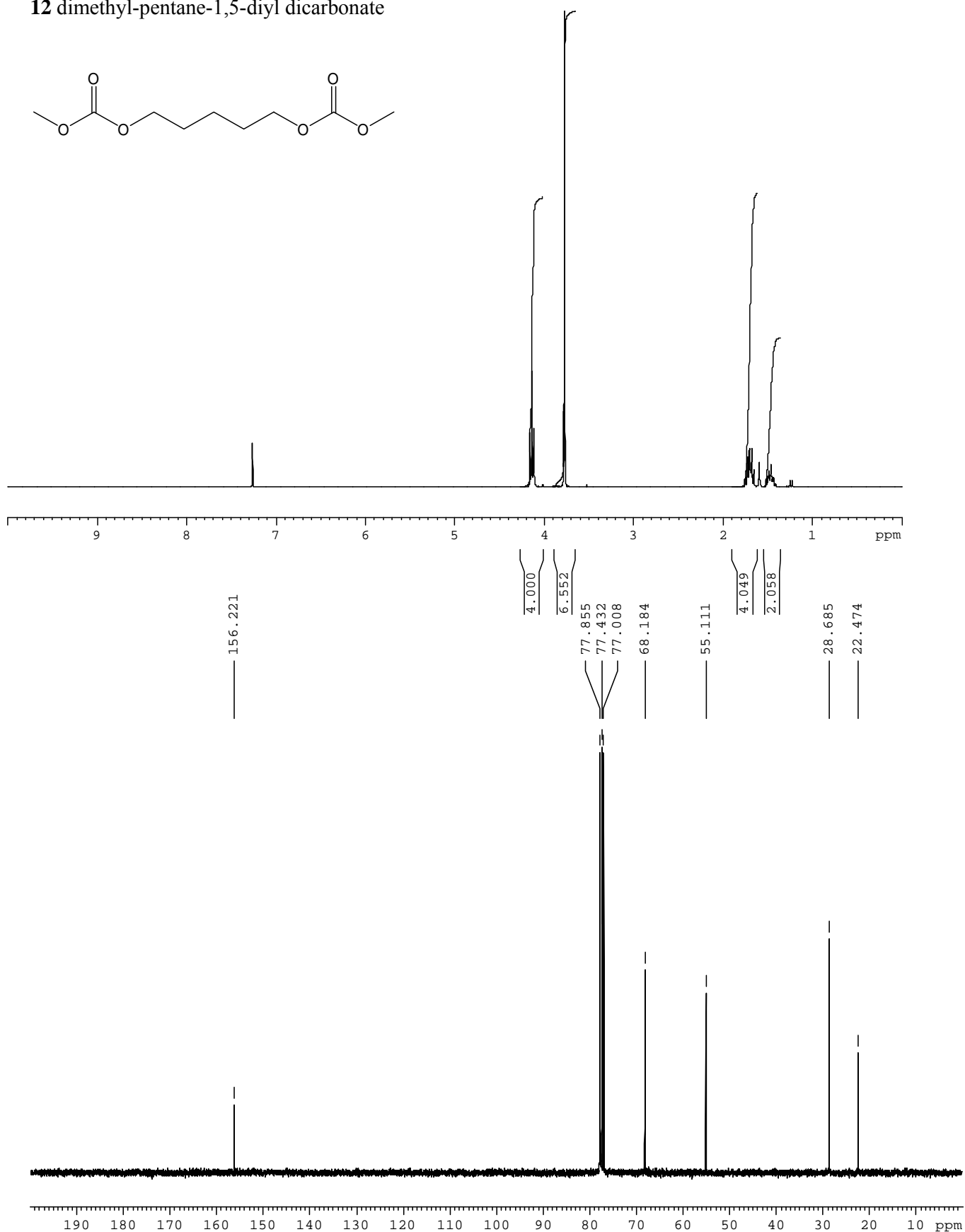
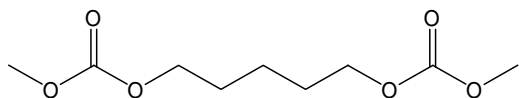
Mass	Calc. Mass	mDa	PPM	DBE	Formula
358.1845	358.1848	-0.3	-0.8	9.5	12C19 13C 1H25 14N2 16O4
	358.1821	2.4	6.7	10.5	12C15 13C 1H21 14N8 16O2
	358.1808	3.7	10.4	5.5	12C14 13C 1H25 14N4 16O6
	358.1888	-4.3	-12.0	13.5	12C24 13C 1H25 16O2
	358.1794	5.1	14.2	0.5	12C13 13C 1H29 16O10
	358.1907	-6.2	-17.2	0.5	12C12 13C 1H29 14N2 16O9
	358.1981	6.4	17.9	6.5	12C10 13C 1H21 14N10 16O4
	358.1920	-7.5	-20.9	5.5	12C13 13C 1H25 14N6 16O5
	358.1933	-8.8	-24.7	10.5	12C14 13C 1H21 14N10 16O
	358.1749	9.6	26.8	14.5	12C21 13C 1H21 14N4 16O
	358.1947	-10.2	-28.4	4.5	12C17 13C 1H29 16O7
	358.1736	10.9	30.6	9.5	12C20 13C 1H25 16O5
	358.1960	-11.5	-32.2	9.5	12C18 13C 1H25 14N4 16O3
	358.1709	13.6	38.1	10.5	12C16 13C 1H21 14N6 16O3
	358.1695	15.0	41.8	5.5	12C15 13C 1H25 14N2 16O7
	358.2000	-15.5	-43.4	13.5	12C23 13C 1H25 14N2 16O
	358.2019	-17.4	-48.6	0.5	12C11 13C 1H29 14N4 16O8
	358.1668	17.7	49.3	6.5	12C11 13C 1H21 14N8 16O5

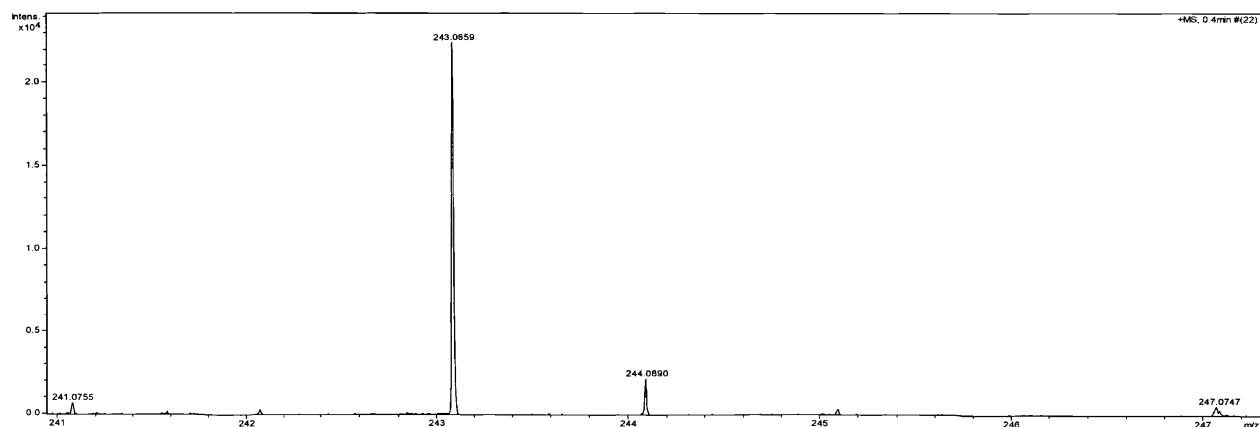
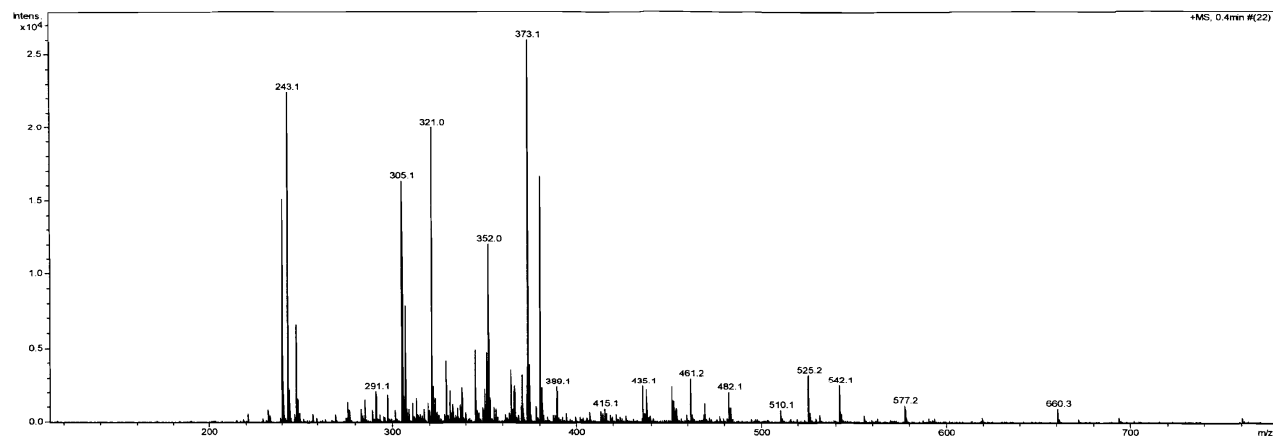
Sample was dissolved in methanol and was diluted in 50% methanol, 50% water and 0.1% formic acid. Then it was directly injected into the Bruker maXis mass spectrometer for analysis in the positive ESI mode. The observed peak at m/z of 357.1831 was within 5 ppm of the theoretical [M+H]⁺ value (357.1814), and the observed C13 peak at 358.1845 was within 1 ppm of the expected value (358.1848)

11 dimethyl-butane-1,4-diyl dicarbonate



12 dimethyl-pentane-1,5-diyl dicarbonate





Elemental Composition Report

Single Mass Analysis (displaying only valid results)
 Tolerance = 50.0 PPM / DBE: min = -1.5, max = 50.0
 Isotope matching not enabled

Monoisotopic Mass, Even Electron Ions
 2564 formula(e) evaluated with 2 results within limits (up to 20 closest results for each mass)

Minimum:								
Maximum:	30.0	50.0		-1.5	50.0			
Mass	Calc. Mass	mDa	PPM	DBE	Formula			
243.0859	243.0845	1.4	5.9	1.5	12C9	1H16	16O6	23Na
	243.0786	7.3	30.1	10.5	12C16	1H12	16O	23Na

Elemental Composition Report

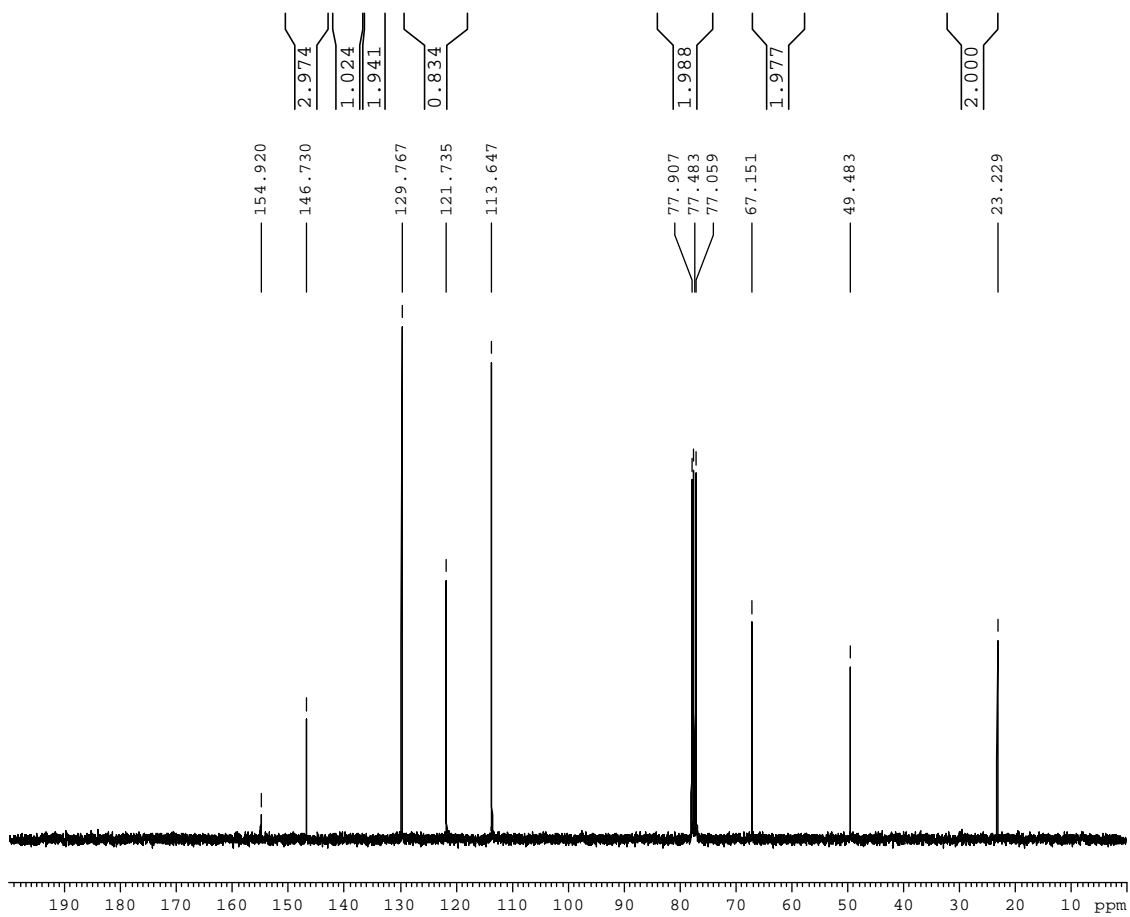
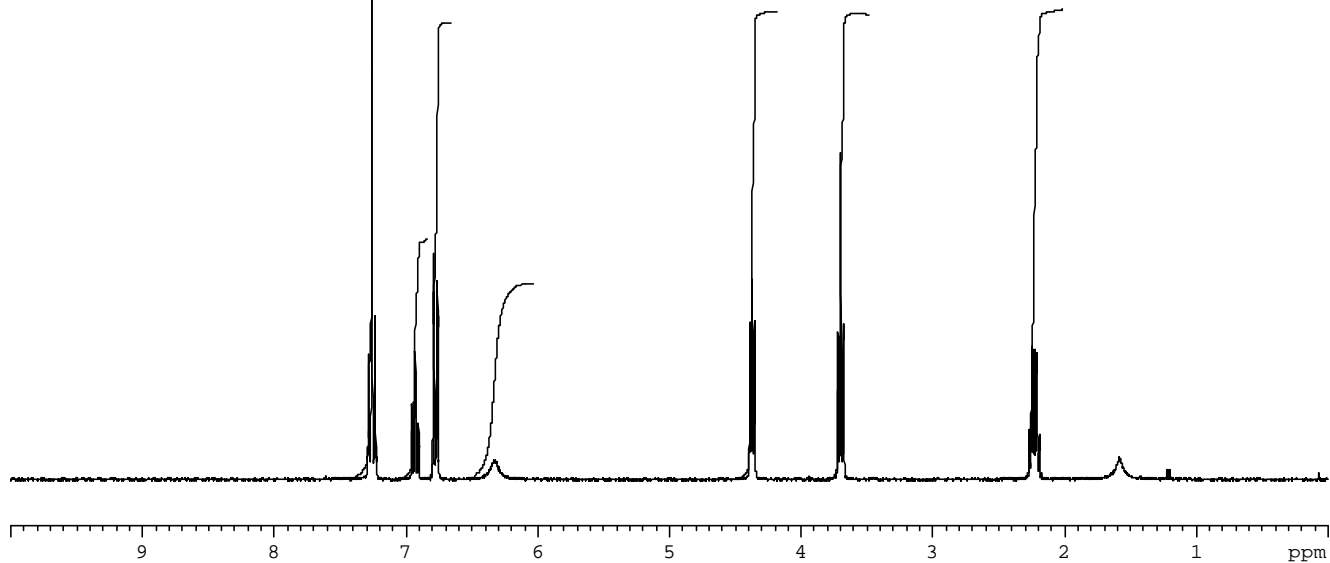
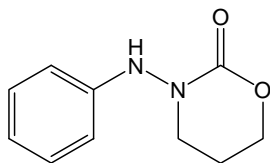
Single Mass Analysis
 Tolerance = 50.0 PPM / DBE: min = -1.5, max = 50.0
 Isotope matching not enabled

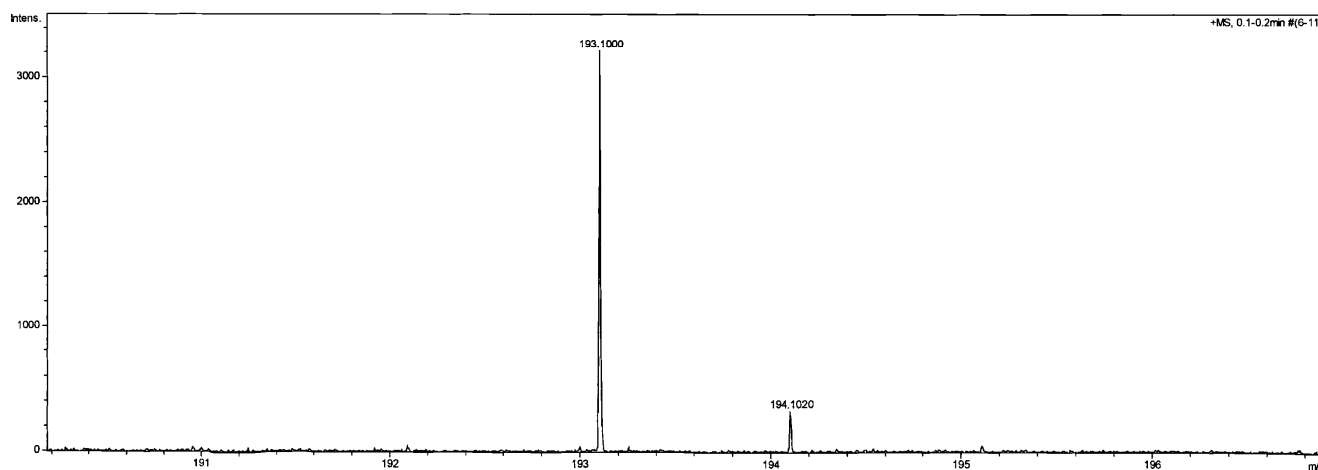
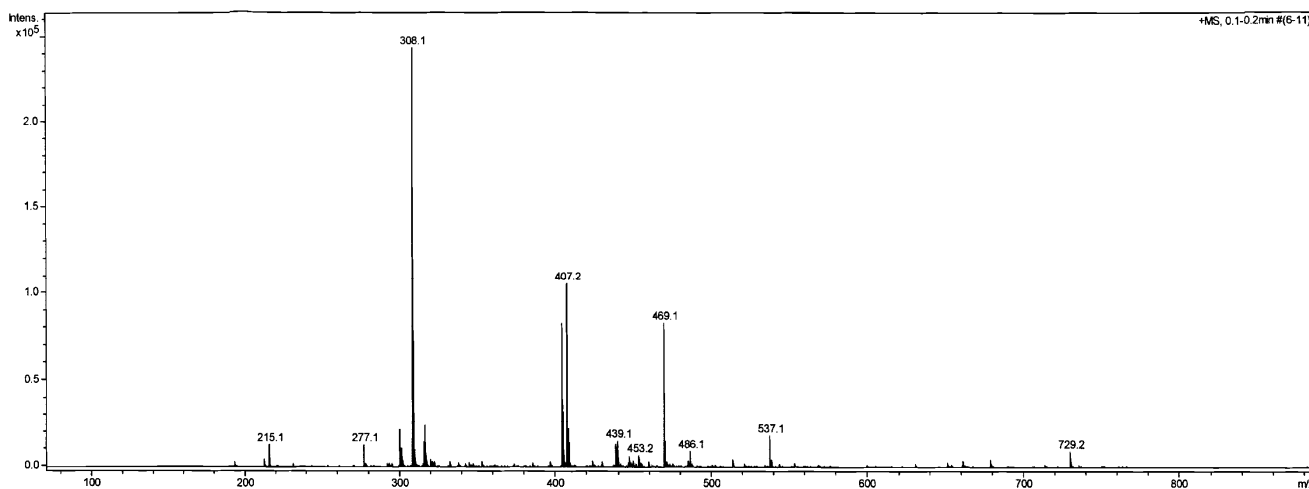
Monoisotopic Mass, Even Electron Ions
 2074 formula(e) evaluated with 2 results within limits (up to 20 closest results for each mass)

Minimum:									
Maximum:	30.0	50.0		-1.5	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	Formula				
244.0890	244.0878	1.2	4.9	1.5	12C8	13C	1H16	16O6	23Na
	244.0819	7.1	28.9	10.5	12C15	13C	1H12	16O	23Na

Sample was dissolved in methanol and was diluted in 50% methanol, 50% water and 0.1% formic acid. Then it was directly injected into the Bruker maXis mass spectrometer for analysis in the positive ESI mode. The observed peak at m/z of 243.0859 was within 6 ppm of the theoretical [M+N]⁺ value (243.0845), and the observed C13 peak at 244.0890 was within 5 ppm of the expected value (244.0878)

13 3-phenylamino-1,3-oxazinan-2-one





Elemental Composition Report

Single Mass Analysis (displaying only valid results)
 Tolerance = 100.0 PPM / DBE: min = -1.5, max = 50.0
 Isotope matching not enabled

Monoisotopic Mass, Even Electron Ions
 397 formula(e) evaluated with 2 results within limits (up to 10 closest results for each mass)

Mass	Calc. Mass	mDa	PPM	DBE	Formula
193.1000	193.0977	2.3	11.9	5.5	12C10 1H13 14N2 16O2
	193.0865	13.5	70.1	5.5	12C11 1H13 16O3

Elemental Composition Report

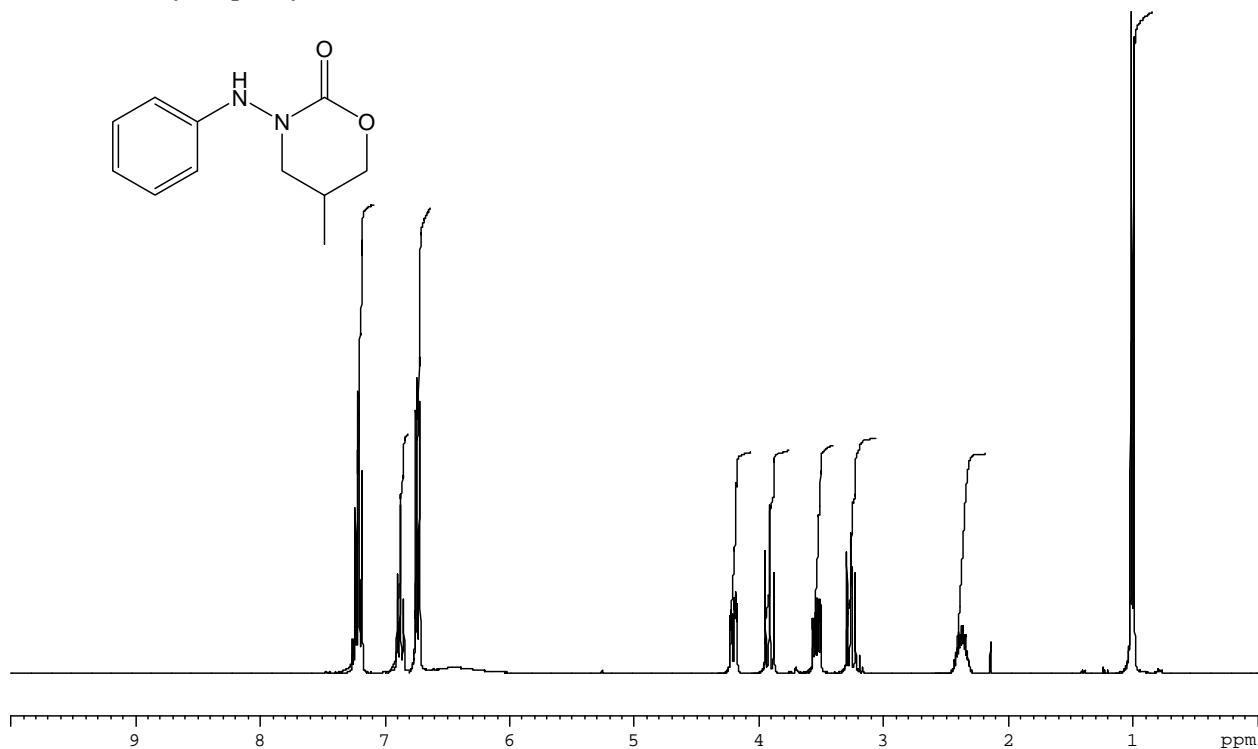
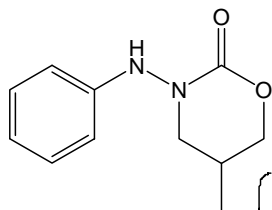
Single Mass Analysis
 Tolerance = 50.0 PPM / DBE: min = -1.5, max = 50.0
 Isotope matching not enabled

Monoisotopic Mass, Even Electron Ions
 3043 formula(e) evaluated with 2 results within limits (up to 20 closest results for each mass)

Mass	Calc. Mass	mDa	PPM	DBE	Formula
194.1020	194.1011	0.9	4.9	5.5	12C9 13C 1H13 14N2 16O2
	194.1110	-9.0	-46.1	0.5	12C7 13C 1H17 16O5

Sample was dissolved in methanol and was diluted in 50% methanol, 50% water and 0.1% formic acid. Then it was directly injected into the Bruker maXis mass spectrometer for analysis in the positive ESI mode. The observed peak at m/z of 193.1000 was within 12 ppm of the theoretical [M+H]⁺ value (193.0978), and the observed C13 peak at 194.1020 was within 5 ppm of the expected value (194.1012).

14, 5-methyl-3-phenylamino-1,3-oxazinan-2-one



2.134
1.091
2.119

1.007
1.015
1.039
1.070

1.000

3.007

154.803

146.748

129.712

121.395

113.659

78.026

77.602

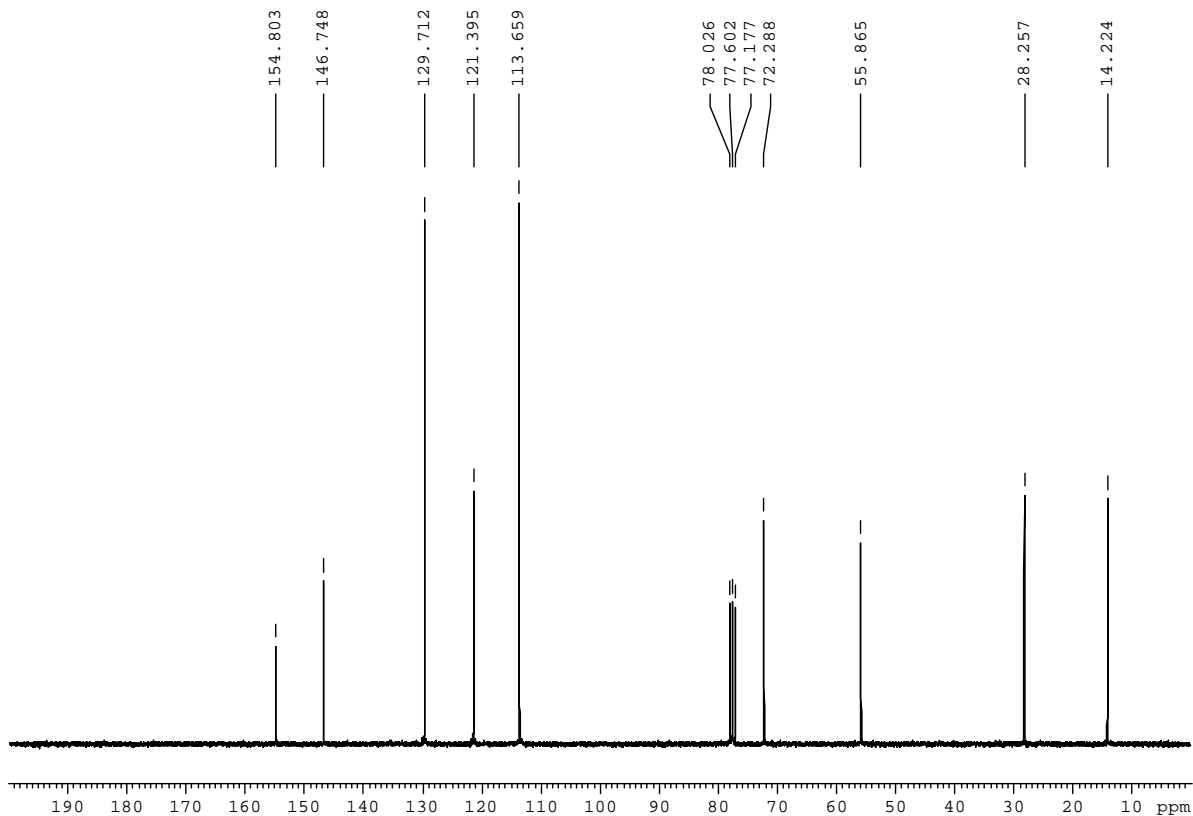
77.177

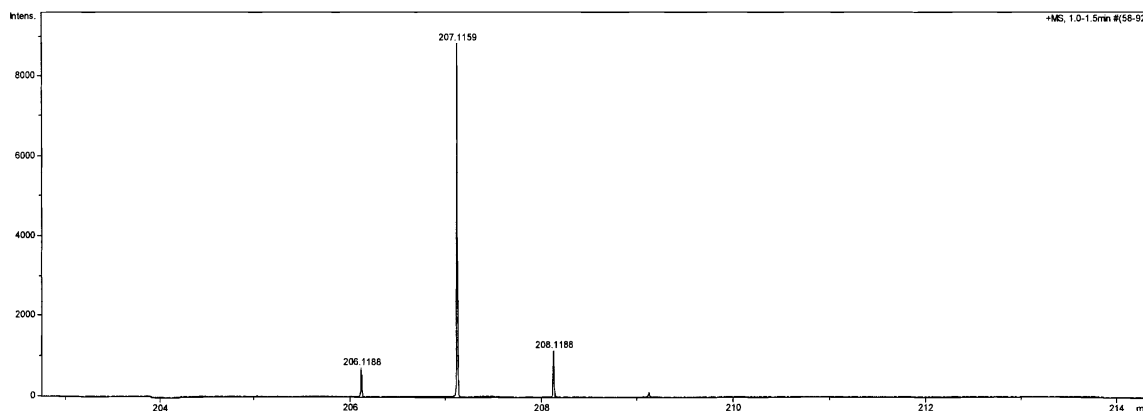
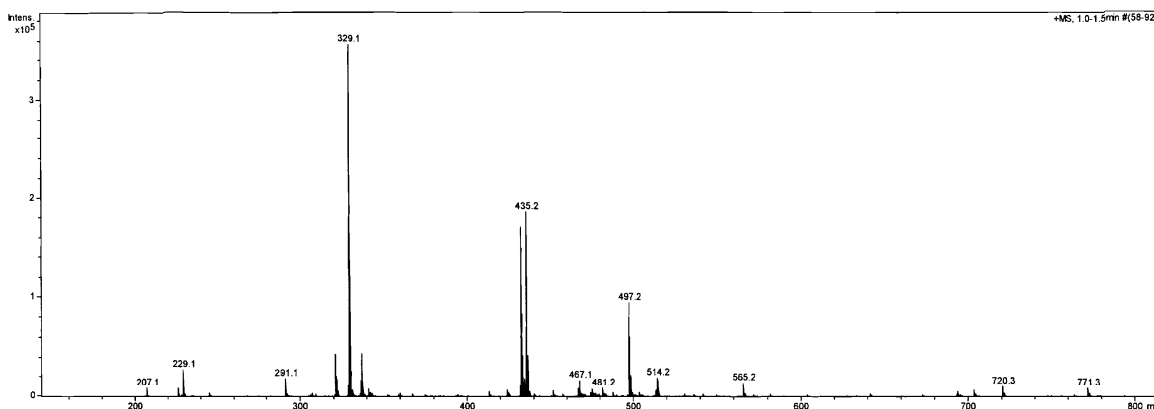
72.288

55.865

28.257

14.224





Elemental Composition Report

Single Mass Analysis (displaying only valid results)
 Tolerance = 100.0 PPM / DBE: min = -1.5, max = 50.0
 Isotope matching not enabled

Monoisotopic Mass, Even Electron Ions
 817 formula(e) evaluated with 3 results within limits (up to 10 closest results for each mass)

Mass	Calc. Mass	mDa	PPM	DBE	Formula
207.1159	207.1134	2.5	12.3	5.5	12C11 1H15 14N2 16O2
	207.1246	-8.7	-41.9	5.5	12C10 1H15 14N4 16O
	207.1021	13.8	66.5	5.5	12C12 1H15 16O3

Elemental Composition Report

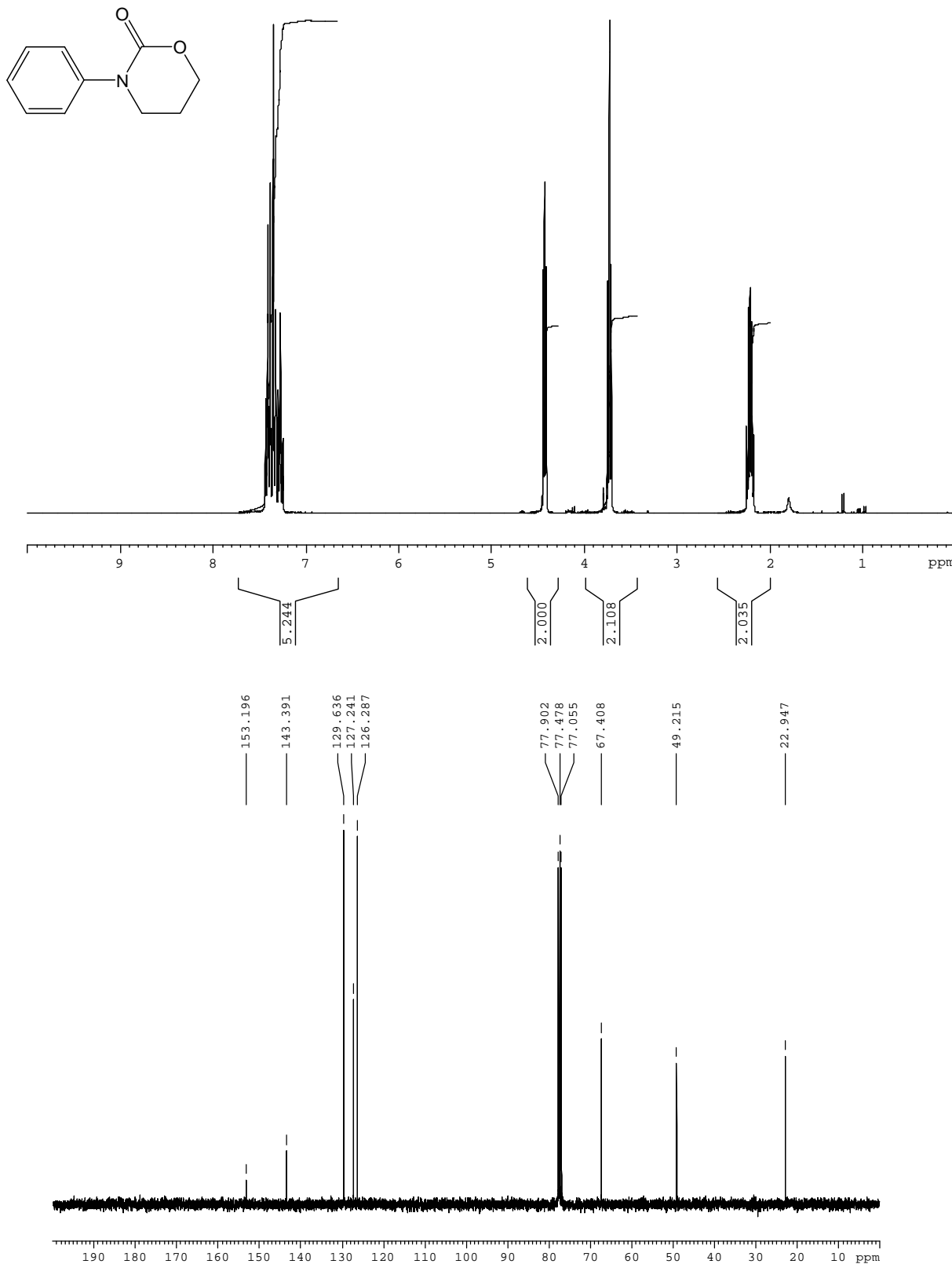
Single Mass Analysis
 Tolerance = 50.0 PPM / DBE: min = -1.5, max = 50.0
 Isotope matching not enabled

Monoisotopic Mass, Even Electron Ions
 4659 formula(e) evaluated with 4 results within limits (up to 20 closest results for each mass)

Mass	Calc. Mass	mDa	PPM	DBE	Formula
208.1191	208.1167	2.4	11.5	5.5	12C10 13C 1H15 14N2 16O2
	208.1127	6.4	30.8	1.5	12C5 13C 1H15 14N4 16O4
	208.1266	-7.5	-36.1	0.5	12C8 13C 1H19 16O5
	208.1279	-8.8	-42.5	5.5	12C9 13C 1H15 14N4 16O

Sample was dissolved in methanol and was diluted in 50% methanol, 50% water and 0.1% formic acid. Then it was directly injected into the Bruker maXis mass spectrometer for analysis in the positive ESI mode. The observed peak at m/z of 207.1159 was within 12 ppm of the theoretical [M+H]⁺ value (207.1134), and the observed C13 peak at 208.1188 was within 10 ppm of the expected value (208.1168).

15 3-phenyl-1,3-oxazinan-2-one



16 5-methyl-3-phenyl-1,3-oxazinan-2-one

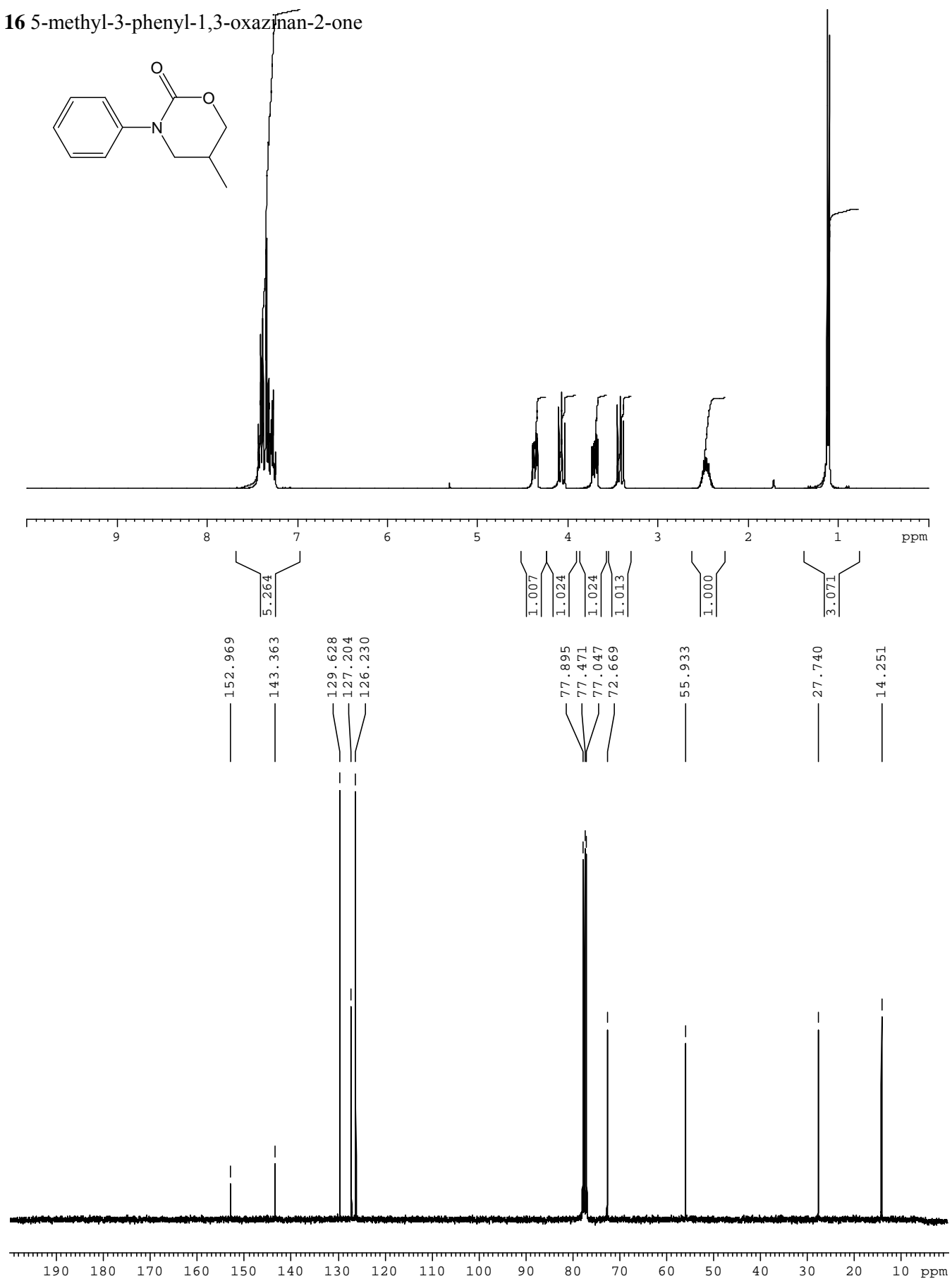
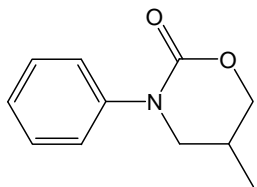


Table 3. Crystal Data and Structure Parameters for Compounds **13-16**

Compound	13	14	15	16
empirical formula	C ₁₀ H ₁₂ N ₂ O ₂	C ₁₁ H ₁₄ N ₂ O ₂	C ₁₀ H ₁₁ NO ₂	C ₁₁ H ₁₃ NO ₂
formula weight	192.22	206.24	177.20	191.22
crystal system	monoclinic	monoclinic	monoclinic	orthorhombic
space group	P2 ₁ /a	P2 ₁ /c	P2 ₁ /c	Fdd2
<i>a</i> /Å	9.508(2)	11.734(3)	5.781(2)	24.356(3)
<i>b</i> /Å	10.527(3)	9.003(2)	12.671(3)	27.958(3)
<i>c</i> /Å	10.065(2)	11.714(3)	12.500(3)	5.831(2)
β /deg	104.47(3)	114.71(3)	100.76(3)	-
Volume (Å ³)	975.5(4)	1124.2(5)	899.5(4)	3971(1)
<i>Z</i>	4	4	4	16
ρ_{calcd} (g cm ⁻³)	1.309	1.219	1.309	1.280
μ (Mo-K α) (mm ⁻¹)	0.093	0.085	0.092	0.088
<i>F</i> (000)	408	440	2112	1632
No. reflns collected	1794	2066	1649	967
No. reflns used [<i>I</i> \geq 2 σ (<i>I</i>)]	1537	1554	1435	918
R = $\sum F_o - F_c / \sum F_o $	0.066	0.072	0.053	0.073
R _w = $\{ \sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2] \}^{1/2}$	0.176	0.186	0.139	0.198