Supplemental Material

Regiospecific Epoxidation of Carvone: A Discovery-Oriented Experiment for Understanding the Selectivity and Mechanism of Epoxidation Reactions

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Students' Handout

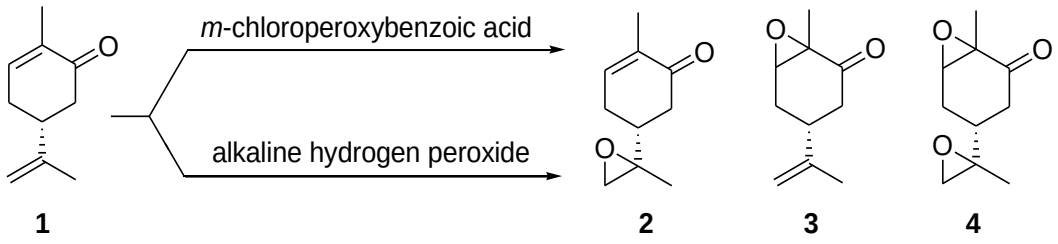
Introduction

This is a discovery-oriented experiment in which you will investigate the selectivity and the mechanism of two commonly used epoxidation reactions:

Epoxidation of alkenes by peroxy acids

Epoxidation of alkenes by alkaline H_2O_2

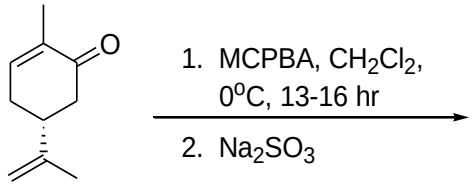
(*R*)-(-)-Carvone (**1**) is the major constituent of the essential oil isolated from spearmint. This compound has two C=C bonds, one is located at the isopropenyl group at the side chain, and the other constitutes the α,β-unsaturated ketone of the cyclohexenone moiety. The first one is an electron-rich C=C bond while the latter is electron-deficient. Both of the alkene groups can undergo epoxidation reactions to give epoxides, but may need different reagents or conditions. In this experiment you will use (*R*)-(-)-carvone as the substrate to investigate the selectivity and the mechanisms of the peroxy acid epoxidation and the alkaline H_2O_2 epoxidation.



Firstly you will carry out the epoxidation reactions of carvone with *m*-chloroperoxybenzoic acid and alkaline H_2O_2 respectively. Then you will have to identify the reaction products and evaluate the reaction selectivity by thin-layer chromatography (qualitative) and ^{1}H NMR spectroscopy (quantitative). Finally, you are required to propose explanations for the observed selectivity in terms of their respective reaction mechanisms.

Part A: Epoxidation of (*R*)-(-)-Carvone by *m*-Chloroperoxybenzoic Acid (MCPBA)

A.1 Synthesis *(1)*



Caution: Put on proper protective gloves and safety spectacles when performing this experiment. (R)-(-)-Carvone is a mildly toxic volatile organic liquid. Handle this compound in a fumehood and avoid contacting with skin. m-Chloroperoxybenzoic acid (MCPBA) is shock sensitive. Commercial MCPBA contains approx. 25% of water as stabilizer. This substance should be stored in a refrigerator and never be dried or ground. Dichloromethane is a very volatile organic solvent and inhalation should be avoided.

To a solution of (*R*)-(-)-carvone (0.5 g, 3.33 mmol) in CH_2Cl_2 (8 mL) cooled at 0^oC, add a solution of 75 % *m*-chloroperoxybenzoic acid (MCPBA) (0.85 g, 3.69 mmol) in 4 mL of CH_2Cl_2 dropwise over a period of 10 min. Stir the reaction mixture at 0°C for 13-16 hr. When the reaction is completed, add 1 mL of 10% aqueous sodium sulfite solution and stir the mixture for 1-2 minutes. Filter the mixture and wash the solid residue with several small portions of CH_2Cl_2 and combine the washes with the filtrate. Wash the combined organic liquid successively with 10% Na_2CO_3 solution (3 x 15 mL) and saturated NaCl solution (15 mL). Dry the organic solution with anhydrous $MgSO_4$ and filter the mixture. Concentrate the organic solution by rotary evaporation.

A.2 Qualitative analysis (by thin-layer chromatography)

Caution: Phosphomolybdic acid is oxidizing and toxic. Handle it with care. Hexane and ethyl acetate are flammable solvents and should be kept away from ignition sources.

Dissolve a small amount of the crude product (a few mg will be enough) in 3-5 mL of dichloromethane. Apply the sample solution to a piece of TLC plate with a capillary. Spot (*R*)-(-)-carvone onto the TLC plate for comparison as well. Develop the TLC plate in a TLC chamber using hexane/ethyl acetate (10:1) as the eluent. Remove the TLC

plate from the chamber and allow the solvent to evaporate completely. Visualize the TLC plate by immersing it briefly into a 3% ethanolic solution of phosphomolybdic acid and then heat it with a hot-air gun. Analyze the composition of the product mixture.

TLC Plates: Merck Silica Gel 60 F_{254} TLC on aluminium backing sheets (Product No. 105554)

A.3 Structural characterization and quantitative analysis (by ^{1}H NMR spectroscopy) *(1,2)*

Caution: Chloroform-d ($CDCl_3$) is a mutagen and a cancer suspect agent. Handle it with great care.

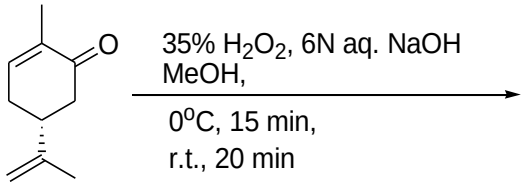
Dissolve about 10 mg of the crude product in 0.6 mL of $CDCl_3$ in a 5 mm NMR tube *(Wilmad-528-PP or Wilmad-509-PP)* and obtain the ^{1}H NMR spectrum. Analyze the spectrum and identify the reaction product(s). Determine which of the alkene groups is converted into an epoxide. Evaluate the regioselectivity of the reaction from the integration of the NMR signals.

**If high-resolution NMR spectrometers are not available, you may either use the spectrum provided in the Supplemental Material from JCE Online, or obtain a copy of the spectrum from your instructor.*

[Hint: Compare the spectrum with that of (R)-(-)-carvone, and identify which of the vinyl proton signals disappeared.]

Part B: Epoxidation of *(R)*-(-)-Carvone by Alkaline H_2O_2

B.1 Synthesis *(3,4)*



Caution: Proper protective gloves and safety spectacles must be put on. (R)-(-)-Carvone is a mildly toxic volatile organic liquid. 35% H_2O_2 is corrosive and can cause painful blistering to skin. 6N NaOH solution is also corrosive. Handle these compounds very carefully and avoid contacting them with skin. Dichloromethane is a very volatile organic solvent and inhalation should be avoided.

Dissolve *(R)*-(-)-carvone (0.72 g, 4.8 mmol) in 8 ml of methanol. Cool the mixture to 0°C and add 1.5 mL of 35% H_2O_2. With stirring, add 1 mL of 6N aq. NaOH solution over a period of 1-2 minutes. Stir the mixture at 0°C for 15 minutes and then at room temperature for 20 minutes.

Dissolve the mixture in 10 mL of CH_2Cl_2 and wash the organic solution twice with water (10 mL x 2). Wash the organic solution with saturated NaCl solution and then dry it with anhydrous $MgSO_4$. Filter the mixture and concentrate the organic solution by rotary evaporation.

B.2 Qualitative analysis (by thin-layer chromatography)

Similar to Part A.2

B.3 Structural characterization and quantitative analysis (by ^{1}H NMR spectroscopy) *(3,4)*

Similar to Part A.3

Part C: Relationships between Reaction Selectivity and Reaction Mechanism.

1. Evaluate the selectivity (qualitatively or quantitatively) of the two epoxidation reactions.
2. Propose detailed reaction mechanisms for the two epoxidation reactions.
3. Correlate and discuss the relationship between the observed reaction reactivity and the proposed reaction mechanism.

Supplementary Review Question

1. Many signals in the ^{13}C NMR spectrum of carvone-7,8-oxide appear as pairs of nearly coincident signals. Briefly explain this observation.

Literature Cited

1. Baldwin, J. E.; Broline, B. M. *J. Am. Chem. Soc*. **1982**, *104*, 2857-2865.
2. Smitt, O.; Högberg, H.-E. *Tetrahedron* **2002**, *58*, 7691-7700.

3. Muralidharan, K. R.; de Lera, A. R.; Isaeff, S. D.; Norman, A. W.; Okamura, W. H. *J Org. Chem.* **1993**, 58, 1895-1899.
4. McChesney, J. D.; Thompson, T. N. *J. Org. Chem.* **1985**, *50*, 3473-3481.

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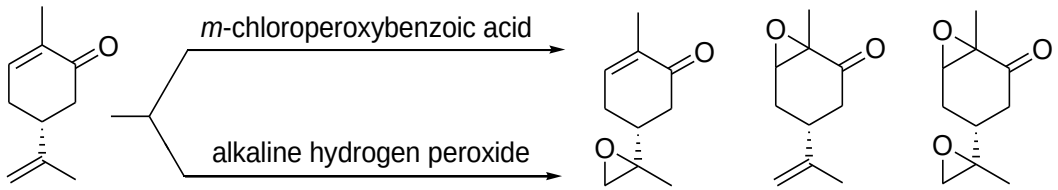
Instructor's Notes

Introduction



1 **2** **3** **4**

This experiment is suitable for organic chemistry courses of intermediate or advanced level. In this experiment students are required to perform the epoxidations of (*R*)-(-)-carvone with two different methods, and then determine the selectivity of these two reactions and investigate the relationship between the reaction mechanisms and the observed selectivity. This is a discovery-oriented experiment which aims at enhancing students' knowledge of the important features of different epoxidation reactions, and consolidating their understanding of the relationship between the mechanism and selectivity of organic reactions. In contrast to the traditional cook-book experiments, this experiment can arouse students' interests in solving chemistry problems and increase their appreciations of the importance of planning in organic synthesis.

Tasks

Students have to complete the following tasks in the experiment:

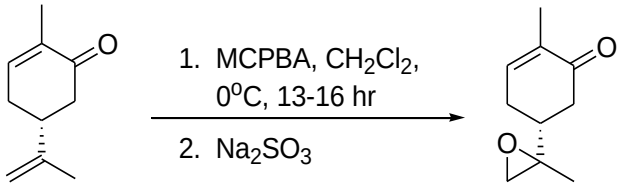
1. Carry out the epoxidation of (*R*)-(-)-carvone with MCPBA.
2. Carry out the epoxidation of (*R*)-(-)-carvone with alkaline H_2O_2.
3. Analyze the reaction mixtures by thin-layer chromatography. (qualitative analysis)
4. Identify the reaction products by ^1H NMR spectroscopy and analyze the reaction selectivity quantitatively.
5. Discuss the relationship between the reaction mechanisms and the observed selectivity.

Time required for the experiment

This experiment can be completed in one or two 3-4 hour laboratory sessions. Since the MCPBA epoxidation needs 13-16 hours of reaction time, instructor may ask students to come to the laboratory on the day before and set up the reaction which may require about half of an hour. To maintain the reaction mixture at 0°C, keep the reaction mixture in a refrigerator which was set to 0-2°C for overnight. The students then come back on the next day to work-up the mixture of the MCPBA epoxidation and perform the alkaline H_2O_2 epoxidation. In this case only one 3-4 hour lab session is formally needed.

Suggestions to instructors (Experimental)

Part A: Epoxidation of (*R*)-(-)-Carvone by *m*-Chloroperoxybenzoic Acid



1 carvone-7,8-oxide (**2**)

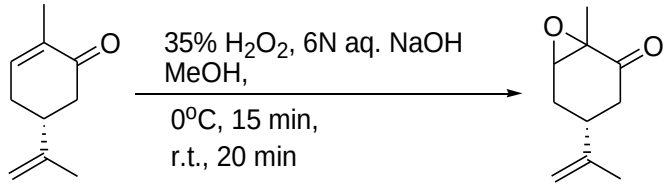
The epoxidation of (*R*)-(-)-carvone by *m*-chloroperoxybenzoic acid was adopted from the literature procedure *(1).* Carvone-7,8-oxide (**2**) was obtained as a colorless oily liquid with an average yield of 78%. This experiment had been performed in a class of 44 junior undergraduate students and the yields obtained were ranging from 57% to 91%. The reaction was regiospecific and no carvone-1,2-oxide (**3**) was present in the product mixture. The compound was structurally characterized by ^{1}H NMR, ^{13}C NMR, IR spectroscopy, and mass spectrometry. The reaction was clean and column chromatographic separation was unnecessary to obtain reasonably clean NMR spectra *(1,2).* The product was obtained as a mixture of stereoisomers in a ratio of 1:1.33 as indicated in the ^{1}H NMR spectrum *(1).* The stereoisomers have non-distinguishable R_f values on TLC and therefore separating these stereoisomers by chromatographic methods was not attempted.

Either (*R*)-(-)-carvone and (*S*)-(+)-carvone can be used for the experiment. (*R*)-(-)-carvone was chosen because of its lower cost.

The reaction mixture has to be kept at 0°C for 13-16 hours. In our class the mixtures were stirred in ice baths for a few hours and then placed in a refrigerator which was set to 0-2 °C for overnight. A large amount of unidentified side-products would be obtained if the reaction was allowed to proceed at ambient temperature.

The work-up procedure is straightforward. Sodium sulfite solution was added to reduce the excess peroxy acid and the product mixture was washed with 10% Na_2CO_3 to remove the by-product *m*-chlorobenzoic acid. TLC analysis showed only one epoxide product was formed in the mixture. Instructors may provide authentic sample solutions of the epoxycarvones **2** and **3** to the students for comparison, or ask them to identify the product by NMR spectroscopy. The R_f values of **2** and **3** in hexane/ethyl acetate (10:1) are 0.46 and 0.26, respectively. For the part of spectroscopic analysis, instructors may either allow students to obtain their own NMR spectra if spectrometers are available, or provide copies of the spectra to the students for analyzing the reaction selectivity. Alternatively, students may characterize the reaction products by IR spectroscopy.

Part B: Epoxidation of (*R*)-(-)-Carvone by Alkaline H_2O_2



1 carvone-1,2-oxide (**3**)

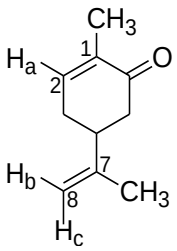
The procedure was adopted from the literature procedure and is straightforward *(3,4)*. Students should be reminded to be very careful when handling the highly corrosive H_2O_2 and NaOH solutions. Vigorous stirring is essential to obtain a good reaction yield. In our trials the reaction yields obtained for compound **3** were ranging from 57% to 91% and with an average of 80%. Again, TLC analysis showed only one product was formed in the mixture. The reaction was clean that the product was sufficiently pure for spectroscopic analysis without chromatographic purification.

Spectroscopic analysis

^{1}H NMR, ^{13}C NMR, IR, and mass spectra of the epoxycarvone products are provided at the end of this *Supplemental Material*. The IR and NMR spectra of *(R)*-(-)-carvone (**1**) are also provided for comparison.

(R)-(-)-Carvone (**1**)

The NMR and IR data of **1** are summarized below:

	^{1}H NMR (300 MHz, $CDCl_3$) δ 1.76 (s, 3H), 1.79 (s, 3H), 2.24 – 2.70 (m, 5H), 4.76 (s, 1H), 4.81 (d, 1H, *J* = 1.24 Hz), 6.75 – 6.78 (m, 1H)
	^{13}C NMR (75.5 MHz, $CDCl_3$) δ 15.6, 20.5, 31.2, 42.4, 43.0, 110.4, 135.3, 144.6, 146.6, 199.7
	IR (neat) 3083, 3021, 2971, 2951, 2922, 2887, 1675, 1645, 1450, 1435, 1367, 1111, 899 cm^{-1}

For the ^{1}H NMR spectrum, the multiplet at δ = 6.75 – 6.78 was assigned to the β-proton of the enone (H_a), and the two signals at 4.76 and 4.81 ppm were assigned to the two vinyl protons H_b and H_c, respectively. These three ^{1}H resonances are very useful for characterizing the structures of compounds **2** and **3**. The coupling constants of geminal vinyl protons are usually very small (0-2 Hz) and the splitting of the signals at 4.76 ppm was not resolved in the spectrum. The multiplets at 2.24 – 2.70 ppm were assigned to the protons on the cyclohexenone ring. Finally the two singlet signals at δ = 1.76 and 1.79 ppm were assigned to the two methyl groups attached to C-1 and C-7.

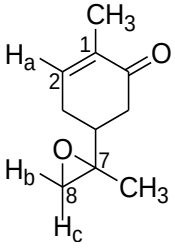
The ^{13}C signal at 199.7 ppm was assigned to the carbonyl carbon atom. The signals at 110.4, 135.3, 144.6 and 146.6 ppm were assigned to the carbon atoms C-8, C-1, C-2 and C-7, respectively. The assignments were made with reference to the typical values of ^{13}C resonances listed in the textbook and cross-referenced with the ^{13}C spectra obtained for compound **2** and **3**. The other five signals between 15.6 and 43.0 ppm were collectively assigned to the three sp^3 carbon atoms of the cyclohexenone ring and the two methyl groups attached at C-1 and C-7.

(R)-(-)-Carvone has several characteristic IR absorption bands in the double bond region. The absorption bands at 1675 and 1645 cm^{-1} were respectively assigned to the

enone C=O and the isopropenyl C=C bond. The C=O band was red-shifted from the typical C=O stretching of 1710 cm^{-1} to 1675 cm^{-1} because of the enone conjugation. The enone C=C absorption band was not observed because the intensity of alkene absorption in *s*-trans enone is usually very low.

Carvone-7,8-oxide (2)

The product obtained from the MCPBA epoxidation was identified as carvone-7,8-oxide (**2**) and the NMR and IR data are summarized in the table shown below. The NMR data are consistent with the literature values *(1,2).* The ^{1}H NMR data suggested a mixture of diastereoisomers were formed in the ratio of 1:1.33, as indicated by the pair of methyl signals at 1.32 and 1.33 ppm and some other signals which also appeared as two nearly coincident resonances. The alkene protons signals of the isopropenyl group at δ = 4.76 and 4.81 ppm in the spectrum of *(R)*-(-)-carvone were not present in the spectrum of carvone-7,8-oxide, which suggested that the epoxidation occurred preferentially at the isopropenyl side chain. The ^{1}H signals of the CH_2 group of the newly formed epoxide ring appeared at 2.58 – 2.72 ppm, and were only barely distinguishable as they are partially overlapped with the multiplet signals of the cyclohexenone protons (figure 1). Similarly, many of the ^{13}C signals appeared as pairs of nearly coincident resonances due to the formation of diastereomers. The two ^{13}C signals which corresponded to C-7 and C-8 of the isopropenyl group of *(R)*-(-)-carvone were transformed into the epoxide carbon resonances at 52.4/52.9 and 57.8/57.9 ppm (figure 2).

	^{1}H NMR (300 MHz, $CDCl_3$) δ*1.32/1.33 (s, 3H), 1.77-1.78 (m, 3H), 2.18 – 2.61 (m, 6H), *2.68/2.72 (2, 1H, *J* = 4.5 Hz), 6.73 – 6.76 (m, 1H)
	^{13}C NMR (75.5 MHz, $CDCl_3$) δ 15.6, *18.3/19.0, #39.9/40.3/40.7/41.3, *52.4/52.9, *57.8/57.9, 135.6, *143.9/144.1, 198.8
	IR (neat) 3053, 2980, 2924, 1675, 1451, 1435, 1384, 1366, 1108, 904, 736 cm^{-1}

** Signals appear in pairs due to the formation of diastereomers.*

 # Two overlapped pairs of diastereomeric signals.

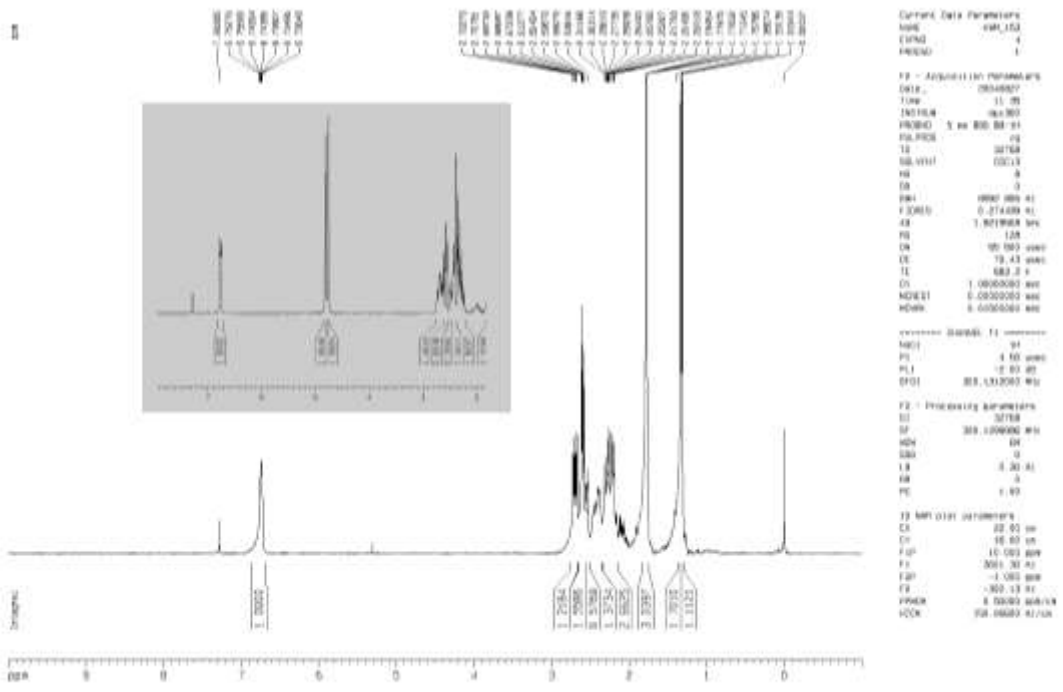


Figure 1. ^{1}H NMR spectrum of **2**. The insert spectrum shown in the gray box shows the partial ^{1}H NMR spectrum of *(R)*-(-)-carvone for comparison.

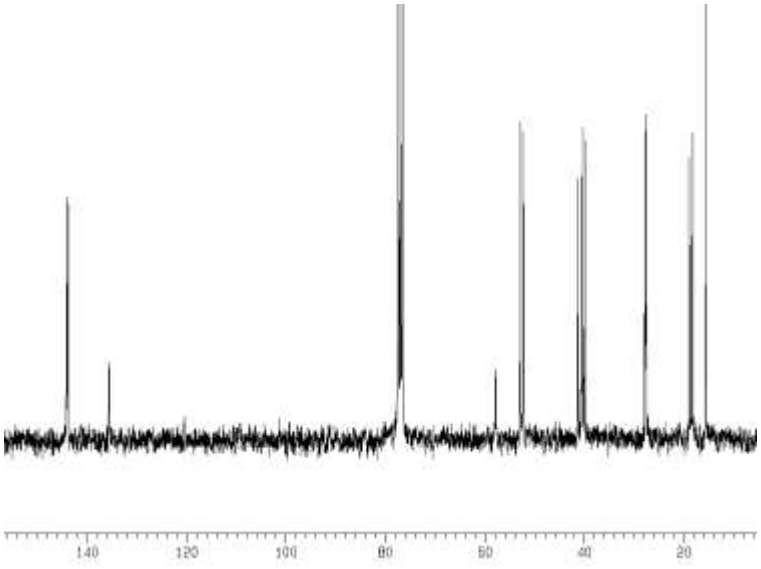


Figure 2. Partial ^{13}C NMR spectrum of **2**.

The IR spectrum obtained for the MCPBA reaction product also showed the

epoxidation occurred selectively at the isopropenyl side-chain. Figure 3 shows the partial IR spectra of the double bond region obtained for compounds **1** – **3**. In figure 3(b), the C=O absorption band remained at 1675 cm^{-1} but the absorption band at 1645 cm^{-1} which corresponded to the isopropenyl C=C bond was absent. It suggested that the enone moiety remained intact but the isopropenyl C=C bond was reacted.

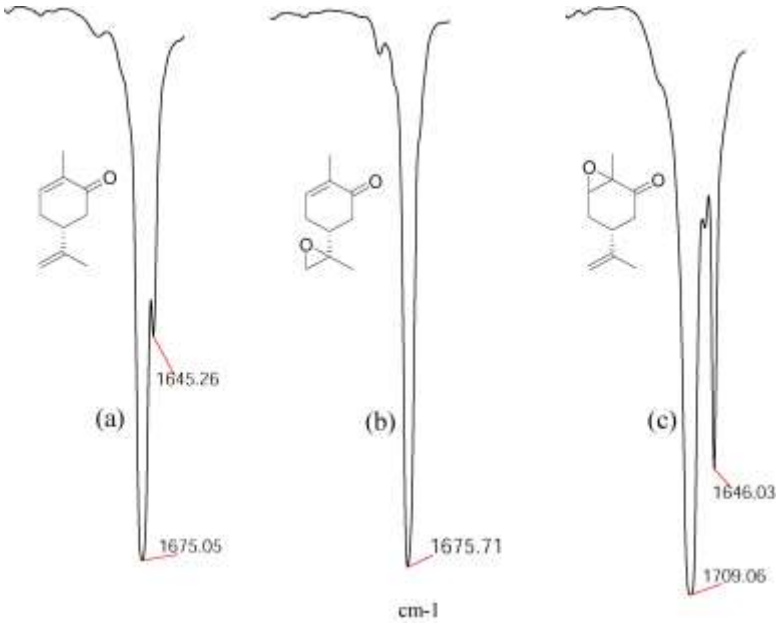
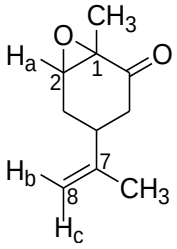


Figure 3. Partial IR spectra showing the C=O and C=C bond region for compounds **1** – **3**.

The product was also characterized by mass spectrometry. The spectrum showed a molecular ion peak at m/z equaled to 167, which corresponded to the $[M+1]^+$ peak of compound **2** ($C_{10}H_{14}O_2$, MW = 166.10).

Carvone-1,2-oxide (3)

The product obtained from the alkaline H_2O_2 epoxidation was identified as carvone-1,2-oxide (**3**). The NMR and IR data are summarized in the table below. The data were found to be consistent with the literature values *(3,4).*

	^{1}H NMR (300 MHz, $CDCl_3$) δ 1.41 (s, 3H), 1.72 (s, 3H), 1.91 (dd, 1H, *J* = 14.8, 11.1), 2.03 (dd, 1H, *J* = 17.6, 11.5 Hz), 2.38 (d-*distorted*, 1H, *J* = 14.8 Hz), 2.59 (dd, 1H, *J* = 17.6, 4.6 Hz), 2.77 – 2.67 (m, 1H), 3.46 (d, 1H, *J* = 2.9 Hz), 4.72 (s, 1H), 4.79 (s, 1H)
	^{13}C NMR (75.5 MHz, $CDCl_3$) δ 15.2, 20.5, 28.6, 35.0, 41.7, 58.7, 61.3, 110.4, 146.3, 205.4
	IR (neat) 3063, 2978, 2935, 1709, 1646, 1440, 1378, 1120, 1051, 892, 815 cm^{-1}

Carvone-1,2-oxide was also formed as a pair of diastereomers as shown in the ^{13}C NMR spectrum. Integration of the methyl ^{13}C resonance of the isopropenyl group at 20.5/19.6 ppm revealed the diastereomers were formed in a ratio of 16:1, which is close to the literature reported value of 19:1 (figure 4) *(4).*

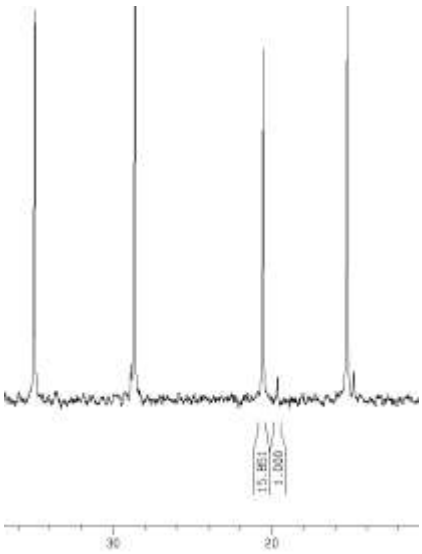


Figure 4. Partial ^{13}C NMR of compound **3** showing the diastereomeric ratio of the epoxides formed.

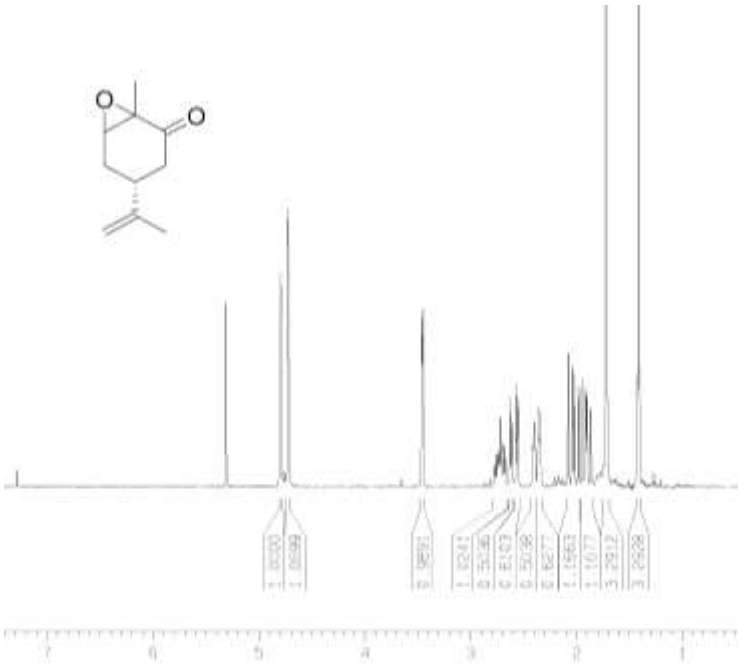


Figure 5. ^{1}H NMR of compound **3**.

The multiplet signal of the β-proton of the enone in carvone at 6.75 – 6.78 ppm was shifted to 3.46 ppm, indicating that the epoxidation occurred preferentially at the enone moiety (figure 5). The two signals at 4.76 and 4.81 ppm, which corresponded to the vinyl protons of the isopropenyl group, remained unaltered. The formation of the epoxy ring caused the cyclohexenone of carvone fix into a rigid bicyclic system. It prevented the six-member ring from converting from one chair conformation into the other. Therefore the coupling constant of the vicinal axial-axial protons became as large as 17.6 Hz. The two ^{13}C signals of the enone group of carvone at 135.3 and 144.6 ppm were shifted to the epoxide ring resonances at 58.7 and 61.3 ppm, respectively.

The partial IR spectrum shown in figure 3(c) also suggested the epoxidation occurred preferentially at the enone moiety. The C=O absorption band was blue-shifted to 1709 cm^{-1} and the isopropenyl C=C absorption remained unaltered. It suggested that the conjugation of enone was lost but the isopropenyl C=C bond was retained, implying that the epoxidation occurred selectively to the α,β-unsaturated ketone.

The product was also characterized by mass spectrometry. The spectrum showed a molecular ion peak at m/z equaled to 167, which is corresponding to the $[M+1]^+$ peak of compound **3** ($C_{10}H_{14}O_2$, MW = 166.10).

Determination of the selectivity of the epoxidation reactions

The selectivity of the epoxidation reactions can be conveniently determined qualitatively by thin-layer chromatography. The R_f values of carvone-7,8-oxide (**2**) and carvone-1,2-oxide (**3**) are 0.26 and 0.46, respectively (in hexane/ethyl acetate 10:1). Both compounds can be clearly visualized on the TLC plate with phosphomolybdic acid. Both reactions gave their respective desired products as a single spot on the TLC and none of the other epoxy products were observed. For undergraduate teaching purposes, this simple and qualitative method is already sufficient to demonstrate these two reactions are highly selective.

Instructors may require students to evaluate the reaction selectivity more precisely by ^{1}H NMR, as the relative amounts of the compounds present in a mixture can be determined from the NMR integration. The NMR spectra should be obtained from the crude product mixture prior to any purification procedure. This is to ensure nothing is removed unexpectedly.

Consistent with the TLC results, the ^{1}H NMR spectra of the crude product mixtures showed that both of the epoxidation reactions were specific. The ^{1}H NMR spectrum obtained for the product mixture from the MCPBA epoxidation showed the isopropenyl group was reacted completely as the two alkene protons signals originally located at 4.76 and 4.81 ppm disappeared. More importantly, the signal at 3.46 ppm which was assigned to the epoxide proton of the α,β-epoxyketone of **3** *(ref. ^{1}H NMR spectrum of compound **3**)* could not be observed in this spectrum. It implied that no epoxidation occurred at the enone moiety. The MCPBA epoxidation of carvone was therefore shown to be regiospecific.

Determining the selectivity of the alkaline H_2O_2 epoxidation, on the other hand, was slightly more difficult. The epoxide ^{1}H signals of the carvone-7,8-oxide (2.58 – 2.72 ppm) could not be unambiguously distinguished from the epoxycyclohexanone signals of carvone-1,2-oxide (1.87 – 2.78 ppm). Therefore, these resonances did not help much to ascertain the absence of **2** or diepoxycarvone from the product mixture obtained from H_2O_2 epoxidation. Fortunately, the epoxides could be distinguished by the ^{1}H signals of the methyl groups. For carvone, both of the methyl groups of the enone and the isopropenyl moieties appear at 1.70 – 1.80 ppm. The methyl groups that attached to the epoxy rings of carvone-1,2-oxide (**3**) and carvone-7,8-oxide (**2**) appear at 1.41 and

1.32/1.33 ppm respectively. The methyl signals at 1.32/1.33 ppm were not observed in the spectrum obtained for the alkaline H_2O_2 reaction mixture, suggesting that no epoxidation occurred at the isopropenyl group. Therefore, the alkaline H_2O_2 epoxidation was found to be regiospecific to the enone moiety.

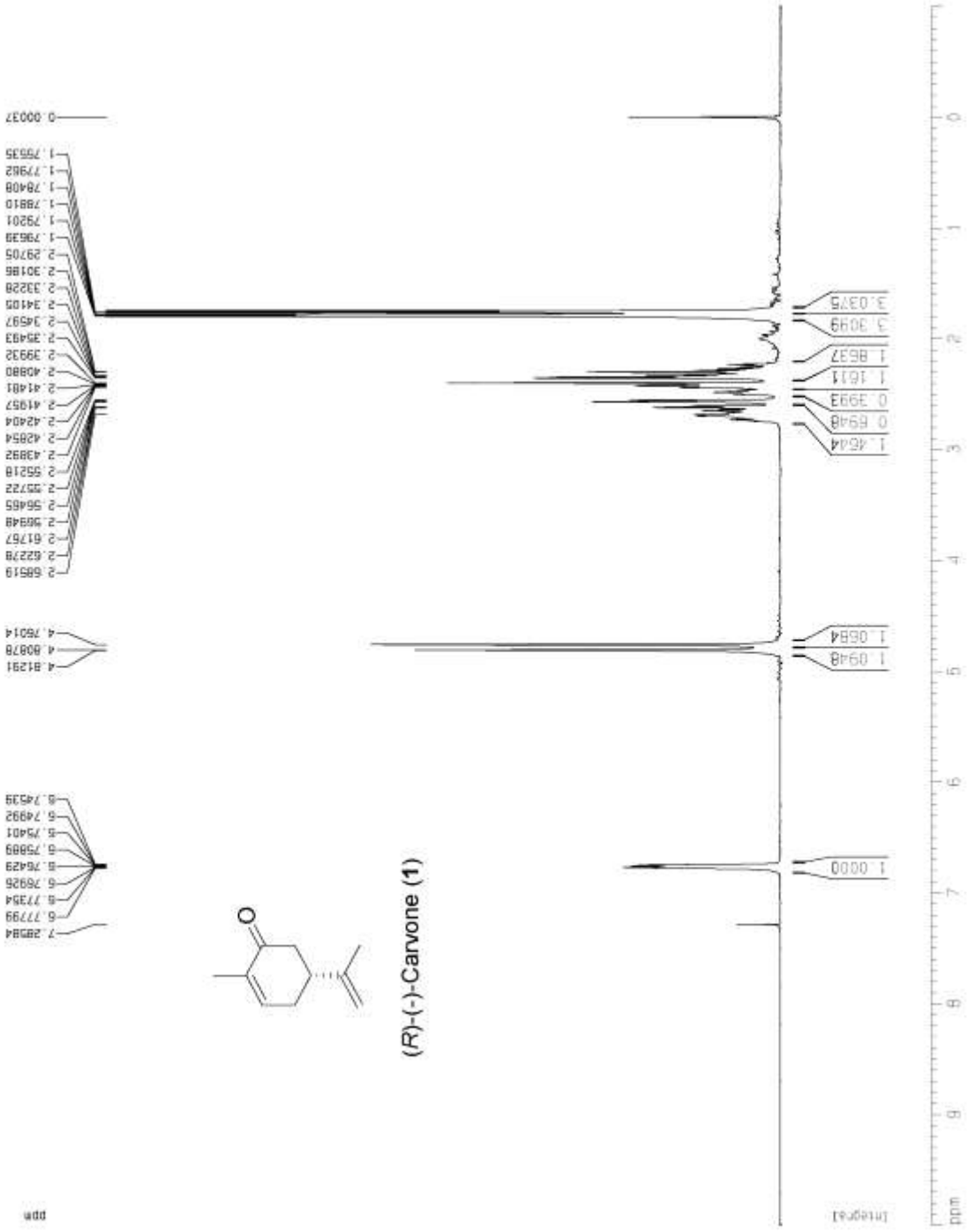
Literature Cited

1. Baldwin, J. E.; Broline, B. M. *J. Am. Chem. Soc*. **1982**, *104*, 2857-2865.
2. Smitt, O.; Högberg, H.-E. *Tetrahedron* **2002**, *58*, 7691-7700.
3. Muralidharan, K. R.; de Lera, A. R.; Isaeff, S. D.; Norman, A. W.; Okamura, W. H. *J Org. Chem.* **1993**, *58*, 1895-1899.
4. McChesney, J. D.; Thompson, T. N. *J. Org. Chem.* **1985**, *50*, 3473-3481.

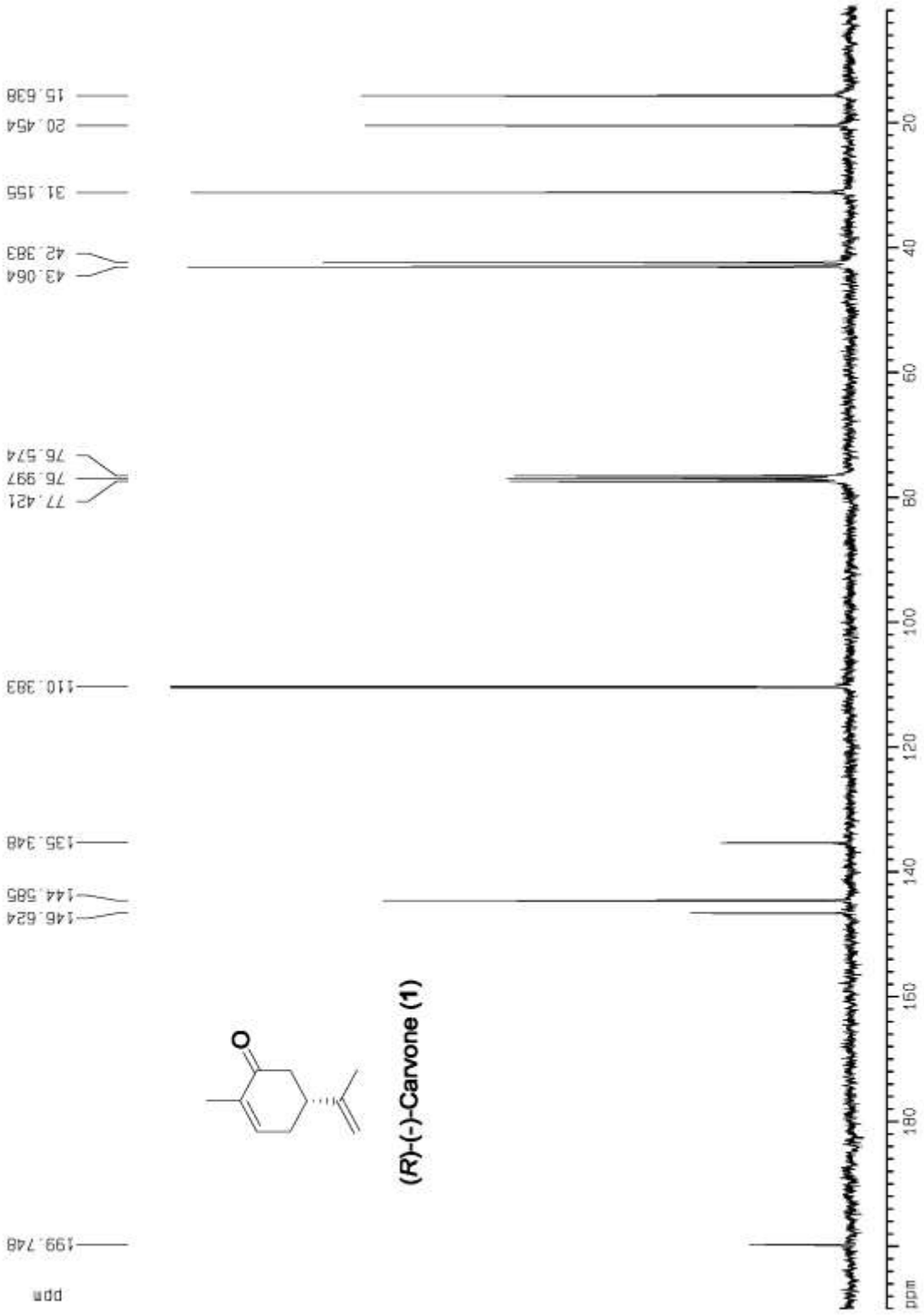
List of Required Chemicals:

Name	CAS #	Aldrich #	Amt. Used (per student)
Epoxidation of *l*-Carvone by m-Chloroperoxybenzoic Acid			
(*R*)-(-)-Carvone	6485-40-1	12,493-1	0.5 g
m-Chloroperoxybenzoic acid (75%)	937-14-4	27,303-1	0.85 g
Dichloromethane	75-09-2	D6,510-0	25 mL
Sodium sulfite	7757-837	20,784-5	0.1 g
Sodium carbonate	497-19-8	22,353-0	5 g
Sodium chloride	7647-14-5	31,016-6	
Magnesium sulfate	7487-88-9	20,809-4	2 g
Epoxidation of *l*-Carvone by Alkaline H_2O_2			
(*R*)-(-)-Carvone	6485-40-1	12,493-1	0.72 g
Methanol	67-56-1	17,995-7	8 mL
35% Hydrogen peroxide	7722-84-1	34,988-7	1.5 mL
Sodium hydroxide	1310-73-2	36,717-6	0.24 g
Dichloromethane	75-09-2	D6,510-0	10 mL
Sodium chloride	7647-14-5	31,016-6	
Magnesium sulfate	7487-88-9	20,809-4	2 g
Thin-layer Chromatographic Analysis			
Merck TLC plates (silica gel 60) w/ fluorescent indicator		Z29,302-4	
Hexane	110-54-3	20,875-2	20 mL
Ethyl acetate	141-78-6	32,030-7	2 mL
Phosphomolybdic acid	22,185-6	51429-74-4	
95% Ethanol	64-17-5	18,738-0	
NMR Spectroscopy			
Chloroform-*d* (with 0.03% v/v TMS)	865-49-6	22,578-9	1.5 mL

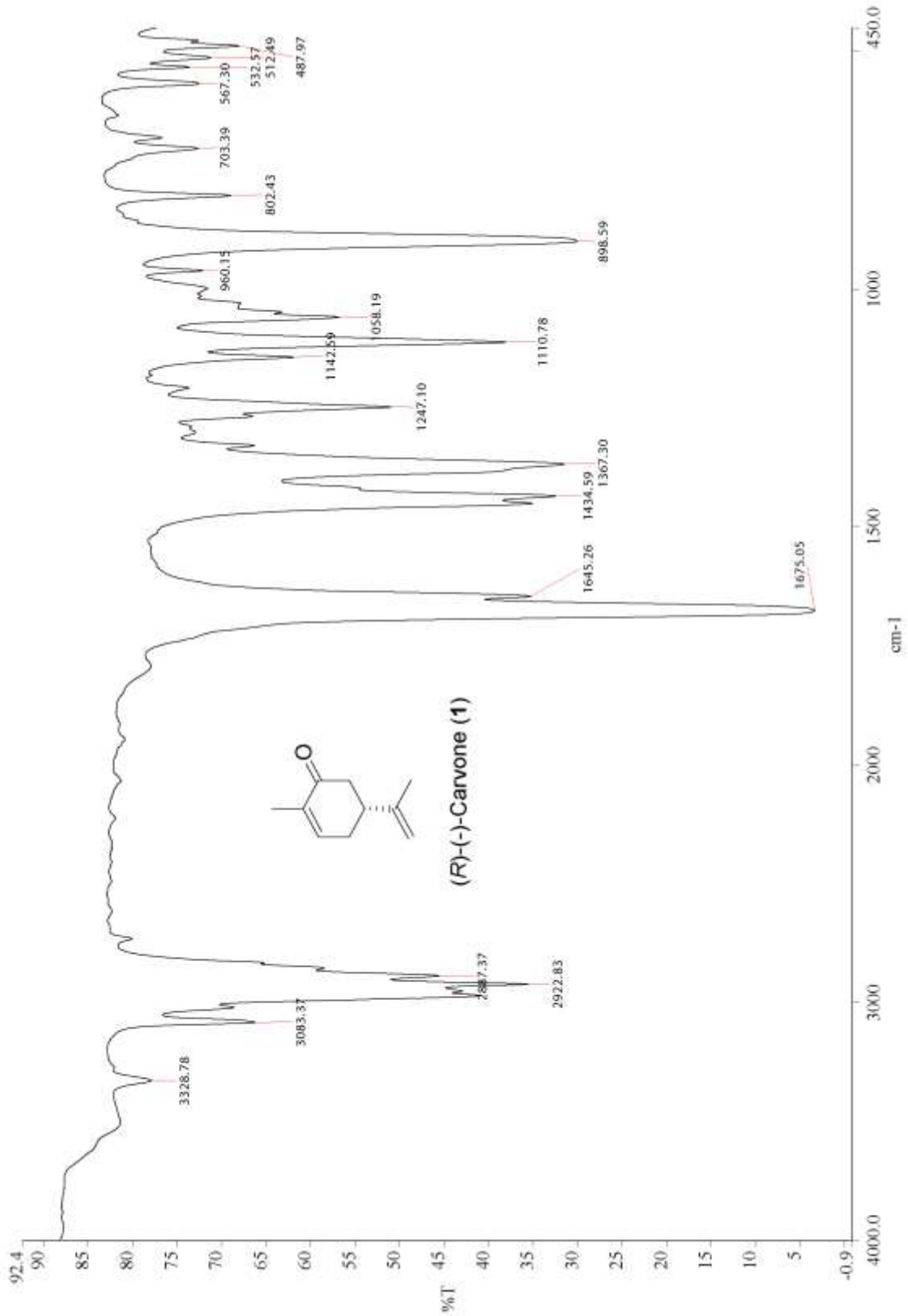
List of Spectra



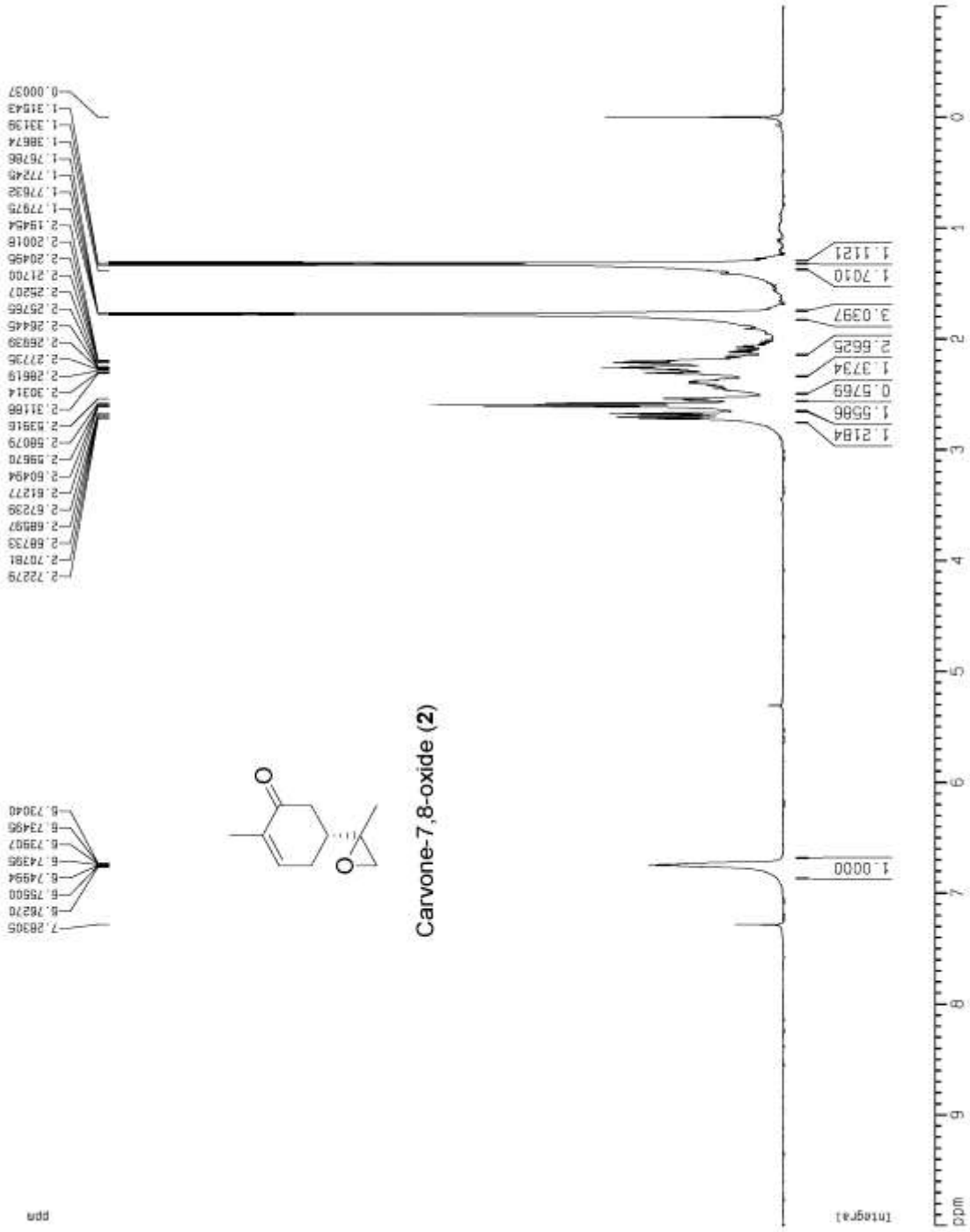
Spectrum 1: ^{1}H NMR Spectrum of (*R*)-(-)-Carvone (**1**)



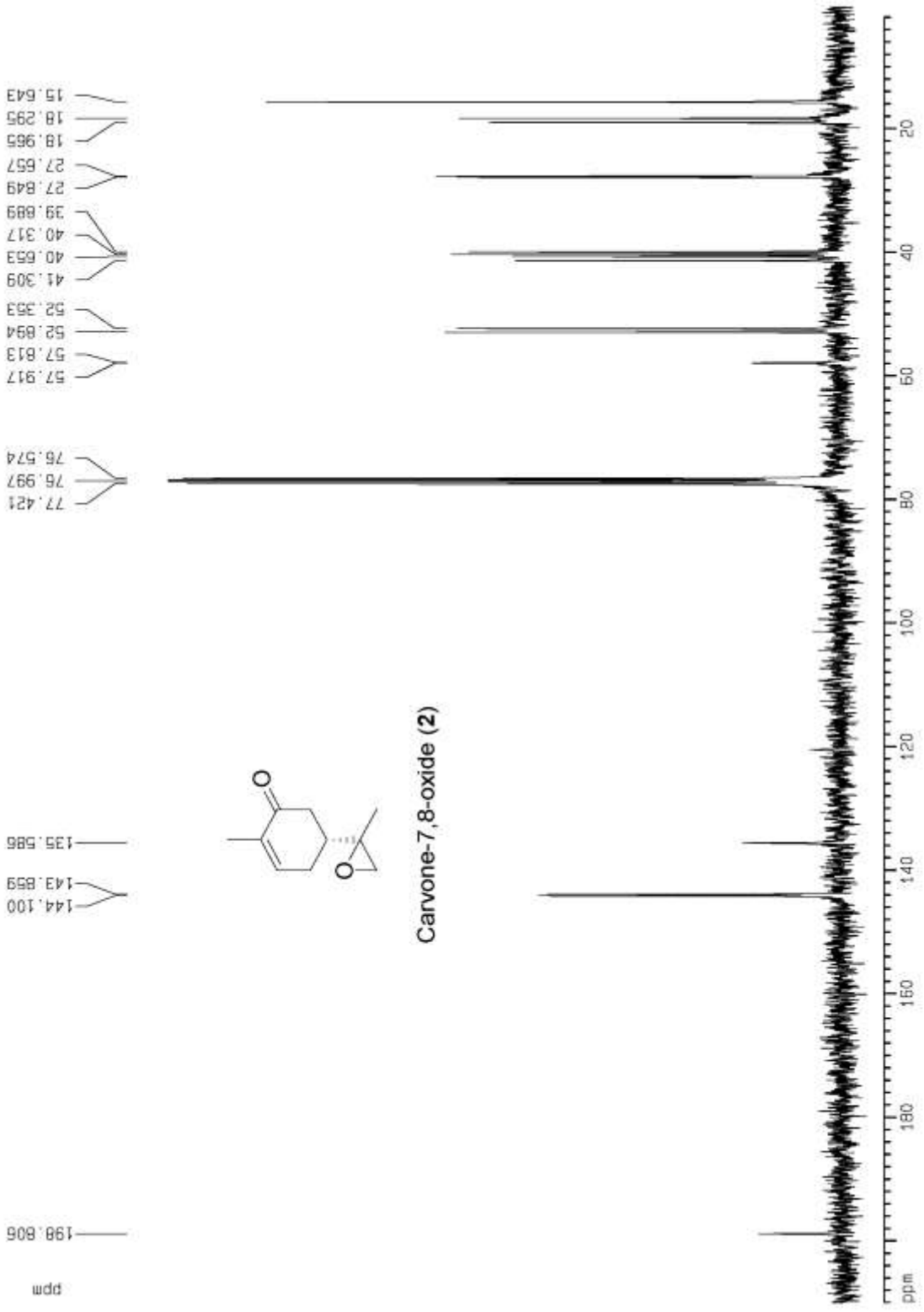
Spectrum 2: ^{13}C NMR Spectrum of (*R*)-(-)-Carvone (**1**)



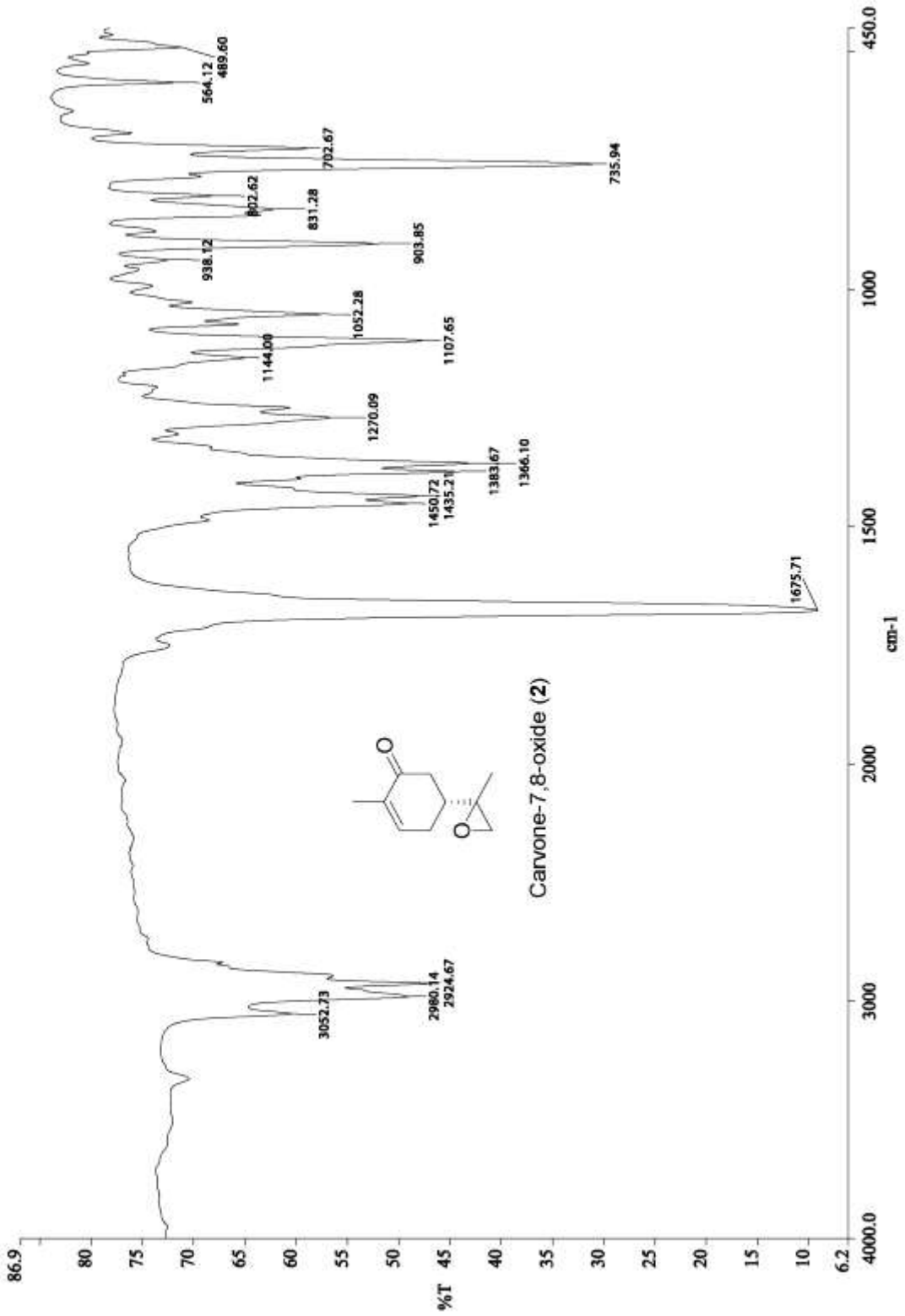
Spectrum 3: IR Spectrum of (*R*)-(-)-Carvone (**1**)



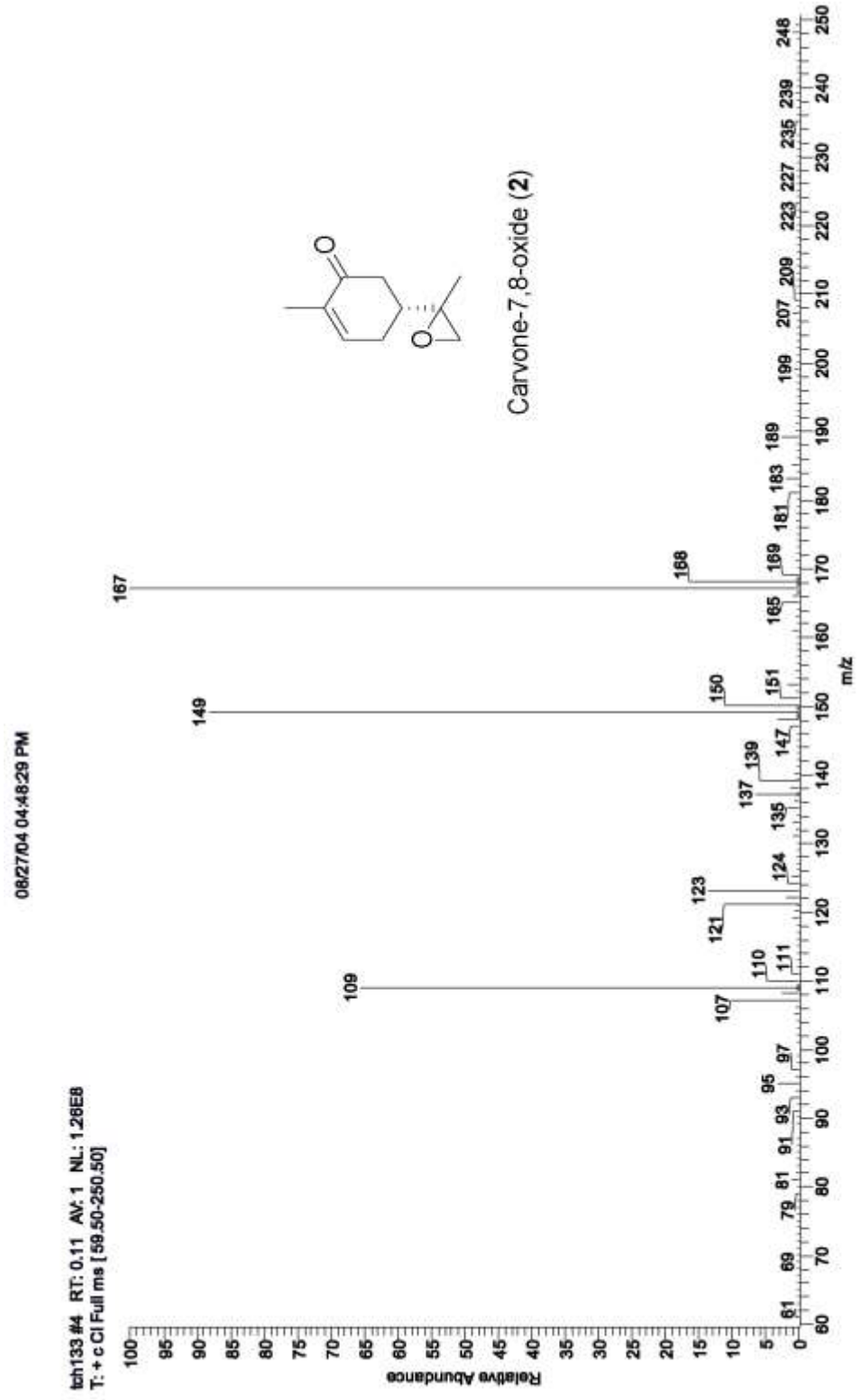
Spectrum 4: ^{1}H NMR Spectrum of the product mixture from MCPBA epoxidation – Carvone-7,8-oxide (**2**)



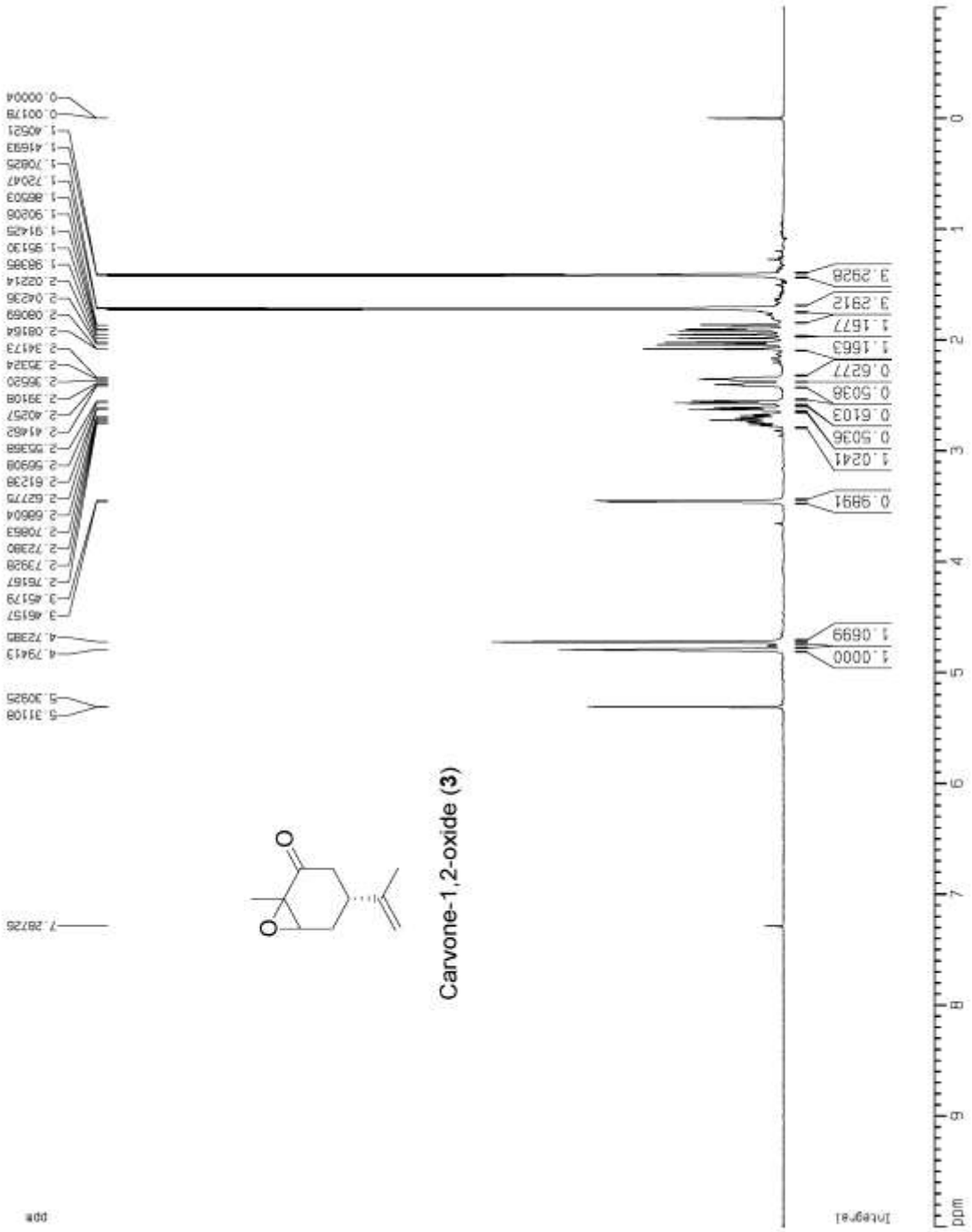
Spectrum 5: ^{13}C NMR Spectrum of carvone-7,8-oxide (**2**)



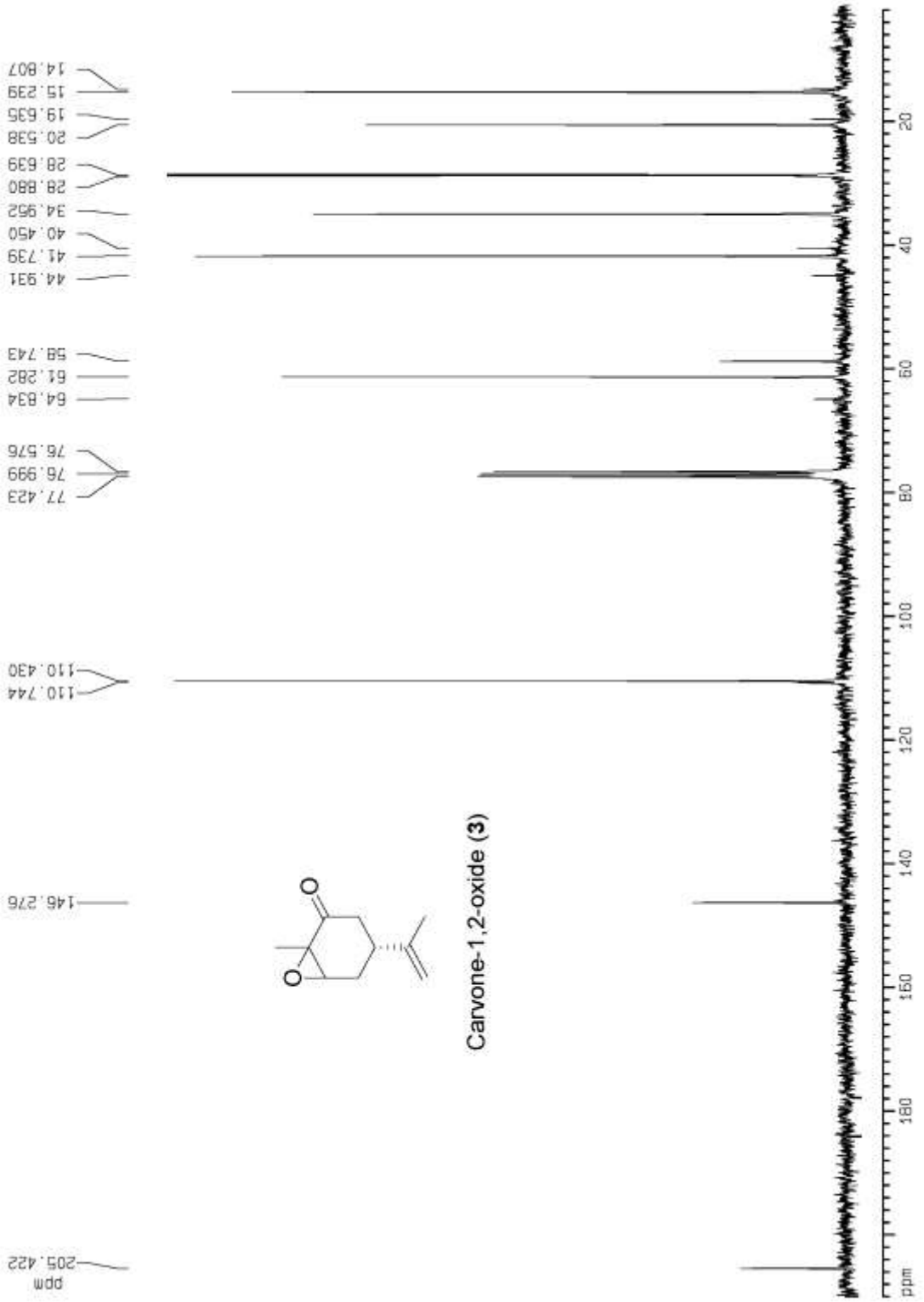
Spectrum 6: IR Spectrum of carvone-7,8-oxide (**2**)



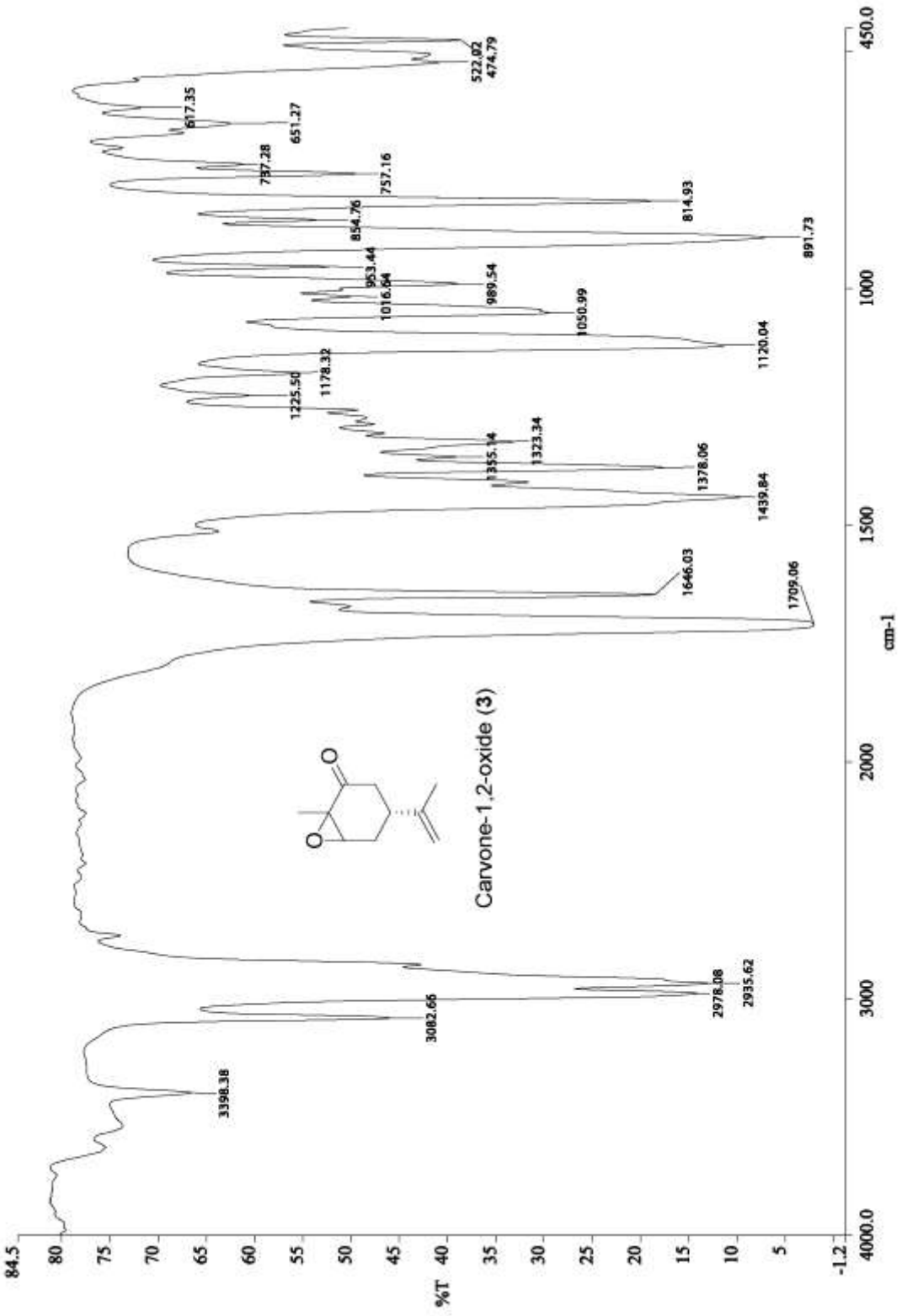
Spectrum 7: Mass Spectrum of carvone-7,8-oxide (**2**)



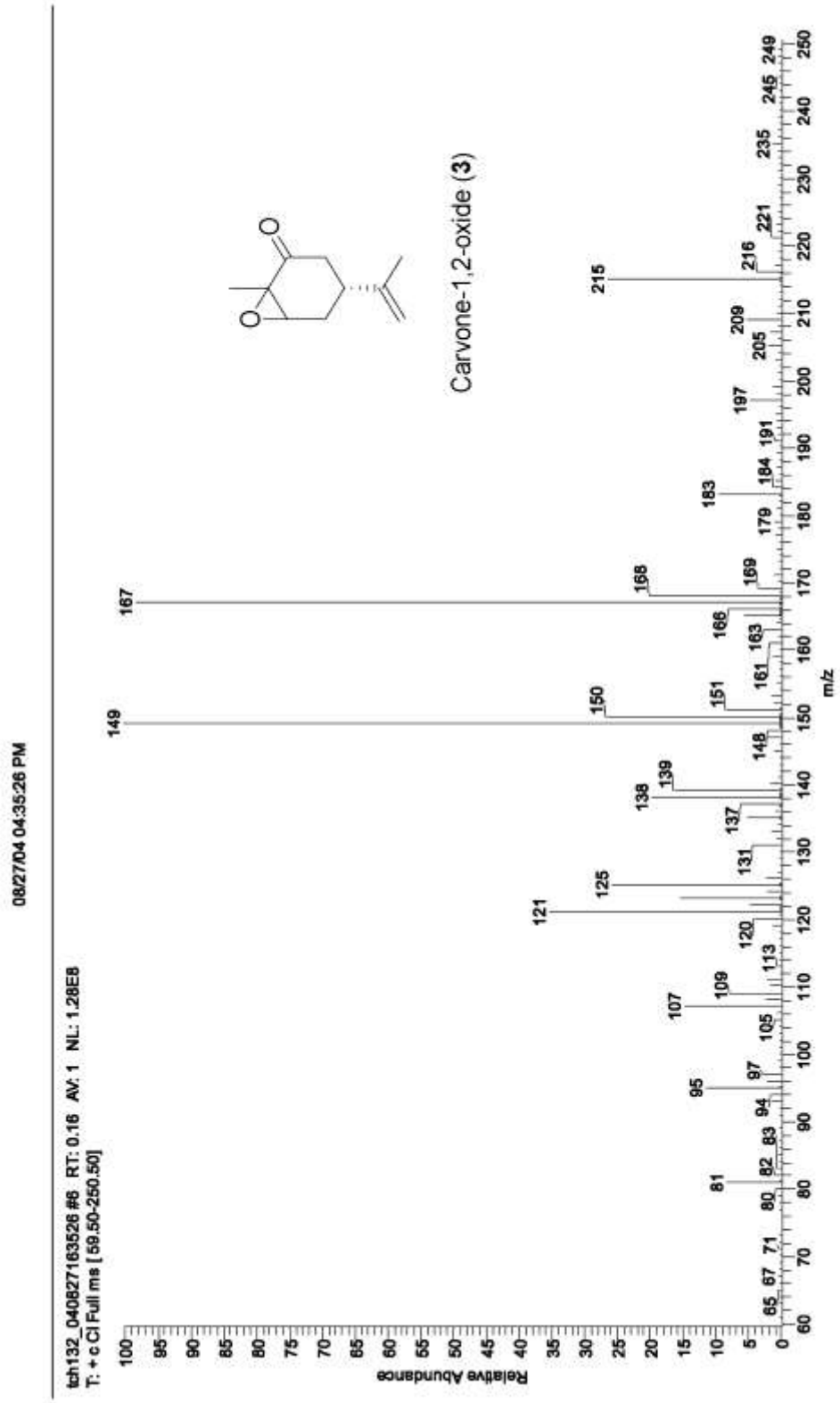
Spectrum 8: ^{1}H NMR Spectrum of the product mixture from alkaline H_2O_2 epoxidation – Carvone-1,2-oxide (**3**)



Spectrum 9: ^{13}C NMR Spectrum of carvone-1,2-oxide (**3**)



Spectrum 10: IR Spectrum of carvone-1,2-oxide (**3**)



Spectrum 11: Mass Spectrum of carvone-1,2-oxide (**3**)