Experiment 9

Isolation of the Essential Oils from Common Spices and Spectroscopic Analysis of Their Major Constituents

Student Handout

Purposes

1. To extract the essential oils from some common spices.
2. To determine the essential oil contents in the spices.
3. To analyse the major constituent of the essential oil by infra-red and mass spectroscopic methods.

Background

The use of plants and spices for food flavouring has been known for thousands of years and has become a very important culture among the human races. Cinnamon, clove, garlic, almond, peppermint and vanilla are some common examples that have a very long history. The leaves, seeds, barks or other parts of many plants are known to have distinctively pleasant odours or flavours due to the presence of essential oils. Essential oils are used in virtually all kinds of food production such as bakery foods, pickles, beverages, etc.

Essential oils are some highly volatile organic substances that can be isolated from odoriferous plants by various physical processes. The oils are usually concentrated in the seeds or flowers but may exist in other parts of the plants as well. Such oils were called essential because they were thought to represent the very essence of odour and flavour. Essential oils can be obtained from plants by a number of processes such as mechanical pressing and grinding, maceration, solvent extraction, distillation and concentration. In many cases a combination of processes are required for an efficient and effective isolation.

Chemists are interested in identifying the chemical structures of the isolated essential oils. Having known the chemical structure of the oils, chemists can synthesise the essential oils in the laboratories, so that the sources of the essential oils are no longer confined by the availability of the plants.

Modern instrumental techniques play an indispensable role in the structural determination of organic compounds. For example, infra-red spectroscopy is an invaluable tool for determining the presence (or absence) of certain functional groups such as carbon-carbon multiple bonds, aromatic rings, carbonyl groups or hydroxyl

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groups in a molecule. Mass spectrometry, on the other hand, not only measures the relative molecular mass of an organic compound but also reveals its structural components through fragment analysis.

Tasks

1. Extract the essential oil from cinnamon or clove by heating the spice in refluxing ethyl ethanoate.
2. Remove the solvent (ethyl ethanoate) from the essential oil solution by simple distillation.
3. Determine the mass content of the essential oil in the spice.
4. Analyse the chemical structure of the major constituent present in the essential oil from the given infra-red and mass spectra.

Safety

The experiment should be performed in a well-ventilated laboratory or fumehood. Handle all chemicals with great care. Avoid direct contact of chemicals with skin. Dispose of chemical waste, broken glassware and excess materials according to your teacher's instruction.

EYE PROTECTION
MUST BE WORN

Ethyl ethanoate is a highly flammable volatile organic solvent which should be handled with care.

Pay special attention when performing reflux and simple distillation. Never heat a closed system or explosion may occur. Do not heat the flasks containing flammable liquids directly with a naked flame. Heat with a hot oil or water bath instead. Make sure the rubber tubings of the water condenser are connected properly. Never leave a distillation unattended.

Safety information on the chemicals used in this experiment can be found in the Material Safety Data Sheet (MSDS). Consult your teacher for details.

Materials and Apparatus Available

Ethyl ethanoate (50 cm^3)  FLAMMABLE  HARMFUL/ IRRITANT

Spices (cinnamon or clove)

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Anhydrous $MgSO_4$ or anhydrous $CaCl_2$ (1 - 2 g) 

Mortar and pestle	Weighing balance
Water condenser	100-cm^3 Round-bottomed flask
Bunsen burner/electric hot plate	Oil bath
Filter paper	Anti-bumping granules
Apparatus for simple distillation	Filtering funnel

Experimental Procedure

Photos of the experiment are available at http://www.chem.cuhk.edu.hk/ssc.htm.

Part A: Isolation of the essential oil from the spice

1. Weigh 5.0 g of spice (cinnamon or clove) and grind it into small pieces with mortar and pestle. Transfer the spice into a 100-cm^3 round-bottomed flask. Add a few pieces of anti-bumping granules to the flask.
2. Add 35 cm^3 of ethyl ethanoate to the round-bottomed flask.
3. Attach a water-cooled condenser to the flask. Heat the mixture under reflux for 15-20 minutes.
4. Cool the mixture to room temperature and carefully decant the solution into a clean Erlenmeyer (conical) flask. Rinse the residue in the round-bottomed flask with a small amount of ethyl ethanoate and combine it with the solution in the conical flask.
5. Add a small amount of anhydrous $MgSO_4$ or anhydrous $CaCl_2$ to the solution (~1 g). Swirl the contents for about 30 seconds and filter the mixture.
6. Place a few pieces of anti-bumping granules into a clean 100-cm^3 round-bottomed flask and weigh the flask. Transfer the filtrate into the flask.
7. Assemble the apparatus for simple distillation. Remove the solvent (ethyl ethanoate) from the mixture by simple distillation.
8. When the solvent is distilled out completely, disassemble the apparatus and weigh the round-bottomed flask with the oily residue. The oily residue is the essential oil.
9. Determine the weight of the essential oil isolated. Calculate the essential oil content of the spice.

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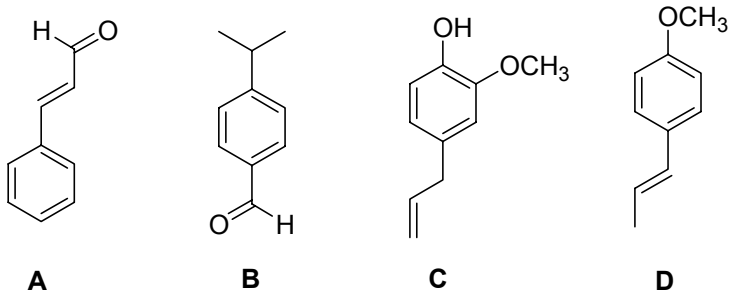
Part B: Identify the major constituent of the essential oil by infra-red and mass spectroscopy

1. The infra-red and the mass spectra for the essential oil that you have isolated are given below. Which of the following compounds is most likely to be the major constituent present in your essential oil?



A **B** **C** **D**

[Hint: in the IR spectrum, try to find the bands corresponding to the following functionalities: C=O (ketone or aldehyde), C-H (aldehyde), O-H (phenol), C-O (ether), benzene ring, C=C (alkene) and the substitution pattern of the benzene ring.]

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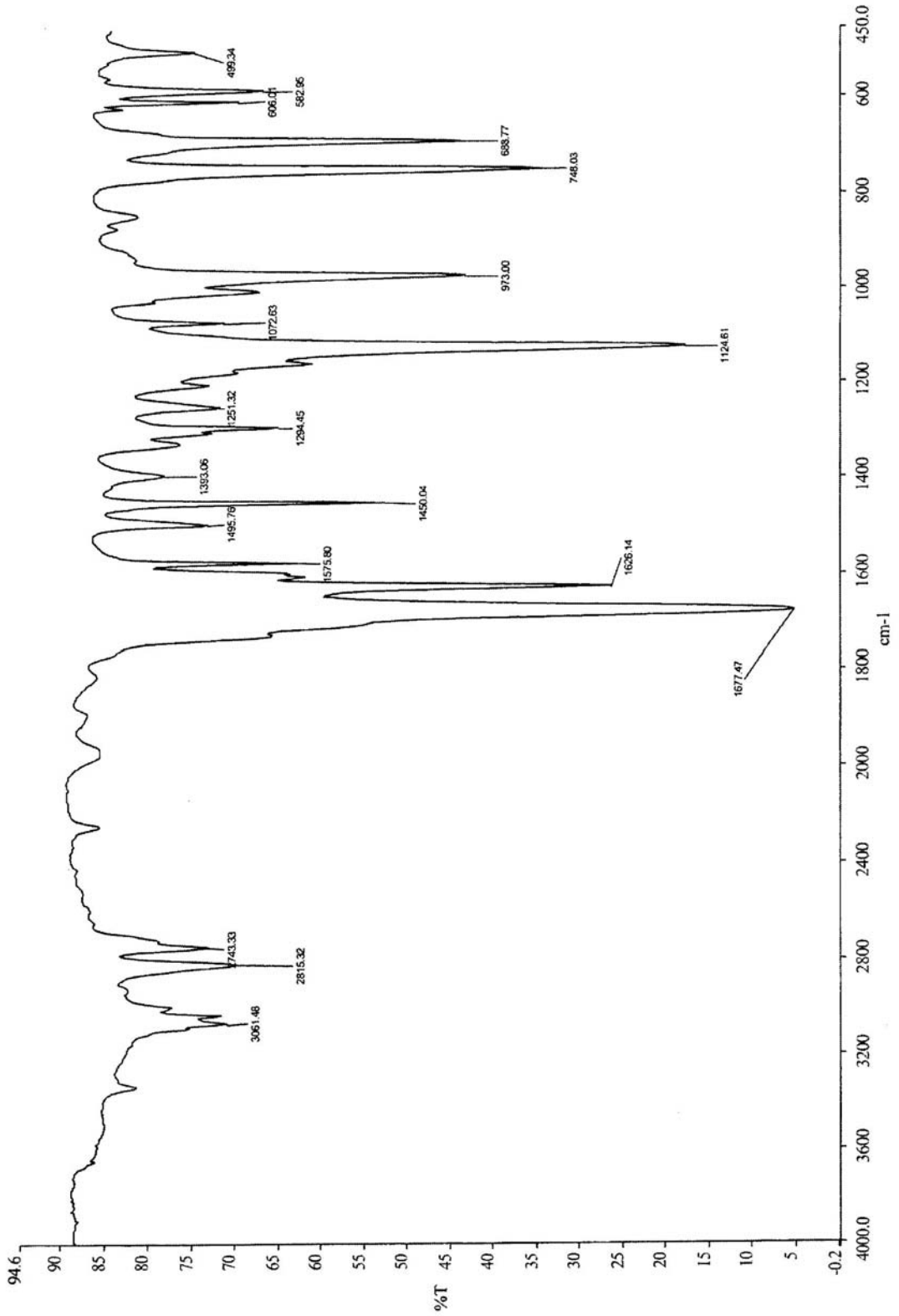
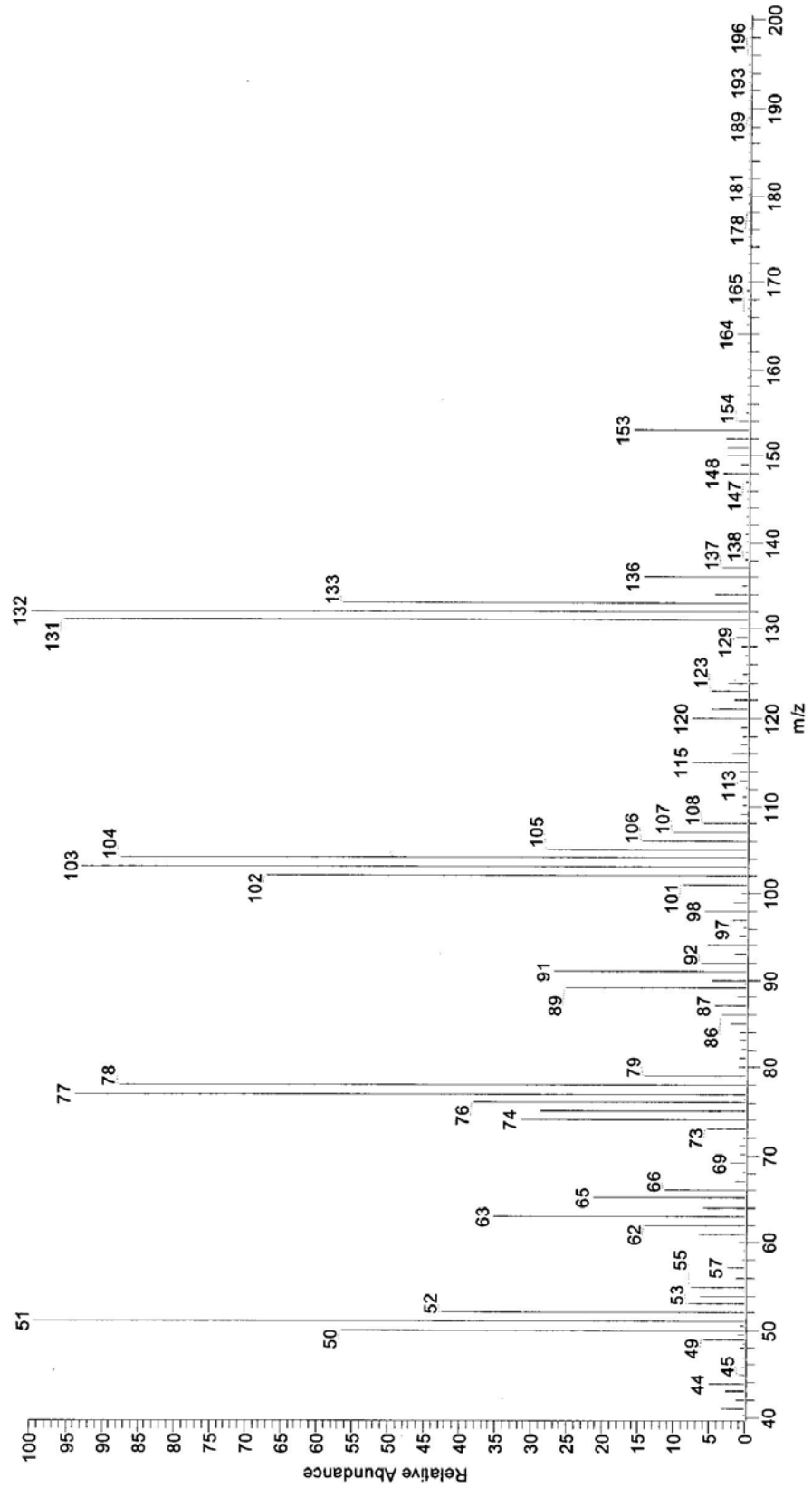
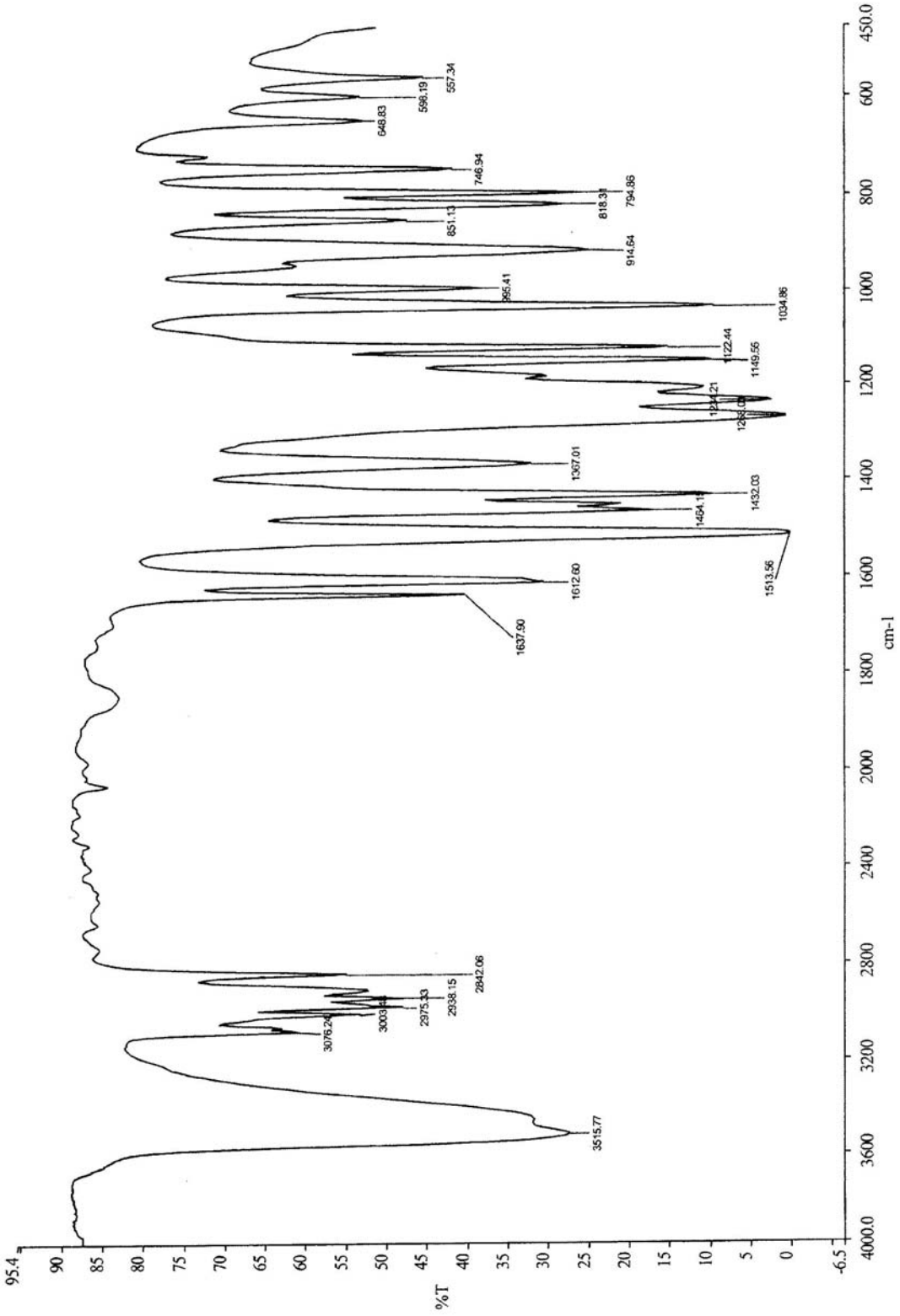
Spectrum 1: IR spectrum of the essential oil isolated from cinnamon



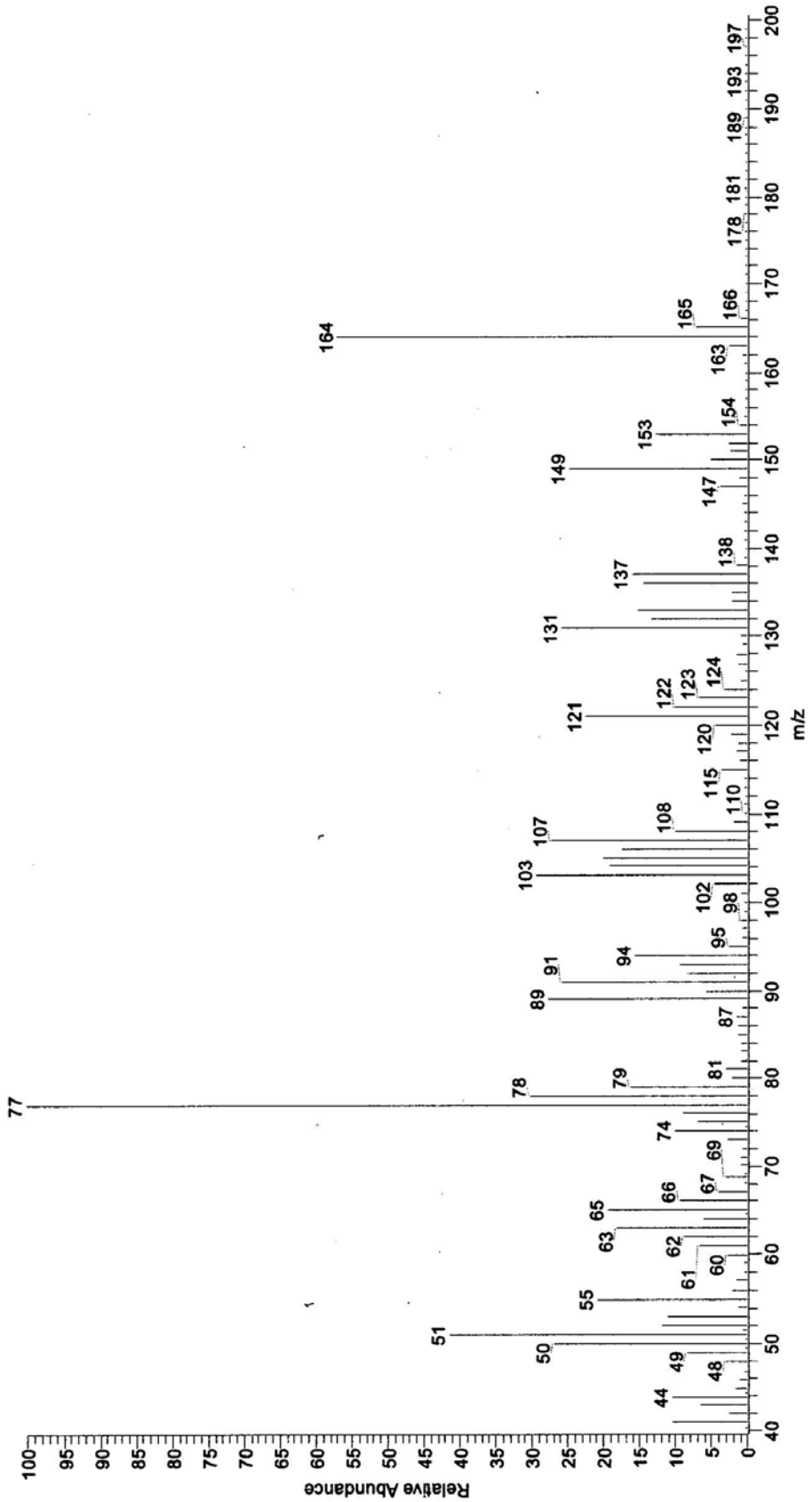
Spectrum 2: Mass spectrum of the essential oil isolated from cinnamon



Spectrum 3: IR Spectrum of the essential oil isolated from cloves



Spectrum 4: Mass spectrum of the essential oil isolated from cloves



Questions for Further Thought

1. Why should the cooling water always enter the water condenser from the lower opening?
2. Explain briefly why heating should never be applied to a closed system.
3. What is the purpose of adding a small amount of anhydrous $MgSO_4$ or anhydrous $CaCl_2$ to the organic solution?
4. Why is it necessary to dry the apparatus thoroughly for the simple distillation?
5. What are the possible sources of error in determining the essential oil content?
6. You are given an authentic sample of the essential oil. Propose a simple experiment to confirm that the isolated essential oil is identical to the given authentic sample.
7. Find out the plant origin for the following essential oils:
   (a) limonene
   (b) menthol
   (c) citral
   (d) myrcene
   (e) eugenol
   (f) anethole

References

1. D. L. Pavia, G. M. Lampman, G. S. Kriz and R. G. Engel, *Introduction to Organic Laboratory Techniques - Small-Scale Approach*, 1st Ed.; Harcourt Brace, Florida, 1998, pp. 165 - 172.
2. R. M. Silverstein and F. X. Webster, *Spectrometric Identification of Organic Compounds*, 6th Ed.; John Wiley & Sons, New York, 1998, pp. 71 - 143.