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Extraction of Essential Oils from Spices using Steam Distillation

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Contents

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Objectives

The principal aims of these experiments are to provide experience in the extraction of essential oils from spices using the steam distillation technique. Students will isolate and purify the key chemical compounds that contribute to the aroma and flavour of the spices. The compounds will then be characterised by NMR spectroscopy using the benchtop Spinsolve NMR spectrometer.

Introduction

Extraction of the aroma and flavour components from dried plant materials is one of the oldest chemical operations developed by humankind. The preparation of a cup of tea or coffee is an everyday example of extracting the flavour and aroma components, including the stimulant caffeine, with hot water. In the following experiments, steam distillation is used to extract the essential oils from two common spices: star anise and cinnamon bark (Figure 1). The main essential oils responsible for the aroma and flavour of these spices are *trans*-anethole [1-methoxy-4-(1-propenyl)benzene] from star anise and *trans*-cinnamaldehyde [(2*E*)-3-phenylprop-2-enal] from cinnamon bark.

Steam distillation is a technique used to distill immiscible liquids, for which steam provides one of the immiscible phases.[1] The two substances mix in the gas phase and co-distill, but when cooled the desired component separates from water as it is immiscible. Steam distillation is commonly used to extract perfume and flavour oils from natural sources.

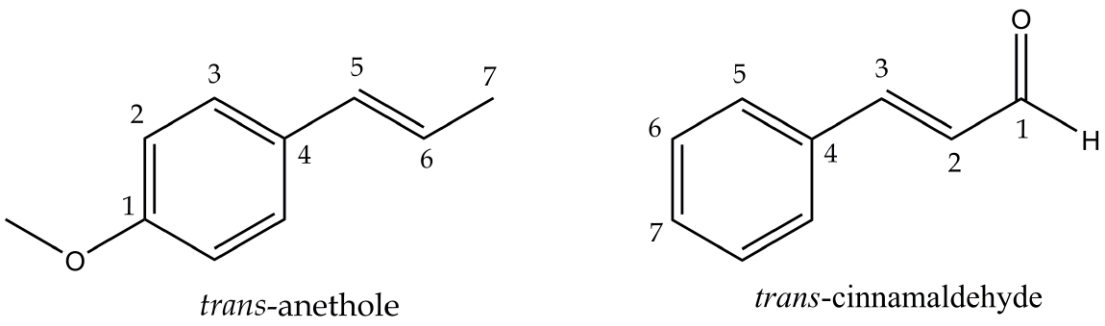

Figure 1. Essential oils to be extracted from spices: *trans*-anethole from star aniseand trans-cinnamaldehyde from cinnamon.

Extraction of *trans*-anethole from star anise

A macroscale direct method for steam distillation is used to extract the essential oil, which is composed primarily of *trans*-anethole, from the star anise spice. In this method, the steam is generated *in situ* by heating the ground dry spice material and water in the distillation flask (Figure 2).

Safety

Diethyl ether is highly flammable, handle with caution. Magnesium sulfate is hazardous with respect to ingestion, inhalation and skin contact. Deuterochloroform ($CDCl_3$) is toxic, handle with caution and do not ingest or inhale.

Procedure

Grind two whole star anise seed pods (approximately 3.5 g) in a mortar and pestle (Figure 3), and place the ground material into a 100 mL 2-neck round bottom flask. Add water (40 mL) and begin the distillation. After collecting about 20 mL of cloudy distillate, add more water (20 mL) into the distillation flask from the dropping funnel. Collect a further 20 mL of distillate then stop the distillation. Combine the distillates in a separatory funnel and extract with diethyl ether (2 × 20 mL). Dry the ethereal layer with anhydrous magnesium sulfate and filter the solution. Remove the ether using a rotary evaporator and collect the colourless star anise essential oil. Record your yield.

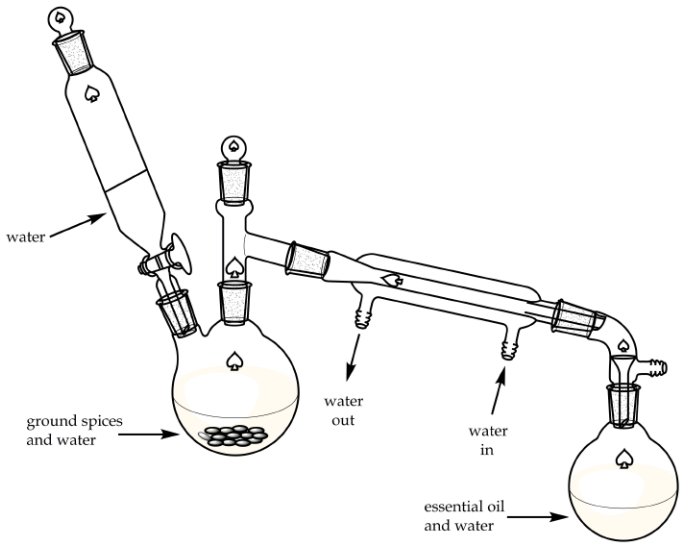

Figure 2. Direct method steam distillation apparatus.

Figure 3. Ground whole star anise seed pods.

Tasks

- Calculate the yield of *trans*-anethole from the dry mass of the seed pods.
- Record the ^1H NMR and COSY spectra of *trans*-anethole using the Spinsolve NMR spectrometer. Prepare the NMR sample using a drop of the oil in 0.6 mL of $CDCl_3$.
- Assign the ^1H NMR and COSY spectra to the structure of *trans*-anethole.

NMR Spectra

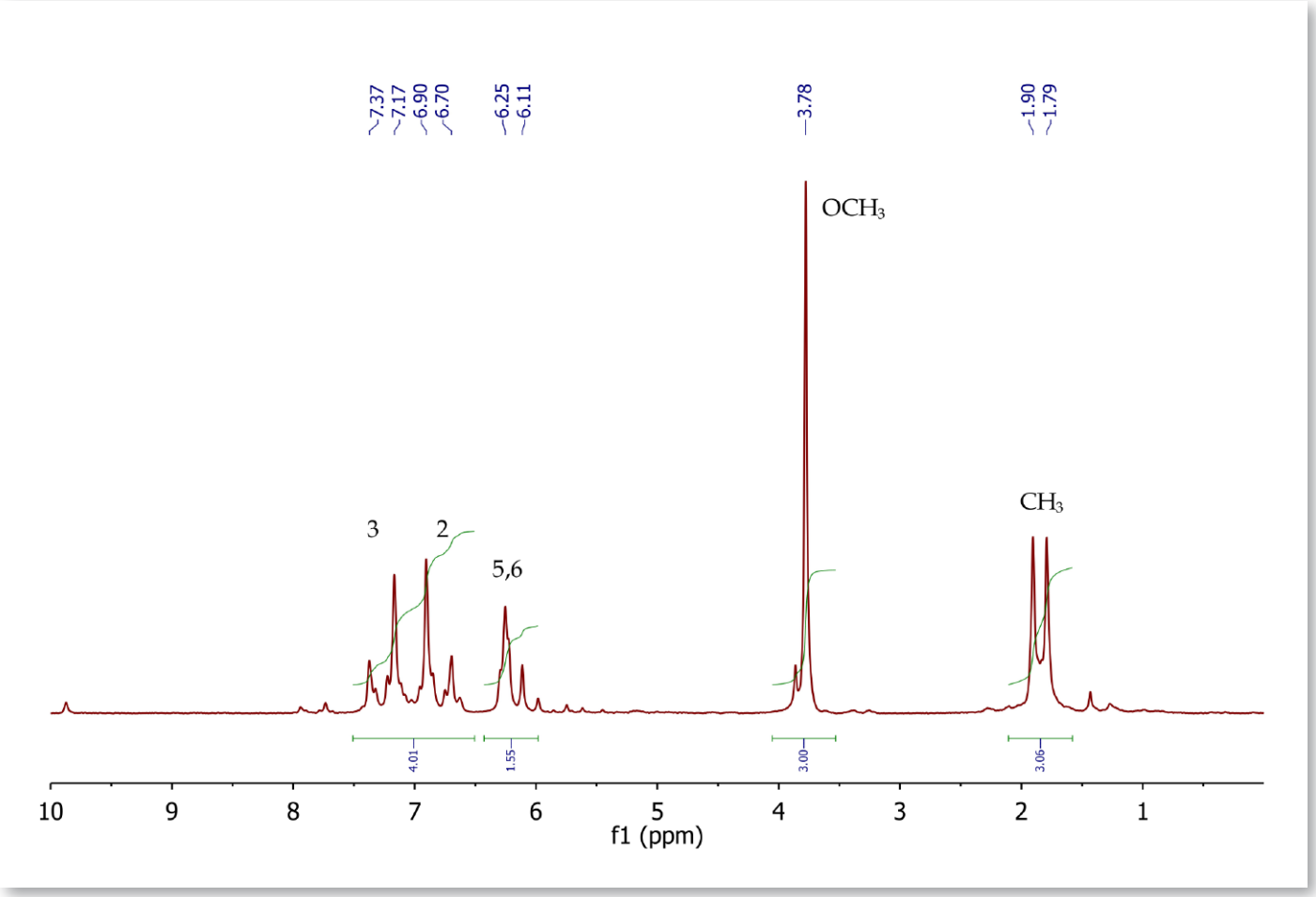

Figure 4. ^{1}H NMR spectrum of *trans*-anethole, $CDCl_3$.

The ^{1}H NMR spectrum of *trans*-anethole (Figure 4) shows a singlet (3H) at 3.78 ppm, corresponding to the methoxy (OCH_3) group. A doublet (3H) is observed at 1.85 ppm for the methyl group at position 7. The two CH protons at positions 5 and 6 appear between 6.11-6.25 ppm as a broad multiplet. The four aromatic protons at positions 2 and 3 resonate as a second-order AA'BB' system with two multiplets centred at 6.80 and 7.27 ppm.

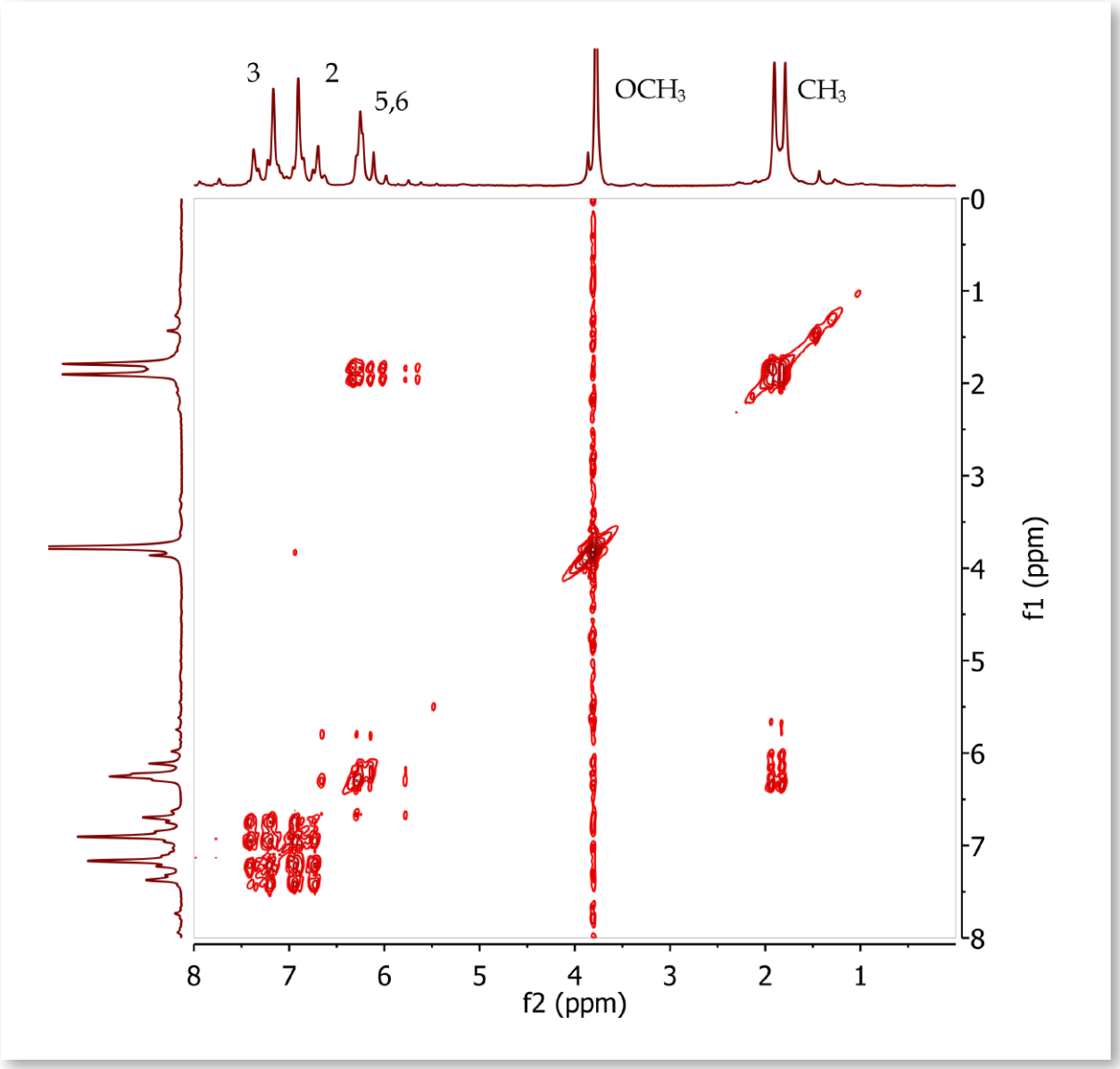

Figure 5. COSY spectrum of *trans*-anethole, $CDCl_3$.

The COSY spectrum of *trans*-anethole (Figure 5) clearly shows the correlations between the two alkene CH protons at positions 5 and 6 and the methyl group at position 7. COSY correlations are also observed between the four aromatic protons at positions 2 and 3.

Extraction of *trans*-cinnamaldehyde from cinnamon

A macroscale direct method for steam distillation is also used to extract the essential oil, which is composed primarily of *trans*-cinnamaldehyde, from the cinnamon spice. In this method, the steam is generated *in situ* by heating the ground dry spice material and water in the distillation flask (Figure 2).

Safety

Diethyl ether is highly flammable, handle with caution. Magnesium sulfate is hazardous with respect to ingestion, inhalation and skin contact. Deuterochloroform ($CDCl_3$) is toxic, handle with caution and do not ingest or inhale.

Procedure

Grind two whole sticks of cinnamon (approximately 5.5 g) in a mortar and pestle (Figure 6), and place the ground material into a 100 mL 2-neck round bottom flask. Add water (40 mL) and begin the distillation. After collecting about 20 mL of cloudy distillate, add more water (20 mL) into the distillation flask from the dropping funnel. Collect a further 20 mL of distillate then stop the distillation. Combine the distillates in a separatory funnel and extract with diethyl ether (2 × 20 mL). Dry the ether layer with anhydrous magnesium sulfate and filter the solution. Remove the ether using a rotary evaporator and collect the colourless cinnamon essential oil. Record your yield.

Figure 6. Ground sticks of cinnamon.

Tasks

- Calculate the yield of *trans*-cinnamaldehyde from the dry mass of cinnamon sticks.
- Record the ^1H NMR and COSY spectra of *trans*-cinnamaldehyde using the Spinsolve NMR spectrometer. Prepare the NMR sample using a drop of the oil in 0.6 mL of $CDCl_3$.
- Assign the ^1H NMR and COSY spectra to the structure of *trans*-cinnamaldehyde.

NMR Spectra

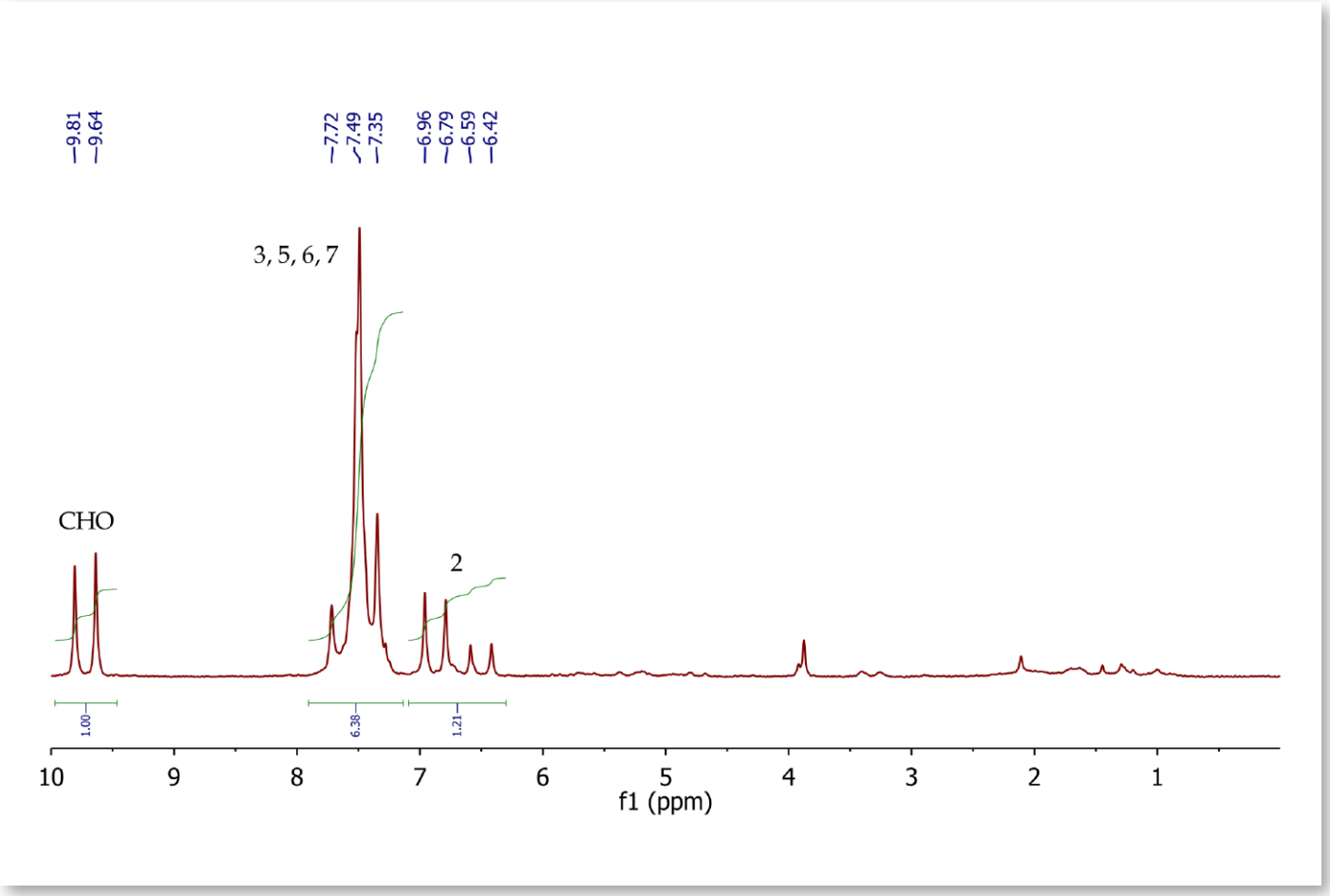

Figure 7. ^{1}H NMR spectrum of *trans*-cinnamaldehyde, $CDCl_3$.

The ^{1}H NMR spectrum of *trans*-cinnamaldehyde (Figure 7) shows a doublet (7.3 Hz) at 9.73 ppm for the aldehyde proton (CHO). The CH proton at position 2 is observed as a doublet of doublets (16.0 Hz, 7.3 Hz) at 6.69 ppm, as it is coupling to both the aldehyde proton (7.3 Hz) and the other alkene CH proton at position 3. The 16.0 Hz coupling constant between positions 2 and 3 is indicative of (*E*)- geometry about the double bond. The five aromatic protons at positions 5, 6 and 7 appear as a broad multiplet centred at approximately 7.49 ppm. The CH proton at position 3 should resonate as a doublet as it is coupling to the CH proton at position 3, but its signal is overlapping with the signal for the aromatic protons.

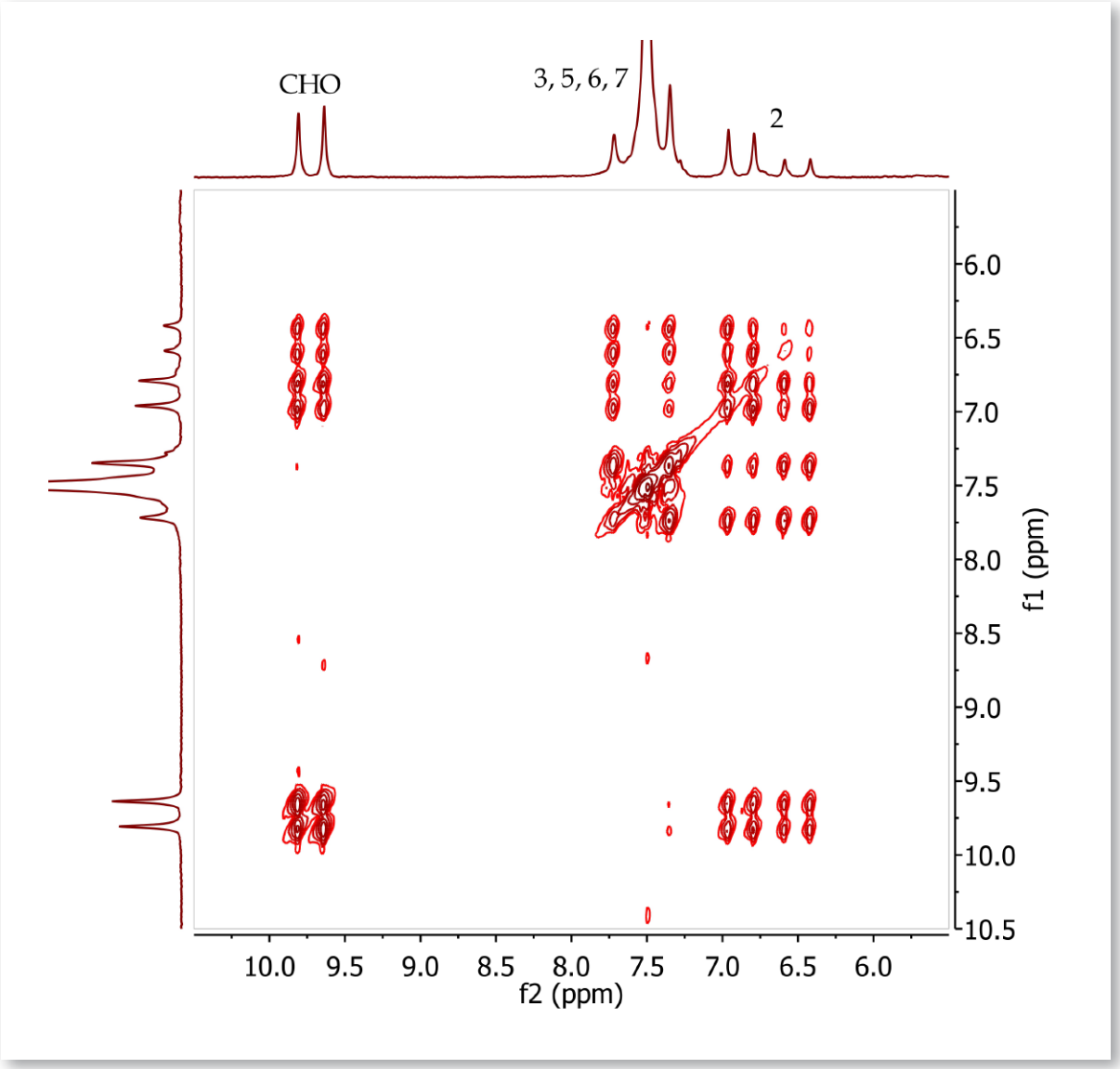

Figure 8. COSY spectrum of *trans*-cinnamaldehyde, $CDCl_3$.

The COSY spectrum of *trans*-cinnamaldehyde (Figure 8) clearly shows the correlations between the aldehyde proton (CHO) at 9.73 ppm and the alkene CH proton at position 2. The CH protons at positions 2 and 3 are also coupling to each other. COSY correlations are also observed between the aromatic protons at positions 5, 6 and 7.

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References

1) Pavia, D., L.; Lampman, G., M.; Kriz, G., S.; Engel, R., G. *Introduction to Organic Laboratory Techniques: A Small Scale Approach;* Thomson Brooks/Coles, 2005.

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