

Analysis of Chemical Flavourings in Chewing Gum

Session 2

In the previous session, you were presented with a problem where a manufacturer of chewing gum (*Pilgrims of Plymouth*) were receiving complaints about one of their products ('Moor-Mint'), and your task was to investigate the source of the problem. As part of the pre-laboratory exercise for the current session, you will have discovered that the main flavour component of spearmint chewing gum is carvone. You may also have discovered that this flavouring component is a relatively small part of the overall product.

Aims and Objectives

The most likely causes of the problem are either contamination from another chemical used in the factory, or an error in the quantity used in production **(Some students may also have noted that the main flavouring in spearmint, carvone, exists as two enantiomers and so the use of the 'wrong' isomer may also account for the difference)**. You will therefore address these alternatives by extracting any flavouring chemicals present in the chewing gum and quantifying them by Gas Chromatography (GC).

By considering your answers to the other pre-laboratory questions, together with the glassware and solvents available to you, discuss an experimental procedure for extracting the chewing gum, including the role of the Internal Standard. Decide, also, on the collation of all relevant data.

(Note: The class will be split into two. As a pair, you will carry out the extraction and analysis on either a sample of the 'normal' chewing gum (the control experiment) or a sample of the 'problem' gum)

Preparation of a solution of camphor in hexane (Internal Standard)

From the following statements, select the one that you think is the most appropriate, and then prepare your solution using the equipment and chemicals available to you.

- Prepare a solution of camphor in hexane which has an approximate concentration of 50 mg cm^{-3} but is accurately known (**BEST OPTION**).
- Prepare a solution of camphor in hexane which has a concentration of exactly 50 mg cm^{-3} .
- Prepare a solution of camphor in hexane which has an approximate concentration of 50 mg cm^{-3} .
- Prepare an Internal Standard solution by accurately weighing some camphor and dissolving it in some hexane.

Soxhlet extraction

Accurately weigh a strip of chewing gum (4 decimal places), add 1 cm^3 of your Internal Standard solution to the strip, chop the gum into suitably sized pieces, and place the chopped gum in the Soxhlet thimble ready for extraction. Add 150 cm^3 of methanol to a round bottomed flask. Assemble the apparatus and turn on the heating mantle. Once the methanol is boiling gently, continue the extraction for 45 mins (about 5-6 cycles) before turning off the heater and allowing the solution to cool (Place in an ice bath to promote cooling). When cooled, remove the round bottomed flask and filter your extract solution into a further pre-weighed round bottomed flask using a glass funnel lined with filter paper and cotton wool. Evaporate the solvent using a rotary evaporator. Re-weigh the flask and calculate the yield based on the mass of extract.

Model Building

Once the Soxhlet extraction has begun, construct a model of carvone using a molecule building kit. Note any differences between your model and the 2-D representation that you have drawn as part of the pre-laboratory exercise.

Session 2 Post-laboratory Exercise

As a preliminary evaluation of the extraction, consider the following points and decide whether you 'agree', 'disagree' or 'cannot be sure'.

- The mass of the extract was greater than expected given the composition data investigated previously (pre-laboratory exercise). **(AGREE)**
- The extraction was completely selective towards the flavouring compounds of interest. **(DISAGREE/CANNOT BE SURE – HIGHER MASS WOULD INDICATE DISAGREE)**
- The aroma of the extract indicates the presence of spearmint flavouring (carvone). **(AGREE)**
- The aroma of the extract from the 'problem' chewing gum is different from that from the 'normal' chewing gum. **(AGREE !)**
- If the mass of the extract was greater than expected, this means that (at least) all of the flavourings have been extracted. **(DISAGREE)**
- Addition of the IS should allow for accurate quantification of the amount of flavouring(s) even if the overall extraction was less than 100%. **(AGREE)**
- The crude extract may contain chemicals that are not suitable for analysis by GC. **(AGREE/CANNOT BE SURE)**

What other conclusions can you make at this stage ? Adapt the flow diagram that you started at the end of Session 1 to include any new information that you now have.

Session 3 Pre-laboratory exercise

From the discussion at the end of Session 2, you will have discovered that the extraction of chewing gum using methanol was probably not selective towards the flavouring compound(s) of interest. However, the extraction of at least some of the carvone must have been achieved as indicated by its characteristic smell. In order to remove unwanted sugars and other non-flavouring chemicals from the extract, it will be necessary to purify it prior to analysis by GC. This will simplify the analysis to those chemicals that are of interest, and prevent more polar compounds from modifying the properties of the GC column (highly polar or non-volatile compounds can stick to the column and do not elute).

- Investigate purification techniques that involve some chromatography. Suggest a method that should be suitable for the current extract.

GC may be coupled with Mass Spectrometry (MS) for combined separation and detection (GC-MS). MS gives additional structural information about chemicals eluting from the column, so that interpretation does not simply rely on GC retention times of authentic standards. In most cases, mass spectra contain information about the chemicals' molecular masses and constituent parts.

- *Simple:* Calculate the Relative Molecular Masses (RMM) of the aroma chemicals used by *Pilgrims of Plymouth*, together with that of camphor. Decide whether this information is going to be useful in identifying the chemicals.
- *Advanced:* Conduct a library search for the mass spectra of the aroma chemicals and that of camphor. How might this information improve your identification of individual chemicals?

RMM data for aroma chemicals:

Limonene (C ₁₀ H ₁₆)	136	
Carvone (C ₁₀ H ₁₄ O)	150	
Citral (C ₁₀ H ₁₆ O)	152	(E and Z isomers seen by GC)
Vanillin (C ₈ H ₈ O ₃)	152	
Menthol (C ₁₀ H ₂₀ O)	156	(M ⁺ at m/z 138 due to dehydration)
Camphor (C ₁₀ H ₁₆ O)	152	(Internal Standard)

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