

Analysis of Chemical Flavourings in Chewing Gum

Session 1

Aims and Objectives

The aim of this introductory session is to familiarise you with the use of aroma chemicals as flavourings, and the chemical features causing them to possess their characteristic aromas. By the end of this session you will have:

- Been introduced to some chemicals responsible for aromas and flavours
- Identified some chemical features which are often present in aroma chemicals.
- Been introduced to a 'real-life' problem concerning the use of aroma/flavour compounds in a food product.
- Suggested some possible causes of the problem.

Background to Aroma Chemicals

Many chemicals, especially organic chemicals, have distinctive aromas. The olfactory system responsible for smell is closely linked with the sensation of taste and, since the human sense of smell is around 10,000 times more sensitive than that of taste, it is thought that 90% of what we call taste is actually due to aroma. Consequently, the use of aroma chemicals is not only exploited by the perfumery and toiletry industries, but also by the food industry. Subtle variations in the composition of aroma chemicals used in foodstuffs can dramatically change the flavour and so the use of aroma plays a critical role in the success or failure of a food product.

Formulation of aroma chemicals for food is quite an involved process. A flavour can be generally considered as characterising, modifying, or differentiating. Characterising components are those that smell reminiscent of the flavour. A flavour will generally be built with a characteristic component as the base. For example, Ethyl 2-methylbutyrate (Figure 1) is characteristic of apple and is found naturally in apples. It is used in concentrations of up to 1% for fragrance, or 10 ppm for flavouring.

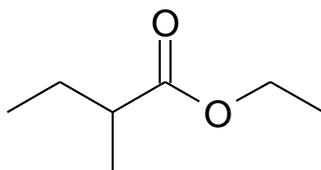


Figure 1. Ethyl 2-methylbutyrate

However, a characteristic flavour alone is usually 'flat', so modifiers and differentiators are used to add subtle nuances to the flavour in order to differentiate them from others on the market, and to make them more difficult for competitors to reproduce. Alterations to appearance, solubility in the product medium and unpredicted interactions are also factors which need to be taken into account, and these can cause problems in formulation and production.

Flavourings and aroma chemicals can be found everywhere in day-to-day life, from natural flavourings in fruits, to vast arrays of blended chemicals in perfumes. As a chemist, it is important for you to appreciate the chemical nature of these compounds, and the types of problems you could be faced with as an analyst.

In small groups, discuss and address the following:

- Name five natural flavours or aromas from fruits or plants.
- Identify some products that you associate with these flavours/aromas and decide whether they are usually considered as 'natural', 'artificial' or both.
- Can you identify any classes of organic chemicals with distinctive aromas/flavours that you are familiar with already? (Note: Think about functional groups in particular)
- Do you know of a specific compound which has a distinctive odour/flavour? If you know the structure, can you suggest which feature of the chemical's structure is responsible for the smell or flavour?
- A number of fragrant chemicals have been impregnated into some filter papers. Remove a strip of each one from the labelled containers using tweezers and make a note of any odours that you can detect. Are the fragrances familiar to you and, if so, what do you normally associate them with? Do you know the chemical names for any of these?

SUGGEST:

- 1. Spearmint oil, Eucalyptus oil, Citronella, Orange Oil, Tea Tree Oil, Vanilla essence. Can add others as available.**
- 2. 20 min for student discussion + 10 min feedback.**

Case Study: Contaminated Chewing Gum

One of the most common chemical fragrances you may have identified is spearmint, which is widely used in the food industry. As a case study, you will be required to investigate a problem presented by a local supplier (*Pilgrims of Plymouth*) of flavoured chewing gum ('Moor-Mint').

Pilgrims of Plymouth has recently been receiving complaints about its spearmint chewing gum. In particular, customers have been claiming that the gum is not as 'fresh' tasting as it should be. The company lacks the technical requirements to investigate the problem, and has therefore requested analysis by you and your laboratory. An extract from a recent correspondence is as follows:

"..... and it would appear that the 'freshness' of the mint flavour is not as it has been in the past. Our survey of customer feedback suggests that this problem is localised to one particular batch (MM2004-6) of Moor-Mint. We have checked our records and we can find no errors in the quantities of flavourings used in the manufacture of batch MM2004-6. However, it is plausible that our flavourings have become contaminated by other chemicals, including one or more of those used in some of our other products. Unfortunately, we do not have the appropriate expertise to investigate this further, and would appreciate your assistance in this matter. We are able to provide you with:

1. Samples of Moor-Mint (batch MM2004-6) (**Add S-carvone – 50 mg cm⁻³**)
2. Control samples of Moor-Mint corresponding to batches free of complaints (MM2004-1) (**Add R-carvone – 50 mg cm⁻³**).
3. A sample labelled 'Moor-Mint flavourings' believed to have been used for batch MM2004-6. However, this has *not* been confirmed. (**limonene/vanillin/citral/menthol**)
4. A summary sheet showing the names and structures of our most frequently used chemicals (Figure 2).

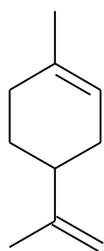
We look forward to hearing from you in the near future."

Discuss the background information to this case study and suggest:

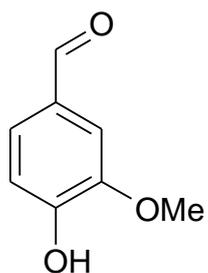
- (a) Some possible explanations to the problem
- (b) A general experimental approach to solving the problem

SUGGEST:

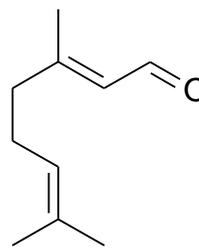
In this first session, provide samples of 'regular' and 'contaminated' gum to see if the students can detect any differences in smell (Spike R and S carvone to samples of gum).



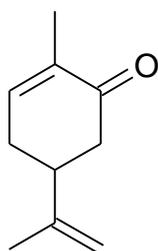
Limonene



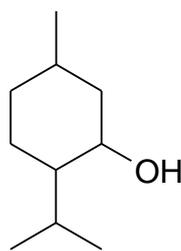
Vanillin



Citral



Carvone



Menthol

Figure 2. Fragrance and Flavourings Chemicals used by *Pilgrims of Plymouth*

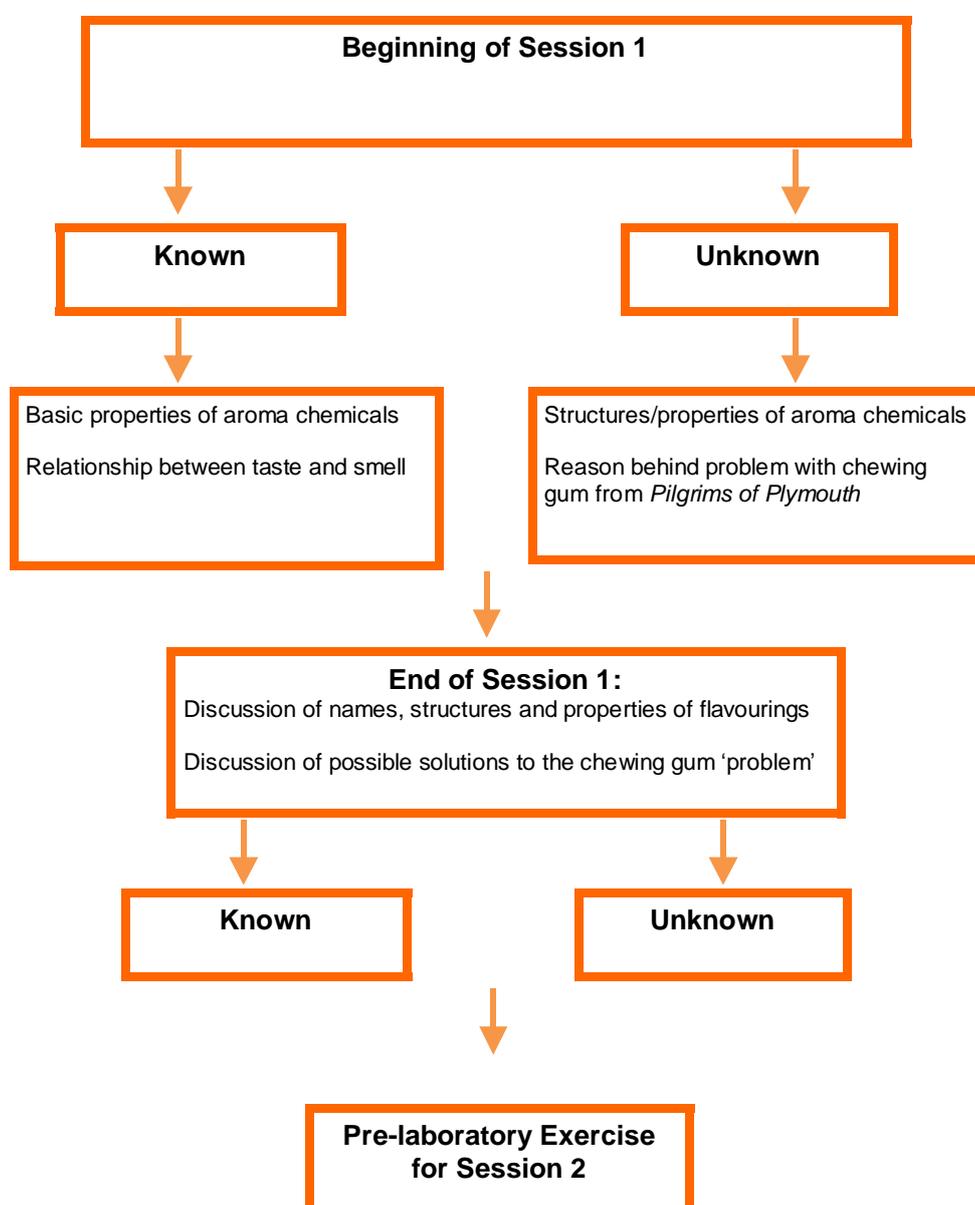
OPTION:

Run Post-Lab (Session 1) and Pre-Lab (Session 2) during Session 1 and get feedback at end of session (plenty of time in 3-4 hr Lab session)

Session 1 Post Laboratory Exercise

As preparation for your final report, begin a flow diagram that describes the information that you have collected during Session 1. You will build on this as the case study evolves. Be careful to compile information about what is known and what is not known at each stage. An example of this, corresponding to the end of Session 1, is shown in Scheme 1. However, this example is for guidance only. In order to prepare for your own final report, you will need to write with greater detail and in your own words.

Scheme 1. Suggested layout of session summary in preparation for final report



Session 2 Pre-laboratory exercise

A general approach for solving the chewing gum problem can be summarised as follows:

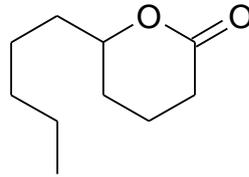
1. Extract the major flavour chemicals from samples of Moor-Mint including some from the (suspected) contaminated batch (MM2004-6).
2. Examine the composition of the 'Moor-Mint flavourings' sample by a suitable method.
3. Analyse and compare the extracts from samples of 'Moor-Mint' and decide whether 'Moor-Mint flavourings' was used for any of these.
4. Determine if any contamination has taken place.

Before Session 2, complete the following tasks which will help you complete the necessary laboratory work. You will not be able to start your laboratory work if you have not completed these.

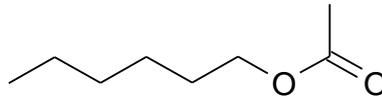
- Investigate some suitable methods for extracting the flavourings from the gum and suggest your preferred method.
- What are the main chemicals found in spearmint chewing gum ?
- You will analyse your extract using Gas Chromatography (GC). Find the boiling points of the 5 flavour chemicals used by *Pilgrims of Plymouth*, and use this information to predict their GC elution order.
- To quantify the composition of your extract by GC, you will need to add an Internal Standard (IS) to the samples of chewing gum prior to extraction. What are the advantages of carrying out the quantification this way ? What chemical/physical properties should the IS have ?
- The IS that you will use in the next session is camphor. Find the structure of camphor and estimate a suitable volume of camphor solution to use assuming that you have made a standard solution of 50 mg cm^{-3} (camphor in hexane).

Some Fragrance chemicals:

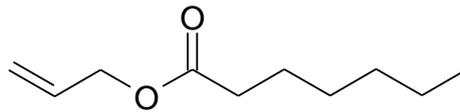
δ-Decalactone (peach, coconut)



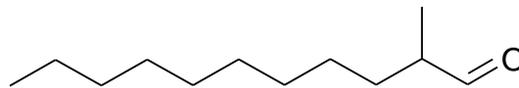
Hexyl acetate (fruity, pear)



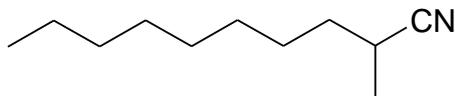
Allyl heptanoate (sweet, banana)



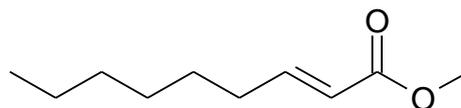
Aldehyde MNA (fresh, aldehydic)



Frutonile (soft, floral, fruity)



Beauvertate (vegetable, earthy)



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