Analysis of Chemical Flavourings in Chewing Gum

Tutor Notes

Overall aims

This laboratory-based resource consists of an investigation into the composition of flavourings chemicals present in chewing gum with the overall aim of resolving a 'problem' associated with certain samples. The investigation was designed with first year undergraduate chemistry students in mind, although it can also be used with Year 10 school children or final year undergraduate chemists following some straightforward modifications.

After a brief introduction to 'chemical flavourings', where students discuss their knowledge of common fragrances/flavourings, a problem is presented, which takes the form of some correspondence sent by a local manufacturer of chewing gum who has been receiving complaints about a certain product. The students consider various potential solutions to the problem and set about designing an experimental procedure that should address these alternatives. Over 4 sessions (maximum), students discuss the problem, carry out extractions from chewing gum samples (adulterated beforehand), perform purifications on the extracts, and finally analyse these using gas chromatography (GC) or gas chromatography-mass spectrometry (GC-MS). Samples of typical flavourings are also provided. The chromatographic analysis reveals that the most probable explanations to the problem that were discussed in the initial session (*viz.* incorrect flavourings and/or quantities used) are not supported by the experimental data. Instead, the mal-taste associated with some of the chewing gum samples is likely due to contamination of the authentic mint flavouring (R-carvone) with its enantiomer, which has a different smell/taste (S-carvone). An additional experiment involving polarimetry can be introduced to test this further, if desired.

Learning opportunities

This investigation provides laboratory experience in carrying out extractions, small-scale purifications and performing quantitative analysis using GC or GC-MS. It also gives students the opportunity to contribute to experimental design before working with agreed procedures. By alternating laboratory work with completion of pre- and post-laboratory exercises, students are encouraged to evaluate the outcomes of each set of measurements. Thus, students are able to identify progress and what further information is needed, at each stage.

Overall, the investigation integrates elements of introductory laboratory techniques (e.g. extractions, small-scale purifications) with structural organic chemistry (e.g. stereochemistry) and instrumental analysis. If the analyses are carried out using GC-MS (for 'real' or as supplied spectra), basic MS methods can be introduced to evidence theory. All flavourings are volatile, separate well using standard GC columns and have characteristic mass spectra.

Timeline (Note: A summary flowchart is included as one of the documents that maps out the various activities in, and between, laboratory sessions)

Session 1 (ca. 2 hours)

The first session focuses on a practical introduction to flavourings/fragrances and an outline of the problem to be investigated. Initially, students are given samples of flavourings/fragrance chemicals and these are described in general and more scientific

terms, where possible. Later, in this first session, the ‘problem’ is introduced and students discuss some potential causes (e.g. contamination, incorrect concentrations) and propose a general approach for an investigation. Since the investigation develops over several sessions, it is important to develop a good record of actions taken and outcomes, so students are asked to create a flowchart showing progress that will be built on in subsequent sessions (this is key for the final report).

In preparation for the next session, a pre-laboratory exercise guides students to establishing key information regarding the normal composition of chewing gum, extraction procedures and quantitative analysis of mixtures using GC or GC-MS methods. With sufficient time, this pre-lab exercise can also be run in the first session with feedback given at the end.

Session 2 (2-3 hours)

At the beginning of Session 2, it is useful to discuss (briefly) the outcomes from the previous session and summarise the main findings from the pre-lab exercise (e.g. carvone is the main flavour chemical in spearmint chewing gum, an internal standard is required for performing quantitative analysis by GC, etc.). Next, students consider various options for making a solution of an internal standard before agreeing on a suitable method. Having prepared the internal standard solution, students perform extractions (Soxhlet) of different samples of chewing gum. While the extractions are taking place, students build 3-D models of carvone, although the significance of this is not drawn attention to, at this point.

Having obtained an extract, students carry out a post-laboratory exercise, which is designed to help students reflect on the key experimental outcomes and, in particular, to identify what is known/not known at this point (Note: some solutions to the post lab exercise questions are given in the tutor version). The answers to some of the questions are definitive (e.g. the aromas of the different chewing gum samples are different, thus confirming the perceived ‘problem’), while others are more open-ended and require further investigation (the extraction procedure can/cannot be considered to be selective towards chemicals of interest (although answering this requires chemical analysis, the high mass of the extract compared with data found for the composition of chewing gum as part of the pre-lab exercise demonstrates that the extraction, in fact, cannot have been selective). Completion of this post-lab exercise is best done at the end of the session as it builds directly on the experimental work and informs some decision-making for the next session.

In preparation for Session 3, students complete a pre-laboratory exercise that requires students to research methods for purifying extracts (a requirement derived from the Session 2 post-laboratory exercise) and locate basic properties and mass spectra of some flavourings chemicals.

Session 3 (1-2 hours)

Feedback on the pre-laboratory exercise for this session should involve a brief discussion on chromatographic procedures for purifying extracts such that they are then suitable for analysis by GC methods. By considering the solvent system used for the extraction (non-selective) together with the polarities of flavourings and other components (e.g. sugars), a simple chromatography system can be designed. To aid this process, students are provided with a list of potential individual steps from which to compile an experimental procedure. Once agreed on, this procedure is then implemented to obtain purified extracts suitable for

GC analysis. A parallel procedure to determine purification efficiencies of the internal standard is optional at this stage, depending on the level of rigour desired.

In preparation for the final session, where the extracts are analysed by GC or GC-MS, a post-laboratory exercise (within the session) and a pre-laboratory exercise (to be completed before Session 4) are provided to the students. These exercises address key issues relating to the identification and quantification of individual chemicals by GC or GC-MS.

Session 4 (2-4 hours depending on student numbers & availability of instrumentation)

Session 4 involves the analysis of the purified extracts by GC or GC-MS. The experimental outcomes are compiled in the session, but the identification and quantification of individual chemicals are (likely) carried out later due to time constraints associated with GC analyses.

Students are encouraged to interpret their data with respect to the original suggestions for the problem (*viz*. contamination and/or variable abundances of flavourings between samples) and tutors should emphasise the importance of making interpretations that are most consistent with the data obtained, rather than working towards a perceived 'correct' answer. In practice, the data (and, therefore, the interpretations) can vary considerably between groups. If samples are adulterated beforehand through spiking of samples with known quantities of flavourings, then one interesting scenario involves the addition of equal amounts of the two enantiomers of carvone (R and S) to the two samples of gum. As a result, the composition of each extract will appear the same in terms of individual chemicals and abundances when analysed by GC or GC-MS (only the smell is different). Some students, however, may recognise the stereochemical feature through the model-making in Session 2 and have some prior knowledge of the different aromas of enantiomers. This scenario illustrates well the intriguing nature and difficulties of tackling certain problems, despite the apparent rigours of an experimental protocol and the elaborate nature of analytical instrumentation.

Documents available

1. Laboratory instruction sheets for students and tutors. The tutor versions are annotated versions of the student sheets with additional comments and suggestions.
2. A suggested template for the written report.
3. Pre- and post-lab exercises (these are contained within the instruction sheets).
4. Technical notes (lists of chemicals and laboratory equipment) and risk assessments.
5. A list of possible purification steps for use in Session 3.
6. A flowchart summarising how the investigation is structured and how the individual sessions are integrated. This flowchart puts emphasis on what is 'known'/'not known' at each stage and demonstrates progression.

Assessment

A report template document is provided that integrates the various aims of the investigation. This can be easily modified according to the tutor's individual aims.