Fatty acids in food supplements – Can you assume what you consume?

Tutor Notes

Overall aims

The primary aim of this study is to provide students with the opportunity to investigate the chemical composition of some food supplements that contain omega-3 and omega-6 fatty acids that are believed to have high nutritional value.

Briefly, the students are involved in designing different experimental procedures to extract omega-3 and omega-6 fatty acids from various capsules, perform the extractions, and use gas chromatography to identify and quantify the individual components. The outcomes are compared with any packaging information, where available.

The experimental work was originally designed with second year (or mid-stage) undergraduate chemistry students in mind, although it can be adapted easily for use with more/less experienced students. As written, the resource assumes a basic knowledge of the structures and properties of organic chemicals, together with the principles and applications of chromatographic methods. Some basic data manipulation is also required. The investigation is normally carried out over 3 laboratory sessions, but this can be modified quite easily depending on the group size and the extent to which various standards (for steps involving chromatography) are prepared in advance.

Learning opportunities

This laboratory-based investigation is set within an applied context and some background information is provided from a local government department (Department for Sustainable Development; DSD) regarding concerns over using cod as a source of omega-3 and omega-6 fatty acids. The role of the students (who work for a contract analytical facility; *Five Oceans*) is to provide specific analytical information regarding the composition of some food supplements that may have been derived from such sources. Although the main remit is to provide quantitative data regarding composition, these outcomes can also be interpreted with respect to some of the other information given; for example, the DSD indicate that the relative amounts of omega-3 and omega-6 fatty acids is a key factor for good nutrition, so the analytical data can be interpreted against this apparent benefit.

The study provides laboratory experience in carrying out extractions of different types and performing quantitative analytical measurements using gas chromatography (GC) methods. There is also an element of experimental design, although some guidance is given on this to make it suitable for the intended level.

Chromatographic separation and availability of standards of the fatty acids means that analysis by GC-FID is adequate and there is no need to use more advanced methods such as GC-MS (this could be an option, however).

The extractions work well with a range of commercially available ‘health food’ supplements and it is interesting to note, in particular, the vast range in both qualitative and quantitative data that is presented via packaging information; this provides a useful context in which to

interpret the analytical outcomes. To demonstrate this diversity, some example data from a range of commercial products are provided.

Timeline

Session 1 (1-2 hours)

In the first session, students are given some basic background information on omega-3 and omega-6 fatty acids and the key points raised by the DSD. The main aims of the investigation are also given. Students discuss some general approaches to the experimental work and then create 3 alternative methods based on a list of individual steps that are provided. Collectively, the 3 methods are largely 'chemical' or 'physical' in nature, or a combination of the two. If desired, this could be simplified to focus on one method only, but the comparison between methods is a useful addition. Having completed the 3 methods of extraction, the recorded data are interpreted within the session via the completion of a post-laboratory exercise that highlights what has been learnt and what remains to be established about composition and quantification.

At the end of the session, students are given a pre-laboratory exercise to complete – the DSD highlights the need to establish whether the fatty acids are in native or 'complexed' form, so students need to investigate experimental methods for distinguishing between these alternatives and, in addition, research analytical methods for quantifying (free) fatty acids.

Session 2 (3 hours)

A brief discussion of findings from the pre-laboratory exercise should demonstrate that the 'complexed' form of fatty acids are triglyceride esters and that these need to be saponified (hydrolysed), firstly, to confirm the status of the fatty acids in the capsules (free or otherwise) and, secondly, to permit analysis using GC methods. The students are asked by the DSD to carry out the experimental procedure according to a method used by another laboratory; however, the details are incomplete, so the students need to create a procedure from a set of 'notes' (Note: the tutor version of this session has a procedure that forms the basis of what experimental work should actually be carried out). The agreed procedure is carried our using extracts and standards to determine recovery efficiencies and the outcomes are interpreted via a question-driven post-laboratory exercise (Note: the conclusion is that most, if not all, of the fatty acids in the capsules are in the 'free' form). The laboratory work is concluded with the conversion of an aliquot of the extracts into methyl esters which are suitable for analysis by GC methods.

The pre-laboratory exercise for the final session requires students to do some investigation into the chromatographic properties of the methyl esters of the fatty acids (so-called FAMEs) in preparation for the GC analysis. Students are also required to investigate the reason(s) behind the investigation of the relative abundances of the omega-3 and omega-6 fatty acids, in particular.

Session 3 (Time depends on student numbers due to GC analysis)

At the beginning of the session, the findings from the pre-laboratory exercise are discussed alongside some labelled chromatograms of standards of fatty acids; thus, elution orders are

established/confirmed. The FAMEs are then analysed by GC (or GC-MS) and the data are analysed by the students to determine composition information.

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. Risk Assessments (COSHH) for laboratory sessions (these are the front pages of each laboratory session).
3. A suggested template for the report to be completed at the end of the investigation.
4. Pre- and post-lab exercises (these are contained within the instruction sheets and some solutions are provided via the tutor versions).
5. Technical notes (lists of chemicals and laboratory equipment).
6. A datasheet summarising the composition of some commercial supplements – this demonstrates the variety of products available and can be interpreted against product information

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. It is also recommended that students keep an on-going account of their note-taking and experimental work which should demonstrate progression throughout the study (a flow diagram might be suitable for this).