Fatty Acid Composition of Triglycerides

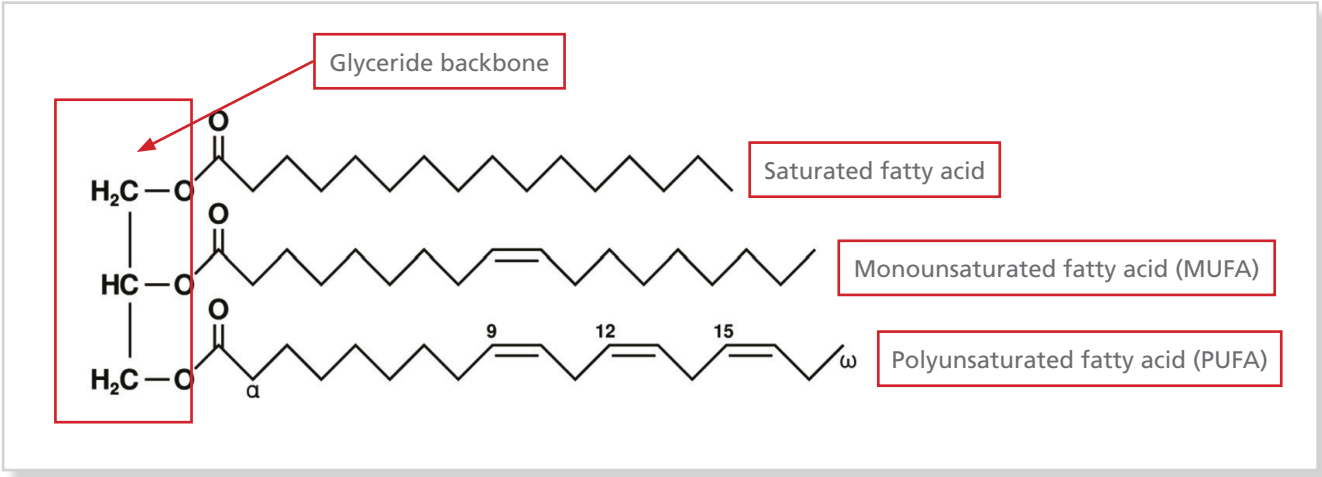
Application Note – Pulsar 005

Background

The fatty acid composition of oils and fats is important for health (for example, high levels of polyunsaturated fats and omega-3 oils are generally considered to be good for health; high levels of saturated fats are considered unhealthy). It will become mandatory in the EU at the end of 2016 to display levels of saturated fats on processed food labels; this is already mandatory in the USA, and other countries can be expected to follow. With this minimum requirement in place, food producers may choose to add further information where health benefits can be promoted through high levels of polyunsaturates and omega-3 oils. It is therefore important to have rapid, simple, and reliable methods for the measurement of fatty acid composition.

The traditional method of analysis

The traditional method for measurement of fatty acid composition of triglycerides is AOAC Method 996.06, which is a hydrolytic extraction gas chromatographic (GC) method. Under this method, fat is first extracted from the sample into ether and then methylated to fatty acid methyl esters (FAME) using boron trifluoride before running through the GC. Individual FAMEs elute in the GC at different times providing a means to separate chains of different length and different numbers of double bonds per chain (i.e. by degree of unsaturation). Often, information on chain length is ignored and the relevant peaks are simply summed to get a total of saturated, mono- and poly-unsaturated components.



A typical triglyceride

Pulsar

Pulsar is a high performance benchtop Nuclear Magnetic Resonance (NMR) spectrometer operating at 60MHz proton frequency. It uses a permanent magnet, so does not require liquid nitrogen, liquid helium, or compressed gasses, and it has no special health and safety requirements. It can be operated in a normal analytical laboratory by non-NMR expert laboratory technicians.

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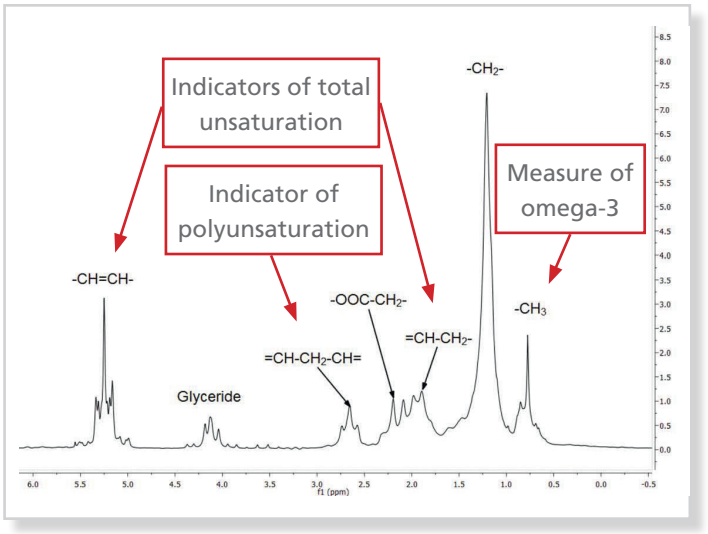
Fatty Acid Composition of Triglycerides

The **Pulsar** method

The **Pulsar** method has been developed in collaboration with the Institute of Food Research (IFR) in Norwich, UK. The method works by analysing the high resolution 60MHz NMR spectrum of an oil or fat sample, either in liquid form or extracted from a food sample using chloroform. The numbers of individual and multiple double bonds per chain are determined using a specially developed algorithm and this can be related to the relative concentrations of saturated, mono- and polyunsaturated oils in the sample. In addition, components having double bonds in the omega-3 position can also be identified, leading to quantification of the omega-3 oil content as well. All measurements are normalised to the signal from the four alpha protons on the glyceride backbone, so no calibration is required.



60MHz NMR spectrum of a typical unsaturated triglyceride

Measurement process

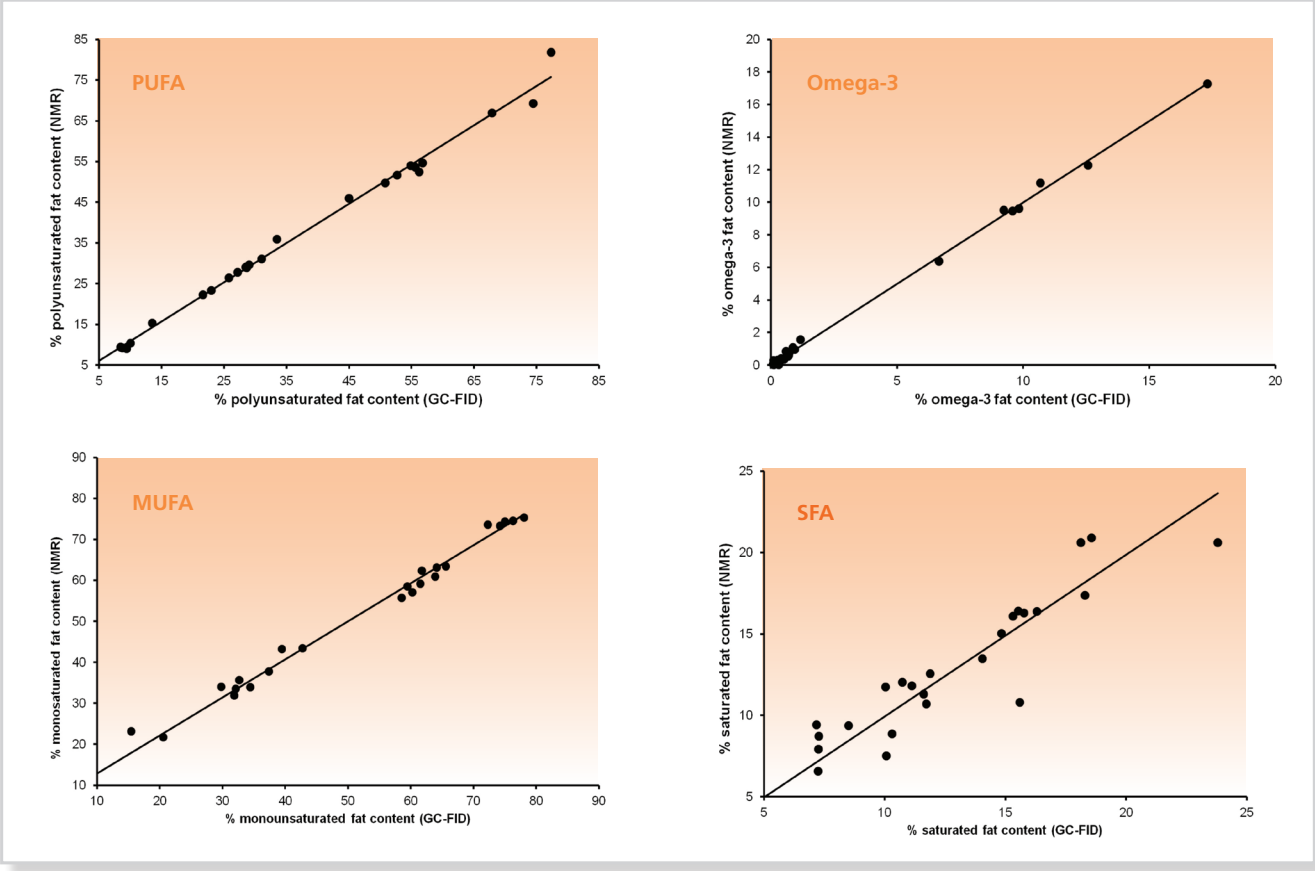
For a liquid sample, an aliquot of about 0.3ml is taken and shaken in a vial with 0.7ml chloroform to dissolve it. The mixture is then transferred into a 5mm NMR tube using a glass pipette. Fat samples, such as lard, can be dissolved directly in chloroform. Solid food samples are first homogenised, then a representative sub-sample of about 3 - 4g is shaken in a vial with 1.5ml chloroform. The mixture is then filtered, and pipetted into a 5mm NMR tube. The tube is then inserted into the **Pulsar** instrument and the special applications software run.

Measurement time

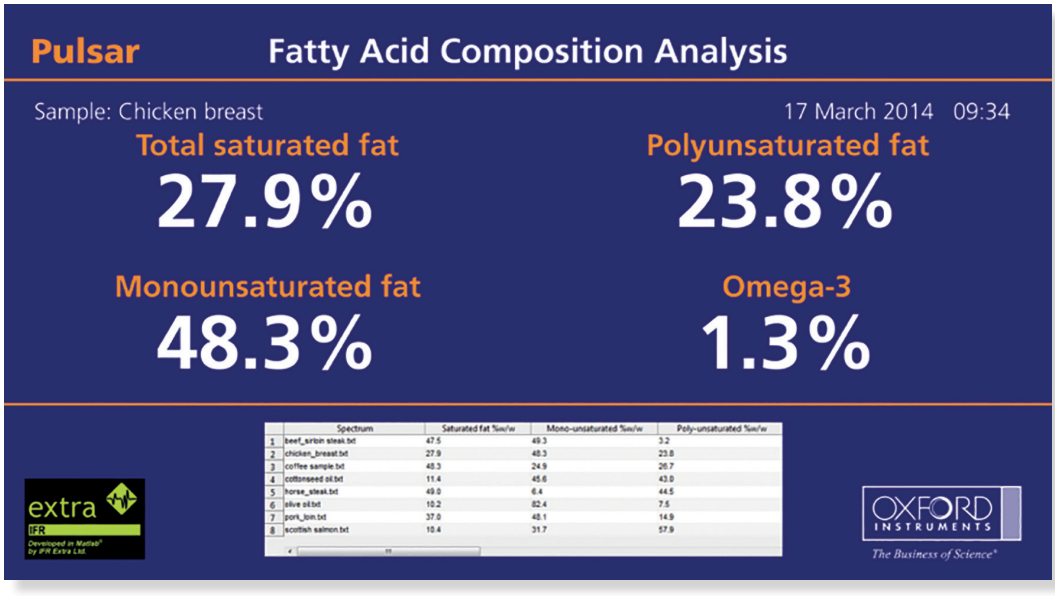
The total measurement time, including sample preparation, is typically less than 5 minutes for most samples, but could be up to an hour for very lean samples.

Results

The graphs opposite show the correlation between the **Pulsar** method and the standard GC method for the measurement of monunsaturated, polyunsaturated and omega-3 fatty acids, together with the total saturated fat content, in a variety of nut, seed and vegetable oils.



Pulsar results are presented as a final summary of the triglyceride components, as shown below:



Summary

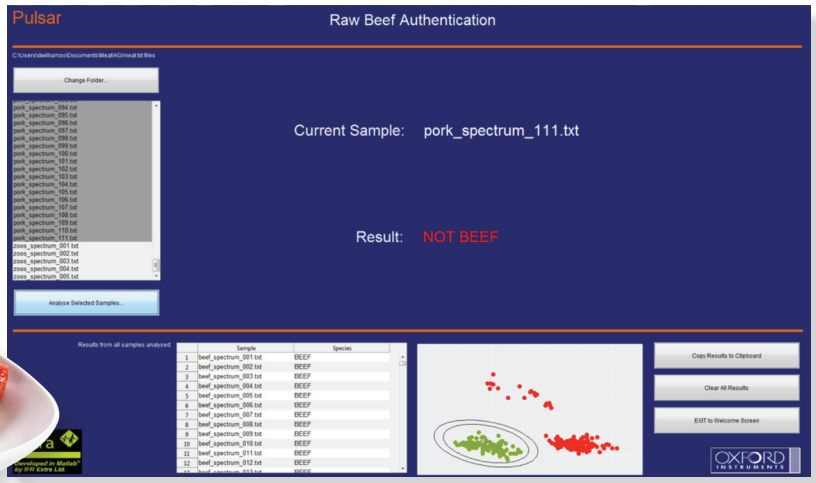
Pulsar provides a fast, convenient and reliable method of determining fatty acid compositions. The method does not require calibration, and it presents results clearly and directly. **Pulsar** does not require liquid helium, liquid nitrogen, or compressed gases, and can be operated in a normal laboratory environment without any special health and safety considerations.

This work receives support from the UK's Technology Strategy Board project no. 101250.

Also available:

Pulsar for meat speciation

The fatty acid composition can be further used to verify the species of a piece of raw or frozen meat for positive identification within the food chain.



MQC benchtop NMR analysers for fast and easy measurement of total oil or fat. Applications include:

- Fat in food
- Solid Fat Content (SFC)
- Oil in snack food
- Fat in chocolate
- Oil & moisture in seeds

visit **www.oxford-instruments.com/pulsar** for more information or email: **industrial@oxinst.com**

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