DEVELOPING LOW-FIELD NMR SPECTROSCOPY FOR TACKLING FOOD FRAUD

Dr Kate Kemsley
Institute of Food Research, Norwich

Dr David Williamson
Oxford Instruments, Tubney Woods, Oxford

In 2012, the Institute of Food Research began a three-year collaboration with Oxford Instruments to develop a new analytical technology – 'bench-top' NMR spectroscopy – aimed at use in industrial settings.

The project was supported by Innovate UK and the Biotechnology and Biological Sciences Research Council. The first application targeted was the measurement of saturation and unsaturation in vegetable oils. A few months into the project, the horsemeat scandal broke, and the team made a timely discovery about the potential of the NMR approach for meat analysis and authentication.

BENCH-TOP NMR – AN EMERGING TECHNOLOGY

Nuclear magnetic resonance (NMR) spectroscopy is a well-respected analytical technique used in chemistry laboratories worldwide. Oxford Instruments (OI) is a British manufacturing company that designs and produces analytical tools for industry and academia; OI pioneered the super-conducting magnets employed in NMR spectroscopy and a related technique, magnetic resonance imaging (MRI). Shared features of these technologies are that they are large and expensive, relying on super-cooled magnets and highly trained personnel to run them. With this in mind, OI recently launched a new benchtop NMR spectrometer, Pulsar™, aimed at making NMR spectroscopy accessible for routine testing. In contrast to their high-field cousins, low-field (60MHz) 'bench-top' spectrometers are based on permanent magnets and ease of operation.

To expand the market for bench-top NMR, key applications needed to be identified. Since 2012, OI has been working with the Institute of Food Research (IFR) to develop applications of interest to the food industry. IFR is the UK's only publicly-funded research institute that focuses on the underlying science of food and health, addressing challenges that include food safety, security and waste. The project is supported by Innovate UK (formerly the Technology Strategy Board) and the Biotechnology and Biological Sciences Research Council (BBSRC), with OI developing the instrumentation and IFR the statistical analysis and software tools. The first samples selected by the team for study were edible oils and fats, exploiting the ability of 60MHz NMR to produce high quality spectra quickly and easily from substances composed mainly of triglycerides. The spectra can be used to determine certain compositional values, such as the amount of mono- or poly-unsaturated fatty acids present in the sample, in a quick and accurate analysis. Distinguishing oils from different seeds and nuts is also possible [1].

A "KILLER APP"…

Early in 2013, a news story broke which dominated the headlines: the discovery of horse meat in beef burgers sold in the UK and Ireland. In the days that followed, undeclared horse meat was detected in a range of other processed meat products, leading to millions of pounds worth of food being recalled from supermarket shelves across Europe, and substantial brand damage to the companies involved. The crisis exposed the potential vulnerability of the meat supply chain to fraud, and highlighted gaps in testing regimes.

Currently favoured methods of verifying the source animals in meat products are based on DNA testing. But there are disadvantages to these methods: they can be prone to contamination and give false positive results; they are not reliably quantitative, and are relatively slow and expensive. DNA methods work by distinguishing one meat from another based on the distinct genetic profiles of each species.

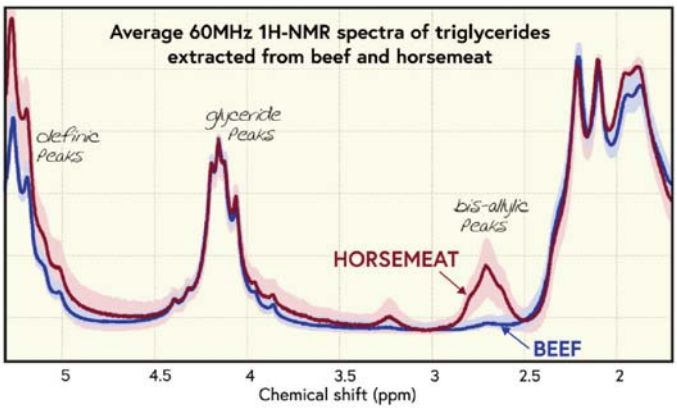
However, animals differ not only in their DNA; there are other compositional factors amenable to measurement which may also provide means of identifying species. For example, it is common knowledge that beef, lamb and pork fat are very different from one another. This is mostly due to dissimilarities in their fatty acid compositions, arising in turn from differences in their diets, digestive systems and metabolism.

The Pulsar bench-top spectrometer running the IFR-developed beef authentication software

This reasoning led the OI and IFR teams, in the wake of the horsemeat crisis, to carry out a series of experiments. The fat components of a range of meat samples – including fresh horse meat – were isolated using a simple solvent extraction, and spectra acquired using the Pulsar. The results obtained were striking and convincing: each of the different meats examined produced clearly different spectra. Over the following months, the method was refined and repeated on hundreds of meat samples across separate laboratories at OI and IFR, and the initial findings were confirmed.

In the case of beef and horse, the spectral profiles were found to be entirely distinct (figure 1). Even with natural variation, no overlap between the two types was found - the test was 100% accurate in determining whether an extract originated from a piece of horse or a piece of beef. The team recently reported their results in Food Chemistry [2].

Easy-to-use software to carry out mathematical analysis of the spectral data was developed at IFR, providing a complete system with which to authenticate beef, in a test that takes ten minutes from start to finish. A patent on this approach to meat speciation is currently pending [3]. The method is rapid and low cost; dozens of samples can be analysed per day, taking 10-15 minutes per test. This makes it an ideal and affordable approach for high-throughput screening, or for pre-screening ahead of more time-consuming and expensive DNA testing. The next step will be to transfer the technology into use within the food sector. In its current form, the test is suitable for key points in the supply chain, for example at meat wholesalers and processors, where the incoming raw materials are in the form of frozen blocks of trimmings. Trials of the system have recently been carried out in this kind of industrial setting, with the results now undergoing evaluation.



1. Low-field NMR spectra of triglyceride extracts of raw beef and horse meat. The shaded areas indicate the range of natural variation found: note that there is no overlap for certain of the spectral bands, e.g. the unsaturated bis-allylic features.

IN CONCLUSION

High-field NMR spectroscopy has long been recognised as a powerful analytical tool, but the equipment to carry it out has historically been too expensive and technically complicated to allow deployment anywhere apart from specialist laboratories. The advent of benchtop NMR looks set to change this landscape. The food sector applications developed by OI and IFR have demonstrated how useful the low-field modality can be, particularly for the analysis of fat-containing samples. The team are anticipating further collaboration on industrially important challenges as the capabilities of the Pulsar continue to evolve.

Find out more on our websites:

Institute of Food Research: http://www.ifr.ac.uk/

Oxford Instruments: http://www.oxford-instruments.com/

References

1 **60MHz ^{1}H NMR Spectroscopy for the analysis of edible oils.** Parker T, Limer E, Watson A, Defernez M, Williamson D, Kemsley EK. TRAC - Trends in Analytical Chemistry (2014) 57 147-158

2 **Authentication of beef versus horse meat using 60 MHz ^{1}H NMR spectroscopy.** Jakes W, Gerdova A, Defernez M, Watson AD, McCallum C, Limer E, Colquhoun IJ, Williamson DC, Kemsley EK. Food Chemistry (2015) 175 1–9

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