Nuclear Magnetic Resonance Technology Improves Sample Throughput

Contract Laboratory uses NMR to Eliminate Bottlenecks in Testing Food Fat Content

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One of Europe's leading independent testing laboratories sought an alternative to the standard solvent extraction/acid hydrolysis (Soxhlet) method for testing the amount of fat in a variety of foods. With numerous sites throughout the UK and Ireland, this contract laboratory provides quality control analytical services to the food industry. By converting from the wet chemistry method to a benchtop nuclear magnetic resonance (NMR) analyser for measuring fat content of foods, the lab reaped significant economic and environmental benefits.

Advantages of NMR over other secondary methods include:

- Calibration possible for samples with fat concentrations ranging from 0.5% to 100%;
- Primary calibration requires only a single fat sample;
- Recalibration needed infrequently;
- Sample measurement time is extremely short;
- Minimal sample preparation necessary;
- Eliminates the use of solvents;
- Suitable for bulk measurements;
- Insensitive to sample granularity and product additives; and
- Nondestructive technique, enabling repeat measurements.

Standard Wet Chemistry Methods Resulted in Bottlenecks

The contract quality control laboratory specialises in testing foodstuff samples, and promotes itself as a provider of a fast turnaround service. A typical customer request includes five or six measurements, including fat (oil) content. The standard Soxhlet method typically used for the oil measurement may take as long as six hours. This long sampling duration was reducing throughput and affecting the lab's ability to deliver its promised rapid analysis service. The process is also rather cumbersome, can be inaccurate, and requires highly skilled personnel. In addition, many of the hazardous chemicals used are becoming increasingly unacceptable according to international environmental standards.

Figure 1. The Oxford Instruments MQC

Using Alternative Methods to Speed Testing Throughput

The lab began seeking a rapid technique that would improve turnaround time without increasing operating costs, but would also be comparable to the industry standard Soxhlet technique.

A number of analytical methods can be used to conduct the testing. Such methods are often referred to as secondary techniques, since they are usually set up to match the results produced by solvent extraction. To provide a result equivalent to the traditional extraction techniques, secondary techniques must be correlated to the reference technique used.

Many secondary techniques may offer fast sampling, but also require frequent maintenance and calibration. Increased maintenance and consumables can add quite a bit to the cost of ownership. For example, supercritical fluid extraction (SFE) is reasonably fast, but the equipment involved requires a great deal of maintenance, and the cost of compressed CO_2 that is used to extract oil is also significant. Near-infrared spectroscopy (NIR) is sometimes used, but it is generally sensitive to the surface rather than the bulk of the sample and can be affected by several calibration and calibration maintenance issues. NIR calibration is complex because measurements are sensitive to product granularity and other physical characteristics and can be affected by additives such as seasoning, making it difficult to maintain accurate calibrations on a large variety of product types. This gives NIR limited applicability for the quality control of fat content in foodstuffs.

Low-field NMR provides a fast, direct, and user-friendly method for determination of the fat and oil content in foodstuffs. The technique is based on measurement of the NMR response obtained from fat in the product, and quantification of the fat content by simple and direct calibration without the use of chemometrics. One such easy-to-operate instrument is the Oxford Instruments MQC, which can be reliably operated without the need for skilled chemists or NMR specialists.

The Oxford Instruments MQC can be calibrated to cover a concentration range from 0.5% to 100% fat. The user can produce a primary calibration using a single sample of fat. The NMR technique yields calibrations that are very stable over the long term; recalibration is only required infrequently. Sample measurement time is short, typically about 20 seconds, allowing a high throughput of samples and efficient laboratory operation. Minimal sample preparation is required because the entire sample is normally loaded into a tube and measured directly, and several different-sized tubes are available.

With NMR, no solvents are required since the sample is analysed in its natural state. The instrument facilitates bulk measurement. Since the signal is generated from the whole sample, the result embodies everything inside the sample, not just on the surface. NMR is virtually insensitive to sample granularity and such additives as spices, flavors, colors, and salt. Finally, unlike Soxhlet, the NMR technique is nondestructive, so it can be used to conduct any required repeatability measurements.

Throughput Challenges Solved with use of NMR

The contract laboratory reviewed the potential alternative solutions that might help them reduce bottlenecks associated with fat measurements. They contacted Oxford Instruments to investigate the technology further and to verify whether the company's benchtop MQC NMR instrument would meet the laboratory's needs. The Oxford Instruments MQC NMR analyser is widely used to measure the oil content in foodstuffs and seeds.

Applications specialists from Oxford Instruments began by measuring the fat content of samples of some of the foods the lab typically analyses by both NMR and Soxhlet extraction. The testing process was designed to analyse 80% of samples using the MQC NMR instrument, achieve correlation to within 5% of the wet chemistry method, and achieve a repeatability of within ±5%.