

Food profiling by high resolution ^1H -NMR analysis

New frontier of authenticity testing

By Dr. Eric Jamin, Eurofins Scientific Analytics, France

A common request from quality control managers in the food industry is to have available a screening method that will not only monitor products or their ingredients for known hazards, but also detect whether a product has an unusual profile or contains atypical components, at an early stage of production.

Fingerprinting using ^1H -NMR (proton nuclear magnetic resonance spectroscopy) may provide the solution: it is fast (just a few minutes needed with little sample preparation) and it is widely applicable as every hydrogen containing molecule present in the sample will generate a specific signal.

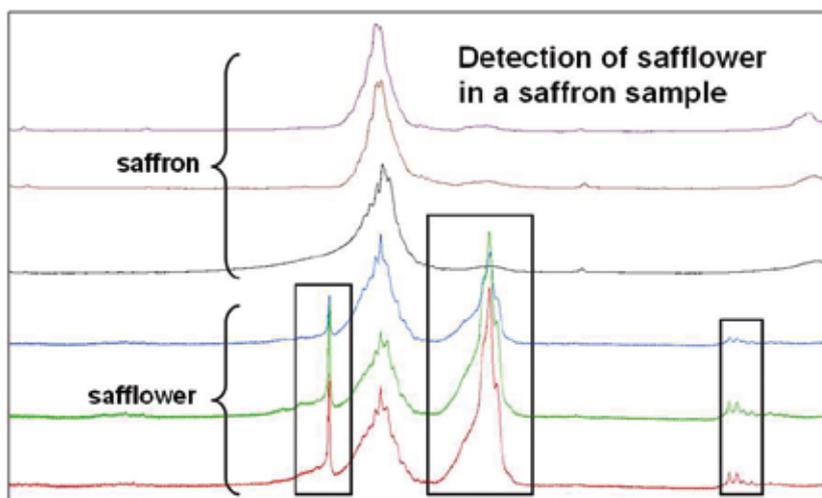
By comparing the resulting spectrum with a database of reference values the technique allows for a rapid quantification of a wide range of known components in a product, with a precision comparable to chromatography for example. It can also detect the presence of atypical components, which can be tentatively identified from the NMR signal. This makes

NMR profiling an ideal screening method for the purpose of preventing food crises such as that posed by melamine.

Eurofins is routinely applying this technique on fruit juices and has successfully established its potential on spices, oils and milk. On request, the technique can be adapted to liquid and solid samples of all kind of food. Spectral fingerprints can also be used to perform traceability checks on finished food products and ingredients within a "global control" scheme applied to the entire supply chain.

Since the analytical cost decreases rapidly with high sample numbers, ^1H -NMR profiling is particularly suited to the routine screening of large series of samples. In case of spectral discrepancies, the sample can then be further analysed by focusing test methods that target the specific compound or group of compounds. It is therefore also a cost effective screening method for monitoring product authenticity.

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Example application of profiling by HR- ^1H -NMR

Food allergens in the focus

By Dr. Carmen Diaz-Amigo and Dr. Bert Pöpping, Eurofins, Germany



The introduction of maximum threshold and action levels for food allergens has been intensely debated across countries that have allergen labelling regulations. The intention is that these thresholds will help industry to protect their customers, and regulators to enforce legislation. Eurofins' scientists have authored and co-authored two publications in the reputable, peer-reviewed *Journal of AOAC International*.

The paper by Dr. Carmen Diaz-Amigo and Dr. Bert Pöpping discusses the introduction of thresholds or action levels for allergen labelling and it clearly shows the benefits and limits of existing and emerging test methods. Furthermore, the authors suggest how industry, despite some variability and uncertainty in methods, can make meaningful risk-management decisions based on analytical results, and how

regulators can approach the formulation of wording within legislation regarding action levels.

The second publication (Abbott et al.) covers validation guidelines for food allergen ELISA assays. It is a milestone in harmonising how validation is conducted. It summarises the outcome of intensive work by experts worldwide over the last three years. The guidance provided will lead to more robust, reliable and cohesive analytical results, allowing industry to integrate analytical testing as a valid tool into the allergen control risk-management decision processes. It will also enable better food enforcement by government. This publication is co-authored by Dr. Bert Pöpping as well as other experts from Health Canada, the European Commission, the Australian National Measurements Institute, Nestlé

Nutrition, FARRP and ICC.

Eurofins is therefore not only directly helping its clients with the analysis and interpretation of results, but also actively engaged with industry and legislators to help drive forward the development of appropriate technical innovation and modern risk evaluation tools for the ultimate benefit of the consumer. This is a consequence of the scientific expertise and experience to be found within the Group and the recognition of this status by participation on panels like the AOAC. By contributing towards the cutting edge of development and information at the international level, Eurofins is a market-leader in translating this expertise into the highest-quality results for its clients.

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Determination of endosulfan in soybeans

By Johannes Jaschik, Eurofins Dr. Specht Laboratorien GmbH, Germany

Endosulfan is a persistent organochlorine insecticide which was banned in Europe a long time ago. However, in some parts of the world this pesticide is still available and in use. Due to the nature of endosulfan it accumulates in foods which have a high fat content such as peanuts or soybeans. One of the most important exporters of soybeans is Brazil, and the content of endosulfan in conventional and even organic Brazilian soybeans is a common problem.

In order to protect consumers, ensure brand confidence and for quality assurance purposes it is necessary to analyse soybeans for pesticide residues. As the final decision about shipments of Brazilian soybeans often depends on the result of pesticide residue analysis, it is important to have a reliable and sensitive method. Therefore, the method has to be able to determine the endosulfan isomers and its metabolite endosulfan sulphate.

Almost all laboratories that perform

pesticide residue analysis offer the determination of endosulfan. However, in order to achieve a reliable result in soybeans, the higher fat content of the product and the non polar properties of the endosulfan compounds has to be taken into account. Therefore, a combination of extraction method using non polar solvents (e.g. the worldwide recognized DFG-S19 method) and a detection with GC-system should be applied. Recent studies confirm that detection with the commonly used GC-MSD using the EI (Electron Impact) ionization mode is less suited to the detection of endosulfan at the required levels. A far better option is to use the long established GC-ECD procedure which allows detection at levels far below 10 ppb.

Eurofins offers a routine chromatographic method using GC-ECD detection for determination of endosulfan. This is the most sensitive and reliable technique for the determination of this pesticide at trace

levels in products with high fat levels that are more difficult to analyse.

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Implication of new GM-Rice varieties for food and feed production

By Nicholas Krohn, Eurofins GeneScan, Germany

Genetically modified (GM) plants have been developed and commercialised for crops such as soy, corn, canola and cotton in the past years. Increased efforts are being invested into the development of GM rice varieties, the staple food in much of Asia. Among the countries developing biotechnologically enhanced rice, China is taking a leading role.

Although no large scale production of GM rice is taking place, notifications on GM rice are listed in the European Rapid Alert System for Food and Feed (RASFF) since 2006. The GM rice line called 'Bt63' has been detected in several products originating from China - which is why all rice imports from China have to be analysed for the presence of 'Bt63' (commission decision 2008/289/EC). Apart from 'Bt63' other rice varieties are known to be in development such as 'Kemingdao' (KMD1) and 'KeFeng6'. The latter GM variety, 'KeFeng6', was presumably detected and notified in the

RASFF recently.

Analytical strategies for the detection of GM rice have to be designed to ensure detection of the above mentioned varieties as none of them are approved



for use in food and feed. Testing for 'Bt63' according to the community reference laboratory (CRL) method only, would comply with the EU Commission Decision, but would not detect these other GM rice varieties.

Eurofins offers a complete analytical

portfolio for the detection of GM rice varieties. Besides testing for 'Bt63' using the CRL method, newly developed methods enable the detection of 'KMD1' and 'KeFeng6'. Historically Eurofins leading competence in the area of GM testing ensures fast reaction times when it comes to the development of new GM testing methods for our customers.

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Hexanal - a parameter for the assessment of spiced oils

By Nadja Liebmann and Dr. Torben Kuechler, Eurofins Analytik GmbH – Wiertz-Eggert-Jörisen, Germany

Spiced oils in the market are enjoying an increasing popularity. They are produced from native or refined oils, where herbs, spices and other flavouring ingredients are added. To assess the quality of such spiced oils, similar criteria as for edible oils are recognized. For pure oils the oxidation status can be identified through the absence of certain sensory characteristics (e.g. no rancid attributes) and chemical parameters (e.g. peroxide value and anisidine value).

With regard to these parameters the following problems can occur: the detection threshold for sensory tests is raised because the flavour components from the ingredients cause an additional strong smell and taste in the product. Similarly, colourful ingredients can bias the result for the anisidine value.

For these products, other parameters that give information about the oxidation state of the spiced oil are needed. Hexanal is a secondary fat oxidation

product and is described as a marker for the beginning of spoilage in the scientific literature. However, in virgin olive oil hexanal is also present as a

component of the typical aroma. An authoritative limit is not given for the hexanal content, but a German non-governmental organisation used this parameter for assessing oils and spiced oils in the past.

Recently, Eurofins Analytik established a new test method for the determination of hexanal. The volatile compounds from the oil are concentrated using a dynamic headspace technique on an adsorbent material, separated via gas chromatography and determined by mass spectrometry. This modern method allows the determination of hexanal content down to 10 µg/kg.



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in brief

European Union consolidates plastic legislation

The European Commission is working on a consolidation of the Regulations concerning packaging and articles that come into contact with food, which are made from plastics. At the moment plastic materials are covered by the Plastic Directive 2002/72/EC which has already been amended 6 times. Additionally, there are several Directives and Regulations dealing with test conditions for plastics intended as food contact material.

Certain changes in test conditions such as time, temperature profiles and the food simulants to be used are under discussion. The Regulation is expected to be released during this year and, in contrast to the current Directives, it will come into force in all European States 20 days after publication without the need for adoption into national law.

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Nanotechnology - Opening Pandora's box?

Nanotechnology is the science of very small particles (10⁻⁹m) which often fundamentally change their properties at this size. The most well known example is the Lotus effect on cars from nanocoatings. However, nanotechnology has long found its way into packaging materials and food e.g. as anti-caking agent (SiO₂). BUND (Friends of the Earth) estimated that more than 90 nanomaterials are already used in more than 600 foods.

This raises questions such as: Do the products containing nanomaterials

need to be labelled? Are they safe? How does one recognise foods containing them? Is existing legislation sufficient to protect consumers but still able to allow the utilisation of the benefits of this technology?

At the recent International Nanotechnology Conference organised by Eurofins CTC in Hamburg with speakers from RIKILT, European Commission JRC, FERA, BfR, BLL, BUND and DLG, the issues surrounding its safe use, acceptance, and existing and forthcoming regulations were discussed.

At this well attended conference, the participants from a wide range of industry sectors as well as consumer organisations, actively engaged in the debate and discussion. After the success of this nanotechnology conference, follow-up events will be organised.

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New multi method for the determination of veterinary drug residues in meat and fish

Eurofins WEJ Contaminants developed a new multi-class, multi-analyte method for the simultaneous determination of about 90 veterinary drug residues (Benzimidazoles, Quinolones, Nitromidazoles, β-Lactams, Macrolides, Triphenylmethane Dyes, Avermectins, Sulphonamides and Tetracyclines) in meat and fish.

The method based on liquid chromatography-tandem mass spectrometry with electrospray

ionization (LC-MS/MS) has proved to be robust and time-saving due to the simple and rapid sample preparation. Full compliance with the Maximum Residue Limits (MRLs) defined by the Commission Regulation (EU) Nr. 37/2010 will be achieved for most of the analytes.

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Monitoring of perfluoroalkylated substances in food

The European Commission recommended the monitoring of perfluoroalkylated substances (perfluorinated compounds - PFCs) in food of plant or animal origin, such as fish, meat, eggs, milk and derived products, to enable an accurate estimation of exposure.

Especially Perfluorooctane sulfonate (PFOS), perfluorooctanoic acid (PFOA), their precursors and polyfluoroalkyl phosphate surfactants (PAPS) in particular need to be monitored to estimate the relevance of their presence in food.

Eurofins GfA Competence Centre for dioxins & persistent organic pollutants, offers the analysis of PFCs in food. At present more than 25 fluorinated and partially fluorinated compounds can be determined. All methods are regularly reviewed to extend the parameter list in accordance to EC-Recommendation 2010/161/EC from 17th March 2010.

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