

## Determination of mineral oil hydrocarbons

### Eurofins establishes groundbreaking online-HPLC-GC technique to separate MOSH- and MOAH-fractions

By Sabrina Ment, Dr. Claudia Schulz and Dr. Susanne Rathjen, Eurofins WEJ Contaminants, Germany

Currently, the main constituent of cardboard is recycled paper. During the process of recycling, mineral oils originating from inks used in newspaper printing may find their way into the cardboard. If food is packaged into cardboard boxes, such mineral oils can migrate into the food in relatively large quantities. Relevant food groups include rice, cereal flours, cereals, muesli, pasta, baking mixes, dried potato products and custard powder.

The mineral oil fraction of concern consists mainly of low molecular weight compounds (C16 up to C24), with the major portion being Mineral Oil Saturated Hydrocarbons (MOSH). About 15-20 % of the mineral oil fraction is composed of Mineral Oil Aromatic Hydrocarbons (MOAH).

In 2009 the German Federal Institute for Risk Assessment (BfR) issued a warning regarding the direct contact of large surface dry foods with recycled cardboard. Food packaging is subject to EU-Regulation 1935/2004 and must not be harmful to consumer health. The implementation of separate maximum levels for MOSH and MOAH originating from food

packaging manufactured using recycled paper is currently under discussion in Germany.

Conventional GC-methods only allow for the quantification of the sum of MOSH and MOAH. A new online-HPLC-GC technique enables now the separate quantification of MOSH and MOAH within one chromatographic run. Separation and clean-up of MOSH- and MOAH-fractions is achieved using normal-phase HPLC. Subsequently, the simultaneous transfer of both fractions to a dual-channel gaschromatographic system with two columns and flame-ionization detection (GC-FID) allows for the separate quantification of MOSH and MOAH.

Eurofins WEJ Contaminants established this groundbreaking technique and is able to offer the simultaneous determination of MOSH and MOAH from food products.

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# Antibiotic residues in dried distillers grains and other fermentation by-products

By John Reuther, Eurofins Central Analytical Laboratories, United States

The worldwide production of ethanol as a gasoline additive is growing rapidly. Commercial fermentation processes such as those involved in ethanol production often have by-products that are used in the food and feed industry. Ethanol plants use only the starch from the corn, and sugar from the sugar cane in the process. The remaining nutrients - protein, fat and fiber as well as residual yeast - are by-products which are currently used to create livestock feed such as Dried Distillers Grains with Solubles (DDGS) and "Brewer's Yeast" amongst others. Conservative estimates by the Food and Agricultural Policy Research Institute at Iowa State University in Ames indicate that the U.S. ethanol industry is projected to produce 43 million metric tons of DDGS during the 2013-14 market year, based on projected ethanol production of 16.8 billion gallons (63.6 billion liters), satisfying both US and global feed markets.

## Regulatory Status

The use of antibiotics is helpful to limit harmful bacterial growth during the early part of the fermentation process. In the US, FDA's Center for Veterinary Medicine (CVM) is responsible for governing the use of antibiotics in



agriculture. For several years manufacturers of fermentation-based ethanol and candidate antibiotics have brought food additive petitions and Generally Recognized as Safe (GRAS) petitions to CVM to allow use of specific

antibiotics in the DDGS process, including the use of virginiamycin, erythromycin, Penicillin G, and ampicillin. To date none of these have been approved, and thus CVM has no policy (and no MRL) with regard to residues of any antibiotics in fermentation by-products. A similar situation exists in Europe.

CVM scientists have developed screening methods based on liquid chromatography coupled with mass-spectrometry (LC/MS-MS) for the determination of residues of antibiotics and have monitored these levels for several years. Most notable is the LIC 4438 method which allows sensitive determination of 13 antibiotics. Eurofins Central Analytical Laboratories in the US and Eurofins WEJ Contaminants in Germany have adapted this method and

provide ethanol plants and the by-product trade with the tools needed for compliance monitoring, and support of regulatory petitions.

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# New method for direct determination of glycidyl esters using LC-MS

By Dr. Claudia Schulz, Dr. Susanne Rathjen and Sabrina Ment, Eurofins WEJ Contaminants GmbH, Germany

Edible fats and oils are subject to processing in order to improve quality, stability, and safety. Although this procedure removes a very large portion of impurities from the oil, the formation of process contaminants can result in a significant health risk. Along with monochloropropanediol (3-MCPD) esters, glycidyl fatty acid esters are of concern as they may occur in refined edible fats and oils and corresponding finished food products, including infant formula.

The toxicological relevance of glycidyl esters is just as unclear as the question of their hydrolysis within the human digestive tract. Glycidol itself – the epoxide of glycerol - has carcinogenic and mutagenic features. The German Federal Institute for Risk Assessment (BfR) presently assumes that all

the glycidol is released for glycidyl esters in the digestive tract. Thus, it is important to minimise glycidyl esters in food.

The analysis of the various 3-MCPD mono- and diesters and glycidyl esters is extremely complex with a couple of direct and indirect methods in existence. Certain indirect methods using GC-MS allow the determination of glycidyl esters by transforming them to 3-MCPD which is then quantified, however, this results in a larger uncertainty. Direct determinations of single 3-MCPD esters and glycidyl esters require a range of reference standards which – in case of 3-MCPD esters – currently are not commercially available.

In order to remain aware of the latest analytical developments

Eurofins WEJ Contaminants is an active member of various analytical working groups, e.g. the German BfR, the German Association for the Science and Technology of Lipids (DGF) and the American Oil Chemists' Society (AOCS).

Eurofins now offers a direct method for the quantification of the seven most relevant and commercially available glycidyl esters (C12 – C18) using two consecutive solid phase extraction steps followed by LC-MS based on a recent AOCS draft.

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# Analysis of heavy metals in food and feed at ultra-low levels

## Introduction of HR-ICP-MS

By Anders Svaneborg, Eurofins Environment, Denmark

Although a misnomer, the metals arsenic, cadmium, lead and mercury are often collectively called "heavy metals". What they have in common are their toxicity and omnipresence within our environment, mainly as a result of various industrial processes. They find their way into the food chain following their uptake from water and soil by plants and animals and as contaminants in mineral raw materials. Elevated levels of lead can lead to neurotoxicity in infants, cadmium is primarily nephrotoxic and may cause bone demineralisation. Mercury in its organic forms is extremely toxic to the nervous system while inorganic arsenic is responsible for cancer of skin, lungs and the urinary tract. In Regulation (EC) No. 1881/2006, the EU-Commission has set maximum limits for lead, cadmium and mercury in different food groups such as meat, marine products, cereals, vegetables, fruits and products thereof, fats and oils, milk, and infant formulae. The maximum levels for feed and feed ingredients are listed in Directive



2002/32/EC. Currently the EU is discussing further reducing the maximum permitted levels of cadmium in certain food groups.

A recently developed technique, High Resolution Inductively Coupled Plasma Mass Spectrometry (HR-ICP-MS) makes it possible to analyse for these metals in food with great specificity even at very low concentrations.

To use this very sensitive technique successfully, every single step in the process chain from sampling to final

result has to be optimised. It needs high quality clean room facilities, highly-skilled staff and years of experience with trace metal determination at ultra low concentration levels. Eurofins Denmark has successfully implemented this technique on a wide variety of matrices including fish, shellfish & crustaceans, wines and alcoholic beverages, and has validated the method for food and feed in general, including cereals, meat, fish and vegetable oils, dairy products (milk, baby milk, cheese etc.), vegetables and soft drinks. The sample preparation is carried out in accordance with the European standard methods EN13804 and EN13805.

Eurofins performs mineral and heavy metal analyses in many laboratories around the world which completes our offer in metals testing.

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## Eurofins Nantes becomes the largest single-site independent food testing laboratory in the world

By Fayçal Bellatif, Eurofins Food France



From left to right:  
Christophe Clergeau, Vice-President of the Pays de la Loire Region; Jean-Marc Ayrault, Member of the Parliament and Mayor of Nantes; Gilles Martin, CEO of the Eurofins Group.

On September 23<sup>rd</sup> 2011, Eurofins inaugurated a 3,000 m<sup>2</sup> extension dedicated to microbiological analyses to its laboratory in Nantes, France. The extension was completed in early summer and turns Eurofins Nantes into the largest single-site independent food testing laboratory in the world. It has

11,500 m<sup>2</sup> total laboratory floor space and capacity to analyse up to 1 million samples per year with a broad range of technologies.

It incorporates the latest state-of-the-art technology, combined with analysis and automation tools developed in-house to ensure high sample throughput in optimal conditions. The technological innovations are the result of the integration

of best practices identified from the 30 Eurofins laboratories in 25 countries active in food microbiology. The benefits should be reflected in high quality testing with short turn-around-times for customers.

In addition to being the largest single-site food laboratory, Eurofins Nantes also offers a unique concentration of complementary knowledge and technologies in food testing to assure the safety, identity, composition, authenticity, origin and purity of food and feed products. The wide range of analytical methods and technologies used in the laboratory include nutritional chemistry and molecular biology (GMOs, allergens), mass spectrometry, Nuclear Magnetic Resonance, DNA sequencing, etc.

With this comprehensive breadth of service offering the Nantes' site fits perfectly into the network of laboratories and Competence Centres within the Eurofins Group and contributes towards serving customers anywhere in the world.

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## in brief

### New regulation on food packaging from plastics

The European Union issued a new regulation on plastics used as food packaging material (Regulation (EU) No. 10/2011). This consolidates legislation from the last decades and introduces some fundamental new requirements. For the very first time a food simulant for all dry foods such as rice or sugar is defined. Furthermore, enhanced test protocols for products with long shelf life, and shared responsibilities for compliance with legislation along the value-added chain are the most important changes. The regulation became effective on May 1<sup>st</sup> 2011.

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### EU: ECJ decided on GM presence in honey

On September 6<sup>th</sup> 2011, a new judgement of the European Court of Justice (ECJ) stated that pollen should be considered as an ingredient, in terms of regulation (EC) 1829/2003, in products such as honey. Hence, honey and food supplements containing pollen from genetically modified organisms (GMOs) are subject to this regulation and are only marketable if an approval for the respective GMO is

given. Products containing pollen from approved GMOs have to comply with corresponding labelling requirements. For non-approved GMOs a zero tolerance applies.

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### Meet Eurofins at FI Europe in Paris

Eurofins will exhibit at the Food Ingredients Europe 2011 (FIE 2011 - <http://fieurope.ingredientsnetwork.com>) which will be held from **November 29<sup>th</sup> to December 1<sup>st</sup>** at the Parc des Expositions de Paris Nord – Villepinte. This fair is the opportunity to meet all leading ingredients suppliers in one place and review the latest product innovations from all over the world.

As a major provider of food quality and safety solutions including testing and auditing, Eurofins invites you to meet and discuss with the experts. The European Competence Centres will be represented with experts on vitamins, contaminants, pesticides, GMOs, allergens, authenticity...

Visit the Eurofins booth at n°**4A33**.

In addition, Eurofins will organise a **technical seminar on Food Safety Solutions** for the customers attending the fair, on November 30<sup>th</sup> at 1 p.m. Please contact us for free registration

and additional information regarding the programme.

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### New maximum levels for PAHs

In August 2011, the EU published new maximum levels for polycyclic aromatic hydrocarbons (PAHs). In addition to those for benzo[a]pyrene (BaP), maximum levels for the sum of 4 PAHs (BaP, benz[a]anthracene, benzo[b]fluoranthene, chrysene) have been included. In 2008, the European Food Safety Authority had concluded that the practice of using BaP as the only marker for PAHs in food is not sufficient.

Regulation No. 835/2011 is already in statute but will not be enforced until September 2012. Maximum levels currently in place have in some cases been lowered, while new ones covering heat treated meat (products) and cocoa beans and derived products have been established.

Eurofins WEJ Contaminants has established a GC-MS method for the determination of BaP, the so called PAH4 as well as further PAH spectra on request.

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## COMING EVENTS

| EVENT                  | DATE & PLACE                       | MORE INFO                            | CONTACT  |
|------------------------|------------------------------------|--------------------------------------|--|
| <b>FIE 2011</b>        | 29.11.-01.12.2011, Paris, France   | <i>Eurofins booth: 4A33, Hall 4</i>  | <a href="mailto:eventsfr@eurofins.com">eventsfr@eurofins.com</a>           |
| <b>Fruit Logistica</b> | 08.-10.02.2012, Berlin, Germany    | <i>Eurofins booth: A-06, Hall 21</i> | <a href="mailto:marketing-food@eurofins.de">marketing-food@eurofins.de</a> |
| <b>BioFach 2012</b>    | 15.-18.02.2012, Nuremberg, Germany | <i>Eurofins booth: 9-217, Hall 9</i> | <a href="mailto:marketing-food@eurofins.de">marketing-food@eurofins.de</a> |

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