1-in-5 laboratories fails European sunflower-oil test

European Commission (EC) controls on the quality of sunflower oil imported from Ukraine are being called into question following publication of a report showing that 1-in-5 analytical laboratories could not measure the presence of mineral oil in sunflower oil satisfactorily [1].

Despite this level of failure in analytical laboratories responsible for testing the presence of contamination in sunflower oil, the EC intends, in the autumn of 2009, to alleviate the measures foreseen in EC Decision (2008/433/EC), which prohibited the importation of sunflower oil from Ukraine unless it is accompanied by a valid certificate for the level of mineral oil present. The EC Decision stipulated that, even after the Ukrainian authorities put in place a control and certification system, EU Member States should effectively double-check the values declared in the certificate until further notice.

France first reported that Ukraine was exporting contaminated sunflower oil to the EC and European Union (EU) Member States via the Rapid Alert System for Food and Feed (RASFF) in April 2008. The EC then imposed restrictions on the importation of sunflower oil from Ukraine. Also, the EC Directorate General for Health and Consumers asked the Joint Research Centre (JRC) Institute for Reference Materials and Measurements (IRMM) based in Geel, Belgium, to scrutinize the capabilities of official control laboratories and industrial food laboratories to measure mineral oil in sunflower oil.

The IRMM organized the tests, in which 55 analytical laboratories from 17 EU Member States plus Switzerland and Ukraine participated. The IRMM sent four sorts of test samples (i.e. contaminated crude sunflower oil, contaminated refined sunflower oil, spiked sunflower oil and a mineral oil solution in n-heptane) to the laboratories, which then had to measure these blind samples using their in-house methods. Analysis of the results revealed that 78–85% of the laboratories were able to measure satisfactorily, depending on the test material, so that 15–22% were unable to do so satisfactorily.

No problems since July 2008

Following the EC Food and Veterinary Office’s positive assessment of the control system put in place by the Ukrainian authorities, double-check controls at import have not revealed any problems with unacceptable presence of mineral oil in sunflower oil from Ukraine since July 2008.

Karasek et al. [1] reported that a number of laboratories stated that they had just started in this field of analysis, so that, at the time of the interlaboratory comparison test, they were still busy with the in-house validation of analytical methods, and lacked experience with this type of analysis.

Some participants in the interlaboratory comparison proposed application of a standard calibration solution and standardization of analytical methods for the determination of mineral oil in sunflower oil. They also considered that application of a well-defined, harmonized analysis procedure would minimize inconsistencies related to calibration and data analysis, according to Karasek et al [1].

Reference


SERS chips are up for arrays

Surface-enhanced Raman scattering (SERS) has passed the first test as a label-free technique with high specificity and sensitivity that could mean that it is preferred over immunoassays and DNA microarrays for array studies in biological applications [1].

Justin Abell and colleagues at the Nanoscale Science and Engineering Center, The University of Georgia, USA, proposed a method of fabricating a microwell-arrayed SERS chip on a standard glass microscope slide. They used oblique angle deposition (OAD) to produce a SERS-active surface, then a well-array patterning mold in which liquid polydimethylsiloxane (PDMS) was added and cured by low-temperature heating.

To test the virus-detection capability of the SERS multi-well-patterned substrate, Abell et al. used the avian influenza virus (AIV).
Phthalates may contribute to pre-term births, according to researchers at the University of Michigan School of Public Health, USA, who found that women who deliver prematurely have, on average, up to three times the metabolite levels in their urine compared to women who carry to term [1].

Women were recruited during pre-natal visits at a clinic in Mexico City, and Professor John Meeker and colleagues looked at data from 60 women: 30 who carried to term and 30 who delivered prematurely (defined as less than 37 weeks’ gestation). They analyzed urine samples collected during the third trimester and compared them to the control group who carried to term. The analysis covered 11 metabolites, which seemed to be active toxicants, as opposed to the parent compounds.

The analytical approach involved enzymatic deconjugation of the metabolites from their glucuronidated form, solid-phase extraction, separation with high-performance liquid chromatography, and detection by isotope-dilution tandem mass spectrometry. The limits of detection were in the low ng/mL range for each phthalate metabolite. They found significantly higher metabolite levels in the women who delivered prematurely.

Phthalates are commonly used in plastics, personal-care products, home furnishings (e.g., vinyl flooring, carpeting and paints) and other consumer and industrial products. The toxicity varies by specific phthalates or their breakdown products. Past studies have shown that several phthalates cause reproductive and developmental toxicity in animals. Human studies have reported associations between phthalates and gestational age, but this was the first study to look at the relationship between phthalates and premature births.

“We looked at these commonly used compounds found in consumer products based on the growing amount of animal-toxicity data and since national human data show that a large proportion of the population are unknowingly exposed.” Meeker said. “One of the problems for consumers is that you don’t know exactly which products contain phthalates because the products do not have to be labeled accordingly.”

Meeker viewed this study as a stepping stone to larger, more detailed studies examining the role of phthalates in premature births. The researchers hope to examine a larger population of pregnant women to corroborate these initial findings, and to explore experimentally the biological mechanisms of how phthalates work in the body.

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Biomass fluorescence tracks breakdown

The natural auto-fluorescence of plant cell walls can dynamically track solubilization during an ionic liquid pretreatment of a biomass sample, according to researchers at the Joint BioEnergy Institute (JBEI), a U.S. Department of Energy Bioenergy Research Center led by the Lawrence Berkeley National Laboratory (Berkeley Lab) [1].

Understanding how ionic liquids are able to dissolve lignocellulosic biomass should pave the way for new and better varieties for use in biofuels.

“Working with switchgrass and using the ionic liquid known as EmimAc (1-n-ethyl-3-methylimidazolium acetate), which is currently...
the most effective for pre-treating biomass, we observed a rapid swelling of the secondary plant cell walls within 10 min of exposure at relatively mild temperatures (120°C) (as shown in Fig. 1),” said Blake Simmons, Vice President of JBEI’s Deconstruction Division and principal investigator for this research. “We attributed the swelling to disruption of inter-molecular and intra-molecular hydrogen bonding between cellulose fibrils and lignin. The swelling was followed by complete dissolution of biomass over 3 h. This is the first study to show the process by which biomass solubilization occurs in an ionic liquid pretreatment using these techniques.

“In comparison to untreated biomass, ionic-liquid-pretreated biomass produces cellulose that is efficiently hydrolyzed with commercial cellulase cocktail and provides sugar yields over a relatively short time interval,” he said. “We are now in the process of evaluating other ionic liquids to discover the optimal combination of cost and performance.

“Our approach can be used to evaluate the deconstruction of lignocellulosics in biomass of different chemical compositions, and also to assist in determining the impact of genetically-engineered feedstocks,” said Simmons. “By utilizing this technique, the development and the selection of pre-treatment conditions for the selective solubilization and the fractionation of either polysaccharides or lignin could be tailored for the development of cost-effective biomass pre-treatments with enhanced yields of sugars.”

The ultimate goal is to find an ionic liquid that can efficiently pre-treat biomass, and then scale up its use into a cost-effective process for biorefineries.

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**Wastewater maps illicit drug use**

Chemical analysis of untreated municipal wastewater has found patterns in illicit drug use across Oregon in the USA [1].

The findings showed how a one-day snapshot of drug excretion might be used to understand patterns of drug use over time and provide a proactive tool for health officials.

The researchers tested the samples from 96 wastewater-treatment plants for evidence of methamphetamine, cocaine and 3,4-methylenedioxy-methamphetamine (MDMA, or ecstasy).

“This work is the first to demonstrate the use of wastewater samples for spatial analyses, a relatively simple and cost-effective approach to measuring community drug use,” said lead author Caleb Banta-Green. “Current measures of the true prevalence of drug use are severely limited both by cost and methodological issues. We believe these data have great utility as a population measure of drug use and provide further evidence of the validity of this methodology.”

“Municipalities across the state generously volunteered to help us test our methods by collecting samples more or less simultaneously, providing us with 24-h composite influent samples from one day –

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**Figure 1.** These fluorescence images of a stem of switchgrass treated with EmimAc ionic liquid show the section (a) before treatment, (b) 20 min after treatment, (c) 50 min after treatment, and (d) 2 h after treatment, when the organized plant cell wall structure has been completely broken down.
4 March 2008,” said Jennifer Field, who led laboratory analyses of the samples at Oregon State University.

The researchers centrifuged raw influent samples and used large-volume injection (LVI) followed by liquid chromatography coupled to tandem mass spectrometry (LC-MS²), which they optimized to eliminate the need for solid-phase extraction as a sample-preparation step [2].

These samples from 96 municipalities represented 65% of Oregon’s population, the researchers calculated the presence, measured as index loads, of three stimulant drugs – methamphetamine, MDMA and benzoylcegonine (BZE, a cocaine metabolite).

The researchers found that the index loads of BZE were significantly higher in urban areas and below the level of detection in some rural areas. Methamphetamine was present in all municipalities, rural and urban. MDMA was at quantifiable levels in less than half of the communities, with a significant trend toward higher index loads in more urban areas.

A cautionary note was that the data used for this study were inadequate as a complete measure of drug excretion for a community or the entire state. The team took only single-day, mid-week samples. Results might be altered depending on the day or time of year the sample was gathered.

“We believe this methodology can dramatically improve measurement of the true level and distribution of a range of illicit drugs,” said Banta-Green. “By measuring a community’s drug-index load, public-health officials will have information applicable to a much larger proportion of the total population than existing measures can provide.’’

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PCR picks out prion proteins
An immuno-quantitative real-time PCR assay for prion proteins, such as those involved in transmissible spongiform encephalopathies [e.g., new-variant Creutzfeldt-Jakob Disease (nv-CJD), scrapie or bovine spongiform encephalopathy (BSE)], has shown a 1000-fold increase in detection sensitivity over the commercial assay with which it was compared [1].

Developed by Tim Mcallister of Agriculture and Agri-Food Canada and colleagues, the assay was made possible by a direct conjugate of a prion-specific antibody and a synthetic 99-bp DNA tail. The DNA tail was engineered to include a single ScrFI restriction site, which enabled subsequent quantification of restricted DNA tails using real-time PCR. They tested the assay with scrapie prions bound to polyvinylidene difluoride membranes and compared it to 96-well plates coated with a capturing antibody from a commercially available immuno-based assay (TeSeE).

Detection of prions from diluted scrapie-positive brain homogenate bound to membranes was linear over the range 106–3.24 × 10² epitopes.

“Given its sensitivity and versatility, the present assay has potential to enable rapid and reliable detection of agents causing transmissible spongiform encephalopathies,” concluded Mcallister et al.

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References

NiO senses toxic chemicals
Extremely pure nickel-oxide (NiO) nanoparticles can be used to detect toxic industrial chemicals (TICs) and biological warfare agents (BWAs) [1].

The challenge is to design a material that reacts quickly and reliably to a variety of chemicals, including TICs, when incorporated into a sensor, according to Professor Patricia Morris, research-team leader at The Ohio State University, USA.

“These are sensors that a soldier could wear on the battlefield, or a first responder could wear to an accident at a chemical plant,” she said. Other applications could include exhaust-gas, pollution and air-quality monitoring.

Morris, with Ohio State doctoral student Elvin Beach, is interested in how NiO’s electrical conductance changes when toxic chemicals in the air settle on its surface. Beach applies a thin coating of the material onto microelectro-mechanical system (MEMS) to identify known toxic substances.

The key to making the sensor work is how the NiO particles are made. Beach and Morris have devised a synthesis method that yields particles that are very small (~5 nm, so that the sensor has a large surface area to capture chemical molecules from the air) and very pure (so that the sensor can detect even very small quantities of a substance).

“Basically, you mix everything together in a pressure vessel, pop it in the oven, rinse it off and it’s ready to use,” said Beach.

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Reference
After he removes the NiO from the pressure cooker, he washes it in solvent (methyl ethyl ketone) to free up the nanoparticles.

Next, with a microsensor silicon-chip array (Fig. 2), Beach adds a layer of particles using a picoliter (pL) drop dispenser – the first time that NiO nanoparticles have been applied in this way, according to Morris.

A 1-g batch of NiO nanoparticles costs about $5.00 to make; one chip carries 4 ng of material, so each sensor costs little to fabricate.

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**Nanobiosensors set to increase**

Nanobiosensors have the potential to account for half the biosensor market by 2020, according to Master of Engineering graduate Stanko Nedic of the School of Electronics and Computer Science (ECS) at University of Southampton, UK [1].

Nedic reviewed mainstream biosensors and compared them with high-performance biosensors based on materials such as nanowires and nanotubes.

He also concluded that silicon nanowire-based devices present the most promising prospect for the future due to their complementary metal-oxide semiconductor (CMOS) compatibility.

He found that modern biosensors used for medical testing are inappropriate for point-of-care diagnostics due to constraints that can be addressed by using highly-portable, cost-effective and high-performance nanobiosensors, which can readily detect glucose and many other biomolecules simultaneously and with ultra-high sensitivity.

“The demand for nanobiosensors is rising fast in response to the increase in obesity and diabetes,” said Nedic. “However, several roadblocks for commercialization of nanobiosensors have been identified, most important of which is the need for low-cost mass-production schemes.”

As it happens, Nedic’s supervisor, Professor Peter Ashburn, Head of the Nano Research Group at ECS has just been awarded a grant of £1.33m ($2.14m) by the Engineering and Physical Sciences Research Council (EPSRC) to develop a unique method for fabricating nanowires, so that blood-testing kits can be mass-produced.

Due to open this month (September 2009), the Southampton Nanofabrication Centre, which provides one of Europe’s leading multidisciplinary and state-of-the-art cleanroom complexes, will make cost-effective mass production of such blood-testing kits a reality.

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**Pittcon co-programs for 2010**

The organizers of Pittcon are to expand its co-programming next year, when the annual event is due to take place 28 February–5 March 2010 in Orlando, Florida, USA.

“These newly formed and existing relationships add diversity and strength to our Technical Program,” said Pittcon Program Chairman Janeth Pifer. “Co-programming offers a mutually beneficial venture, which permits organizations to combine ongoing efforts with
Pittcon to help achieve the common goal of advancing the chemical and laboratory sciences on a global level.”

For the first time, the Association for Laboratory Automation (ALA) has formed an education-based collaboration with Pittcon to focus on student research involving laboratory automation.

The Japanese Professionals’ Network in Advanced Instrumentation Society (PAI-NET) has also agreed to participate. The primary focus of PAI-NET is to make available the best practice of instrumental analytical technology to promote peace and prosperity in the world of tomorrow. Its vision is to educate technicians in and promote the usage of instrumental analysis.

The American Chemical Society will again present symposia at Pittcon 2010. There will also be technical sessions by the Society for Applied Spectroscopy (SAS), the Association of Lab Managers (ALMA), the Japan Analytical Instruments Manufacturers Association Society (JAIMA), and the Society for Electroanalytical Chemistry (SEAC), and speakers from ANALITICA Latin-America.

Plenary Lecturer on the opening day (28 February 2010) will be Professor Alan G. Marshall of Florida State University and Director of the Ion Cyclotron Resonance Program at the National High Magnetic Field Laboratory.

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Aggressive youths are chemically different

Delinquent adolescent boys with conduct disorder (CD) have much higher levels of platelet poor plasma (PPP) serotonin (5-HT) than normal controls, according to research by Pavel Golubchik of Geha Mental Health Center, Israel, and colleagues [1].

Their study involved 16 delinquent adolescents and 14 normal healthy controls aged 14-19 years. All the delinquent adolescents met diagnostic criteria for CD, had police records, and were held at the Adolescent Correctional Services Facility of the Ministry of Social Affairs.

The adolescents’ PPP 5-HT concentrations were determined by high-performance liquid chromatography with electrochemical detection (HPLC-ECD). The HPLC system comprised a high-pressure pump, siloxane injector, a reverse-phase chromatographic column, and an electrochemical detector equipped with a stainless carbon electrode. The mobile phase comprised a monochloroacetate buffer containing EDTA. For 5-HT determination, ethanol was added. All samples were assayed in the same run to avoid inter-assay variability. The lower detection limit was 0.2 ng/mL and the coefficient of variation was 5%.

Golubchik et al. found that delinquent CD adolescents had levels of PPP 5-HT about 3 times that of the normal controls. Moreover, the level of PPP 5-HT in the delinquent adolescents correlated positively with severity of aggression, as had been shown previously with whole blood 5-HT. They therefore concluded that juvenile delinquency was associated with high PPP 5-HT levels, and that modulating 5-HT neurotransmission might have a role in treatment of adolescents with severe CD.

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Reference

Fingerprints found on spent bullets

Fingerprints can be found on spent bullets, following advances at the University of Leicester, UK, where the technology is being developed in collaboration with Northamptonshire Police.

With University scientists, John Bond of the Northamptonshire Police Scientific Support Unit and an Honorary Research Fellow at the University’s Forensic Research Centre developed a method to visualize a fingerprint even after the fingerprint itself had been removed.

Now, Alex Goddard has uncovered a natural technique that involves studying the chemical and physical interactions occurring between the metal and the deposits of sweat from a fingerprint. Using advanced surface-imaging techniques (e.g., atomic force microscopy), nanoscale observations of fingerprinted brass samples can identify optimum conditions to promote the natural enhancement of the fingerprint, greatly improving their recovery rate (Fig. 3). Components of the sweat deposit survive washing and wiping of the surface.

“Once a finger has touched the metal surface, a residue remains behind,” Goddard explained. “This starts to react with the metal and an image of the fingerprint can be developed by use of elevated temperature and humidity, with the resultant image becoming a permanent feature on the surface of the metal.”

“This new technique promotes a naturally-occurring process, which does not involve adding anything to, or damaging, the evidence. Instead, it employs heat and humidity to promote the enhancement of the fingerprint image. There are also indications that it could be used after other techniques have failed, perhaps as a last resort,” said Goddard.

“I am delighted that this research in the Chemistry Department is producing really interesting and useful
results,” commented Bond. “This is an important area of forensic research and Northamptonshire Police is proud to be associated with the University. I look forward to further developments.”

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Best instrument at ASMS 2009

Instrument Business Outlook selected the LTQ Velos ion-trap liquid chromatography coupled to mass spectrometry (LC-MS) system the most innovative product from the American Society of Mass Spectrometry (ASMS) 2009 meeting in Philadelphia, PA, USA.

The LTQ Velos features a dual-pressure trap and advanced ion optics, which make it the world’s fastest and most sensitive ion trap mass spectrometer, according to manufacturer Thermo Scientific, which has available an application note on its performance [1].

“The sensitive detection and identification of components within a complex proteome sample is crucial for the characterization and understanding of proteome dynamics,” concluded the authors. “The technological advancements of the LTQ Velos dual-pressure linear ion trap mass spectrometer, including increased speed of acquisition and sensitivity, have resulted in significant improvement in the identification of peptides and proteins, including an increase in identification of lower intensity precursors, when compared to existing state-of-the-art technologies.”

Faster scan rates reduce cycle times by up to half, doubling the number of proteins and peptides identified.

The fragmentation techniques available on the LTQ Velos also enable more confident assignment of sequences and identification of post-translational modifications.

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Reference

Ray Scott wins Desty Award

Ray Scott will present the 14th Desty Memorial Lecture for Innovation in Separation Science, after he receives the Desty Award in honor of his lifetime achievements on 7 October 2008 at the Royal Institution of Great Britain in London.

Presentation of the Desty Award follows five lectures [1–5] and presentation of the inaugural award of The Knox Medal, which the Separation Science Group of the Royal Society of Chemistry commissioned in honor of last year’s Desty Award winner, Professor John Knox.

While Head of the Physical Chemistry Department at Unilever from 1962 onwards, Scott developed his theories about chromatographic separations, based on careful experimental work because, as he stated: “the truth of any theory is found by careful experimental work at the bench and it is how these results are interpreted which is important.”

From 1980, he was Director of the Applied Research Department at Perkin Elmer, where he continued to develop his theories with co-workers.

For his work on chromatography, Scott received many awards: The American Chemical Society Award in Chromatography (1990); The M.S. Tswett Chromatography Medal (1978); The Tswett Medal of the (former) USSR (1979); The A.J.P. Morton Chromatography Award (1982); and, The Royal Society of Chemistry Award in Analysis and Instrumentation (1988).

Attendance at the Desty Memorial Lecture is free of charge, but anyone wishing to attend should contact Professor Peter Myers in advance.

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Lectures