2020 AOCS Annual Meeting & Expo Analytical Abstracts

June 29 to July 3, 2020

Hosted online by the American Oil Chemists' Society (AOCS)

For more information, please visit https://annualmeeting.aocs.org.

Presentations dated Friday, January 1, 2021, were provided on-demand.

Analytical

Monday, June 29, 2020
Session Time: 8:25 AM - 10:30 AM
Presentation Time: 8:25 AM - 8:30 AM
Track: Analytical
Introduction: Proposed Updates to AOCS Official Methods

Co-Chair: Susan Seegers, MS - Bunge
Co-Chair: Scott Bloomer, PhD - American Oil Chemists' Society

This session will provide an overview of the proposed updates and upcoming release of additions and revisions to the *Official Methods and Recommended Practices of the AOCS.*

Monday, June 29, 2020
Session Time: 8:25 AM - 10:30 AM
Presentation Time: 9:40 AM - 9:40 AM
Track: Analytical
(3960) An international collaborative study on the proposed AOCS Method Ba 12a-20 for measuring trypsin inhibitor activity

Presenting Author: Keshun Liu, PhD - U.S. Department of Agriculture

Trypsin inhibitors (TI) are naturally present in grains of oilseed, legume and cereal crops. These proteinaceous compounds have nutritional and health implications to humans and animals. The American Oil Chemists Society (AOCS) approved an official Method Ba 12-75 in 1975 for measuring TI in soy products, based on Kakade et al. 1974 (Cereal Chem. 51:376-382). The method was reapproved in 2017. For the past three quarters, AOCS has undertaken a collaborative study on a proposed AOCS method, Ba 12a-20, which is a significantly modification of the current method, based on two recent publications (Liu, 2019, J. Am. Oil Chem. Soc., 96: 619–633; 635–645). The new method features the enzyme-last sequence, a single sample extract in duplicate, and 5 mL total assay (half of the current method), with an

expanded sample scope to include soybeans, pulses, beans, grains and related products. For the study, 15 collaborative labs from North America, South America, Europe, and Asia were recruited to analyze TI activities in 11 coded samples provided. This presentation reports the results of this collaborative study, while a manuscript is being written for publication in J. Am. Oil Chem. Soc. As increasing volumes of plant proteins are being used for food or feed in recent years, it is increasingly important to have a standard method that can measure TI in various protein products with high sensitivity and precision.

Monday, June 29, 2020
Session Time: 8:25 AM - 10:30 AM

Presentation Time: 10:05 AM - 10:05 AM

Track: Analytical

(4060) AOCS Method updates

Presenting Author: Scott Bloomer, PhD - American Oil Chemists' Society

New AOCS methods under development will be briefly presented. In addition, AOCS recently established a Memorandum of Understanding with the Japan Oil Chemists' Society (JOCS), which resulted in the adoption of three JOCS Methods by AOCS. Finally, the 2020 Additions and Revisions to the 7th Edition of AOCS Methods will be presented.

Monday, June 29, 2020
Session Time: 10:40 AM - 12:25 PM

Presentation Time: 10:40 AM - 10:45 AM

Track: Analytical

Introduction: Trace Contaminants

Co-Chair: Jessica K. Beekman, PhD - US Food & Drug Admin
Co-Chair: Jan Kuhlmann, PhD - SGS Germany GmbH

Monday, June 29, 2020
Session Time: 10:40 AM - 12:25 PM

Presentation Time: 10:45 AM - 10:45 AM

Track: Analytical

(3936) Chloroparaffins – highly complex process contaminants of particular concerns

Presenting Author: Walter Vetter, Dr rer nat - University of Hohenheim

Objective. Chloroparaffins are a class of polyhalogenated compounds which share the adverse environmental properties with polychlorinated biphenyls (PCBs) and other persistent organic

pollutants (POPs). Chloroparaffins are currently produced at more than 1.3 million metric tons per years which is equal with the total production volume of polychlorinated biphenyls (PCBs) from 1929 until their global ban. Chloroparaffins are mainly used as coolants and lubricants in cutting fluids, as well as plasticizers and flame retardants in polymers or sealant materials. Throughout the world, chloroparaffins were found to be high-concentrated in recent human milk samples, topped only by DDT. Typically, humans are not primarily exposed to chloroparaffins by consumption of raw food. Rather, they enter the products during industrial food processing or in homes. Fats and oil belong to the main food group contributing to dietary exposure with chloroparaffins. Methods used. Chloroparaffins were co-extracted with lipids, liberated from the lipid matrix and quantified by GC/ECNI-MS-SIM. Results. Chloroparaffins were particularly high concentrated in kitchen equipment from which they can enter food by leaching from the products in which they were used. Spiking experiments at low doses confirmed that exposure by carry-over effects could be higher than by food. Particularly high chloroparaffin concentrations were detected in palm oil-based vitamin E dietary supplements. Conclusions. Chloroparaffins are abundant and widespread contaminants. Indirect contamination during food processing and food preparation play a major role in human exposure, with oils being particularly affected. There is an urgent need to revise processing steps and equipment for possible leaching of chloroparaffins.

Monday, June 29, 2020
Session Time: 10:40 AM - 12:25 PM

Presentation Time: 11:10 AM - 11:35 AM

Track: Analytical

(4068) Analysis and Updated Occurrence of MCPD and Glycidyl Esters in Infant Formulas and Processed Foods

Presenting Author: Jessica K. Beekman, PhD - US Food & Drug Admin

3-monochloro-1,2-propanediol (3-MCPD) esters, 2-monochloro-1,3-propanediol (2-MCPD) esters, and glycidyl esters are chemical contaminants that are present in refined edible vegetable oils. These contaminants, which form as a result of the high temperatures required for the deodorization step of the refining process, are considered potentially carcinogenic and/or genotoxic. Therefore, their presence in refined oils and foods containing refined oils, particularly infant formula, poses potential health concerns. Numerous research studies over the last several years have focused on the development of methods and the collection of occurrence data for MCPD and glycidyl esters in complex food matrices (including infant formula) in an effort to estimate levels of exposure. In addition, recent EU regulations for bound glycidol with EU regulations for bound 3-MCPD likely imminent, as well, highlight the need for robust analytical methodologies and accurate, up-to-date occurrence data. Over the last 8 years, researchers at the U.S. Food and Drug Administration (FDA) have developed direct methodologies for the analysis of MCPD and glycidyl esters in edible oils, infant formulas, and other food products containing refined oils. This presentation will detail the results of several occurrence studies encompassing over 300 infant formula products (from the U.S. and Europe) and over 100 food products containing refined oils (from the U.S.) purchased between 2013 and 2019. 3-MCPD and glycidyl

ester concentrations in infant formulas varied widely among different U.S. manufacturers, but were generally similar among the European formulas analyzed. In addition, average contaminant concentrations in various food products were generally lower than the EU's active or proposed regulations.

Monday, June 29, 2020
Session Time: 10:40 AM - 12:25 PM

Presentation Time: 11:35 AM - 12:00 PM

Track: Analytical

(4082) Standardization of a High Throughput Method for the Quantification of MCPDe and GE in edible oils and fats

Presenting Author: Ralph P. Zwagerman, ing - Bunge Loders Croklaan

In recent years the necessity for routine analysis of glycidyl esters (GE) and 3-monocholoropropanediol esters (3-MCPDe) in edible oils intended as a food ingredient has become increasingly relevant. As existing legislation within the EU on the maximum levels of GE in edible oils is anticipated to be supplemented with maximum levels for 3-MCPDe in 2021, food producers are looking for fast and cost-effective ways to implemented the quantitation of these process contaminants in their products. As a result, we published an automated procedure that effectively addressed the needs of Quality Control (QC) laboratories within edible oil production facilities. Recently, further optimizations improved the robustness and sample throughput even further. Both the American Oil Chemists' Society (AOCS) and International Organization for Standardization (ISO) committee for Animal and Vegetable Oils (ISO TC34/SC11) unanimously approved to develop the method as a new standard. Hence, a collaborative study was set-up to provide the statistical data for standardization for which the round robin was completed in December 2019 while the second part of the study has been scheduled to start in early 2020. An update on the standardization project is presented including collaborative study data collected thus far.

Monday, June 29, 2020
Session Time: 10:40 AM - 12:25 PM

Presentation Time: 12:00 PM - 12:00 PM

Track: Analytical

(4185) Towards a Fully Integrated LC-GC×GC-Tofms/FID Platform to Unravel the MOSH&MOAH Hump in Food

Presenting Author: Giorgia Purcaro, PhD - University of Liège

Mineral oil hydrocarbons (MOHs) are a very complex mixture of isomers mainly associated with two classes of compounds, namely MOSH (composed by linear, branched, and alkyl-substituted cyclo-alkanes) and MOAH (which includes mainly alkyl-substituted (poly)aromatic

hydrocarbons at a different number of fused rings). MOHs can contaminate foods through different sources. The analysis of such a contaminant in food is a challenging task, mainly due to the high complexity of the matrices and the high affinity with the lipid fraction and many of its components. The method of election for the quantitative determination of MOSH&MOAH is LC-GC-FID, but the chromatographic profile obtained is a hump of unresolved substances. GC-MS alone fail to work as a confirmatory method. On this scenario, GC×GC-ToFMS/FID is the most promising solution to characterize the MOSH and MOAH fraction in details and to detect markers that can support corrective action towards the identification of the source of contamination. Moreover, work is in progress to validate a fully integrated LC-GC×GC-ToFMS/FID platform for merge both routine and confirmatory analyses.

Tuesday, June 30, 2020
Session Time: 8:25 AM - 10:10 AM

Presentation Time: 8:25 AM - 8:30 AM

Track: Analytical

Introduction: Rapid, Spectroscopic and Spectrometric Methods

Co-Chair: Magdi Mossoba - US Food and Drug Administration
Co-Chair: Herdis Adams, Archer Daniels Midland Co., USA

The use of traditional targeted quantitative methodologies, particularly chromatographic methods, is oftentimes consuming and inappropriate for the high-throughput screening needed today in many areas such as authentication or regulatory verification of quality and purity of fat and oil matrices. Although a targeted analytical method can detect a given analyte with high sensitivity and specificity, unknown compounds, such as contaminants or intentional adulterants, cannot be detected in this manner. This session addresses the urgent demand for rapid, untargeted screening analytical tools, particularly those based on spectral methods in conjunction with chemometrics.

Tuesday, June 30, 2020
Session Time: 8:25 AM - 10:10 AM

Presentation Time: 8:30 AM - 8:55 AM

Track: Analytical

(4184) Multidimensional 1H NMR Relaxation Morphological and Chemical and Morphological Sensor for Monitoring PUFA Oxidation

Presenting Author: Zeev Wiesman - Ben Gurion University of the Negev

Polyunsaturated fatty acids (PUFA) are components in many commercial products such as edible oils, foods, cosmetics, medication and in biological systems such as phospholipids of cellular membranes. Though PUFA aggregates are important functional components, they are also related to system degradation, since PUFAs are susceptible to oxidation via their multiple double

bonds and allylic carbons. Current technologies are not effective in characterizing the morphological and chemical structural domains of saturated, mono-unsaturated (MUFA) and PUFAs materials, or how the morphological structures of fatty acids, on the meso, nano and molecular levels, affect their oxidation mechanisms. In present study, the 1H Low Field (LF) NMR energy time relaxation sensorial technology is proposed as a tool to analyze PUFA-rich oils undergoing thermal oxidation. This technology generates 1D and 2D/3D chemical and morphological spectra using a primal dual interior method for the convex objectives optimization (PDCO) solver for computational processing of the energy relaxation time signals T1 (spin-lattice) and T2 (spin-spin). The 2D/3D graphical maps of T1 vs T2 generated for butter, rapeseed oil, soybean oil, linseed oil and pomegranate seed oil show that the different degrees of unsaturation of fatty acid oils affect their chemical and morphological domains, which influences their oxidative propensity. The technology of 1H LF-NMR energy relaxation time proved to be an effective non-destructive accurate and rapid sensorial tool to characterize and monitor PUFA oxidation.

Tuesday, June 30, 2020
Session Time: 8:25 AM - 11:45 AM

Presentation Time: 8:30 AM - 8:55 AM

Track: Analytical

(4166) Canola Oil Evolves to Meet Consumer Trends

Presenting Author: Lorin R. Debonte - Global Edible Oil Solutions, Cargill Inc

Consumer trends, health and regulatory requirements, and sustainability influence the development of new oil products. For the last 30 years canola/rapeseed oils have been at the intersection of these trends with new oil developments. Pairing the advances in crop breeding technologies with the Brassica species diversity we have accelerated the canola oil evolution into commercial products. During the 1980's the need to limit partially hydrogenated oils (PHO) in the our diets due to the high saturates and trans-fat levels become apparent. A new canola oil with shelf stability, frying performance, nutritional balance, and taste was required to replace PHOs. In response we created three commercial products having <3.0% linolenic with oleic levels of 65%, 75%, 85%. Our plant breeding team released high oleic canola hybrids for production on a million acres aligning with the 2005 FDA requirement for trans-fat labeling. This forever changed the industry making high oleic canola oil the backbone for stability in frying and shelf life around the globe. A new trend is forming with medical and health organizations recommending a diet low in saturated fat. Consumers are responding by making healthier choices in the foods they eat. To meet this growing health demand we have commercially released a low saturate high oleic canola oil with <4.5% saturates delivering the stability and taste performance of high oleic oils. Our next nutrition step change is underway with a <3.5% saturate oil.

Wednesday, July 1, 2020

Session Time: 9:45 AM - 12:35 PM

Presentation Time: 10:55 AM - 10:55 AM

Track: Analytical

(3985) Simultaneous analysis of various triacylglycerol isomers by supercritical fluid chromatography

Presenting Author: Koji Masuda - The Nisshin OilliO Group, Ltd.

Triacylglycerol (TAG) isomers have been reported to function differently in terms of physical and nutritional properties. Therefore, the analysis of TAG isomers in edible oils and biological samples is essential to control the physical properties and obtain detailed knowledge concerning their digestion and absorption. However, analytical methods for evaluating TAG isomers are still under development. At the 2019 AOCS Annual Meeting & Expo, we reported the practical analysis of TAG regioisomers and enantiomers comprising two oleic acids and one palmitic acid. In this study, we aimed the analytical method that reported previously was applied to various TAG isomers to verify its versatility. In this study, we utilized supercritical fluid chromatograph-tandem quadrupole mass spectrometer (SFC-MS/MS) equipped with a chiral column and acetonitrile and methanol were used as modifiers. The results confirmed the separation of TAGs comprising two palmitic acids and one oleic acid as well as those comprising two stearic acids and one oleic acid (sn-POP, sn-PPO, sn-OPP, sn-SOS, sn-SSO, and sn-OSS). Furthermore, standards of TAG enantiomers comprising three fatty acids, i.e., palmitic, stearic, and oleic acid (sn-SPO, sn-OPS, sn-PSO and sn-OSP) were successfully separated. Moreover, we were able to simultaneously quantify various TAG isomers in edible oils. In conclusion, we applied analytical methods that previously reported to various TAG regioisomers and enantiomers and simultaneously quantify these isomers in edible oils. Employing this approach will make it possible to grasp the detailed TAG composition, which will lead to improve controlling the physical properties and development of functional structured oils and fats.

Wednesday, July 1, 2020
Session Time: 9:45 AM - 12:35 PM

Presentation Time: 12:10 PM - 12:10 PM

Track: Analytical

(4135) Resolution of isobaric oxo-glycerophospholipids by normal phase LC/MS and differential hydrolysis with group V and group X sPLA2s

Presenting Author: Arnis Kuksis - University of Toronto

LC/MS provides an effective resolution and identification of intact molecular species of natural glycerophospholipids, especially when combined with reversed phase HPLC and MS/MS. The oxo-glycerophospholipids (hydroxides, epoxides, isoprostanes and others) when present, yield mixtures of lipids, where difficulties arise due to chemical instability and presence of isobaric components with similar chromatographic and mass spectrometric properties. The above problems are further complicated, when isobaric ether glycerophospholipids are present. We had

previously observed that group V sPLA2 preferentially hydrolyses the oligoenoic species of GroPChos, while group X sPLA2 favors the hydrolysis of the polyunsaturated GroPCho species, both failing to hydrolyze the plasmanyl and plasmenyl cholines, which accumulate in the hydrolysis residue. Using human HDL3 as example, we now demonstrate that both group V and group X sPLA2s may be used to completely destroy the diacyl GroPChos and their oxo derivatives. The plasmanyl and plasmenyl cholines and their oxo-derivatives left in the digestion residue can then be resolved and quantified by normal phase LC/MS without interference from overlapping isobaric diacyl GroPChos and their oxo derivatives. The new method allows to search the ether glycerophospholipids for presence of epoxy, hydroxy, hydroperoxy and isoprostane derivatives, which have not been previously determined.

Thursday, July 2, 2020
Session Time: 8:25 AM - 9:45 AM

Presentation Time: 9:20 AM - 9:45 AM

Track: Analytical

(3580) HS-GC-IMS: A Screening Method Discriminating Quality Grades in Virgin Olive Oils by Specific Volatile Compounds

Presenting Author: Enrico Casadei - Alma Mater Studiorum - Università di Bologna

Sensory evaluation, carried out by Panel test, is essential for the quality classification of virgin olive oils (VOOs). The presence and perceived intensity of fruity attribute and sensory defects are linked with the occurrence of specific volatile compounds. Instrumental screening methods based on analysis of volatiles can support the Panel test through fast pre-classification of samples with a known probability, thus increasing the efficiency of quality control. A Headspace Gas Chromatography Ion Mobility Spectrometer (HS-GC-IMS) was used to analyze 198 commercial VOOs by a semi-targeted approach. PLS-DA models were built by data matrices composed of 15 selected volatiles. The performance (intra-day and inter-day repeatability, linearity) of the method was evaluated with satisfactory results. Percentages higher than 75% and 73% of correctly classified samples were achieved in cross and external validation, respectively. Quantification of the 15 volatiles highlighted that some of these compounds can be used as headspace key markers to determine VOO quality grades. The analytical approach presented herein is a promising, relatively easy-to-use and rapid screening tool to support the Panel Test for the discrimination of VOOs according to their quality grade, both for industries and official control laboratories. This work is developed in the context of the project OLEUM "Advanced solutions for assuring authenticity and quality of olive oil at global scale", funded by the European Commission within the Horizon 2020 Programme (GA no. 635690). The information expressed in this abstract reflects the authors' views; the EC is not liable for the information contained therein.

Friday, January 1, 2021
Session Time: 1:00 AM - 2:00 AM

Presentation Time: 1:00 AM - 2:00 AM

Track: Analytical

(3633) A Non-targeted LC-qToF-MS based Approach to Verify the Geographical Origin of Virgin Olive Oil

Presenting Author: Ina Willenberg - Max Rubner-Insitut

Extra virgin olive oil is one of the most counterfeited foods within the European Union. Possible types of fraud include application of unpermitted treatment and incorrect classification of the oil but also false labelling of the geographical origin. The motivation for misleading declaration of the origin is based on the fact that consumers prefers individual countries of origin, resulting in higher prices for corresponding origins. However, the verification of the geographical origin based on analytical techniques is still a challenge. The aim of the presented work was to develop and optimize a non-targeted HPLC-ESI-qToF-MS/MS approach which enables the verification of the origin of virgin olive oils from Italy, Spain, Greece and Portugal. For this purpose, the polar extract of a set of 95 oils with known origin was prepared based on liquid/liquid extraction. Subsequently, the samples were analyzed by HPLC-ESI-qToF-MS/MS. After peak detection and bucket table generation the resulting features were stepwise reduced in order to identify the most predictive features, e.g. based on ANOVA with Tukey post-hoc-test. Finally, 68 features were selected and used for building a classification model based on linear discriminant analysis (LDA). Test set validation of the model revealed a correctness of prediction of more than 80 % for oils from Spain, Greece and Italy within the training set (n=75). Portuguese samples showed a slightly lower value of 67%. Within the independent test set (n=25) all samples were classified correctly which demonstrated the possible usefulness of the approach in the verification of origin of olive oils.

Friday, January 1, 2021
Session Time: 1:00 AM - 2:00 AM

Presentation Time: 1:00 AM - 2:00 AM

Track: Analytical

(4030) Deducing structures of unusual fatty acid methyl esters with high sensitivity without chemical standards

Presenting Author: Tom Brenna, PhD - University of Texas at Austin

The vast quantity of fatty acids produced and consumed have straight chains, and double bonds are of cis (Z) geometry, and when polyunsaturated, are methylene-interrupted. FAME have superb analytical properties on high resolution capillary GC columns. Mass spectrometry (MS) is the most sensitive method for fatty acid analysis. Electron ionization (EI) of FAME causes rearrangement of double bonds and is unsuitable for deducing unknown structures. Specialized esters provide structural detail, however methods to characterize FAME directly are preferred.

The revolution in MS techniques has resulted in refinement of physical methods, particularly high mass resolution and exact mass measurement, that excel at characterizing ions that include heteroatoms, however they do not distinguish isomers. That problem remains one requiring fragmentation possibly after selective derivatization, both fundamentally chemical processes. We developed a gas phase ion-molecule reaction termed covalent adduct chemical ionization (CACI) which results in derivatives that, upon collisional activation, yield fragments that identify double bond structure and in some cases, geometry. Separately, we realized that collisional dissociation of EI generated molecular ions yields very different mass spectra than MS-1 with, for instance, no McLafferty rearrangement ion. Instead, strong ions characteristic of chain branching in saturated FAME are observed. Recent advances in these methods are extension to modern GC/MS/MS instruments via solvent-mediated (SM) CI on Shimadzu instruments, as well as combining the power of these approaches using low energy CID. We demonstrate the power of these methods to characterize minor PUFA FAME with subtle differences in double bond position.

Friday, January 1, 2021
Session Time: 1:00 AM - 2:00 AM

Presentation Time: 1:00 AM - 2:00 AM

Track: Analytical

(4169) In Depth Analysis of Fats and Oils Applying Multidimensional Chromatographic Techniques

Presenting Author: Pierluigi Delmonte - US Food and Drug Administration

In contrast to other fields of scientific research that greatly benefit from modern analytical tools such as high-resolution mass spectrometry and ultra-high-performance liquid chromatography, lipid chemistry is largely dominated by traditional techniques. Despite this resistance to innovation, recent developments in multidimensional separation techniques are finally leading lipid chemistry into a new era. Two-dimensional gas chromatography (GCxGC), and the variant GCxGC with online reduction (GC-ORxGC), achieved by adding a capillary reducer between the two separation columns, provides the capacity to separate almost all fatty acids (FA) contained in the most challenging lipid samples and provides identification of analytes based on the interpretation of their elution patterns. The combination of liquid and gas chromatography simplifies the characterization of highly poly-unsaturated samples, such as marine oils, particularly when silver ion HPLC is used for the first dimension of separation. In this study, liquid and gas phase separations are combined in a GCxGC and LC-GC scheme to assess the composition of seed oils, ruminant fats and marine oils. In addition, the chemical structure of fatty acids is determined by interpreting the mass spectra of 4,4-Dimethyloxazoline (DMOX) derivatives acquired by GCxGC-MS. The innovative combination of legacy separations and chemical reactions can provide the multidimensional separations capable of solving the most challenging lipid chemistry problems.

Friday, January 1, 2021

Session Time: 1:00 AM - 2:00 AM

Presentation Time: 1:00 AM - 2:00 AM

Track: Analytical

(3991) Application of Vacuum UV Detection to the gas chromatographic analysis of fatty acids methyl esters.

Presenting Author: Pierluigi Delmonte - US Food and Drug Administration

Application of vacuum ultraviolet detection (VUV) to gas chromatography (GC) provides very valuable information for the analysis of fats and oils which is not provided by any other technique. The VUV spectrum of fatty acids (FA) at 125-145 nm is dominated by the absorbance of -C-C- bonds, and it may be used to achieve quantification based on theoretical response factors. The following segment of the spectrum, 150-200 nm, in which -C=C- bonds absorb, provides a unique fingerprint for each type of unsaturated fatty acid. VUV may distinguish between cis and trans mono-unsaturated fatty acids (FA) based on their different UV maximum absorbance, 185 versus 180 nm, respectively. VUV spectra of methylene-interrupted poly-unsaturated FAs are characterized by a more intense signal in the 150-220 nm region, proportional to the number of double bonds, and a shift in the maximum absorbance at slightly higher wavelengths. Two or more conjugated double bonds provide very distinct spectra above 200 nm, as previously exploited for the HPLC analysis of conjugated linoleic acid (CLA). In this study, VUV detection is combined with GC and comprehensive GCxGC to achieve to the identification and measurement of FA in fats and oils.

Friday, January 1, 2021

Session Time: 1:00 AM - 2:00 AM

Presentation Time: 1:00 AM - 2:00 AM

Track: Analytical

(3800) Rapid Analysis of Intact Canola Seeds using a Handheld NIR-spectrometer

Presenting Author: Veronique J. Barthet, PhD - Canadian Grain Commission

The NIR models for canola quality were developed with samples from Canadian canola seeds harvested in 2016 and 2017. All calibration models were first tested on a 2017 external validation sample set. The handheld NIR spectrometer used in this study has a limited wavelength range 908.1 nm to 1676.2, however, the validation results showed that it could be used to predict several important parameters that defined canola seed quality. Final testing was done using calibration models with the least number of factors on a second external canola validation sample set (2018 harvest). Some calibration models showed excellent stability and predictive powers with R2val values of 0.94 to 0.99 (i.e., oil, protein, oleic acid and iodine value) and low SEPs for both external validation sample sets. The α-linolenic acid model had an R2val of 0.93 when applied to the 2017 external validation set, the correlation fell slightly to 0.88 when applied to the 2018 external validation sample set, potentially indicating a slight instability in the

model. The prediction model for total glucosinolate was not very good, but still could be used to segregate the samples into low or high glucosinolate samples. Finally, the predictive models for chlorophyll and total saturates were unusable. The chlorophyll model was very unstable, likely due to the instrument's limited wavelength range.

Friday, January 1, 2021
Session Time: 1:00 AM - 2:00 AM

Presentation Time: 1:00 AM - 2:00 AM

Track: Analytical

(3655) A study on the characterisation of Baobab (*Adansonia digitata*) seed oil

Presenting Author: Sara B. Abdalkreem - International Islamic University Malaysia

The oil quality parameters of the seed oil of Baobab (Adansonia digitata) were evaluated using standard methods of analysis. The iodine value (Wiji's method), Peroxide value, Saponification value were 86 g/100g, 4.08 meq/Kg, 188 mg/g, respectively, for seed oil. Baobab seed is one of the significant additional sources of oil, due to the appreciated amount of oil extracted from baobab seed kernel. The oil extraction process using a Soxhlet extractor, extraction of the kernel was higher 23% compared to the hulls that contain 5.4% oil. The kernel oil contains substantial quantities of calcium, potassium, and magnesium, which were found to be 4116, 2339 and 1629 mg/Kg, respectively. The fatty acid composition analysed by gas-liquid chromatography, the result showed that oleic and linoleic were the primary unsaturated fatty acids, whereas palmitic was the major saturated acid. Baobab oil is rich of polyunsaturated fatty acid, were expected to be faster oxidation, which is not seen because the Baobab seed oil has high resistance to rancidity due to the presence of high antioxidant content, the oil showed a considerable amount of total phenolic content (TPC) and charitable antioxidant activity. Baobab oil has excellent nutritional and industrial potentials. It is, therefore recommended that more and advanced research should be undertaken for this abundant source of natural nutritious oil.

Friday, January 1, 2021
Session Time: 1:00 AM - 2:00 AM

Presentation Time: 1:00 AM - 2:00 AM

Track: Analytical

(4161) Comparison and Optimization of Extraction Procedures for Analysis of Isoflavones and Proteins from Soybeans

Presenting Author: Dave Luthria

Friday, January 1, 2021
Session Time: 1:00 AM - 2:00 AM

Presentation Time: 1:00 AM - 2:00 AM

Track: Analytical

(4159) Update on EU regulations concerning 3-MCPD and glycidol

Presenting Author: Jan Kuhlmann, PhD - SGS Germany GmbH

3-Monochloropropane-1,2-diol (3-MCPD) is known as food-borne contaminant since the late seventies. It is classified as possibly carcinogenic to humans (IARC, category 2B) a maximum level for soy sauce and hydrolyzed vegetable protein was set by EU regulation in 2001 correspondingly. Decades later, the occurrence of heat-induced fatty acid esters of 2-MCPD and 3-MCPD (2- and 3-MCPD-E) as well as glycidyl fatty acid esters (GE) was discovered in refined edible oils and oil- & fat-containing foods. Glycidol is considered to be genotoxic, probably carcinogenic to humans (IARC, category 2A). Animal studies showed that free 3-MCPD and glycidol are released to large extend from the ester-bound form during digestion in the gastrointestinal tract. The European Commission took several measures in terms of collecting data on occurrence and bioavailability of 3-MCPD and glycidol respectively the corresponding esters. Based on this data, a potential risk to some consumers was identified and a regulation on GE in vegetable oils as well as infant formula was set in force 2018. In 2019, amendments to the existing regulation on analytical performance criteria for certain contaminants were released. Finally, several draft regulations on 3-MCPD and 3-MCPD-E in edible oils and fats and infant formula were circulated. It is assumed that in the EU a partially complex regulation on free and ester-bound 3-MCPD and ester-bound glycidol in different foods will come into force in 2021. This presentation intends to give an update on existing and future regulation on 3-MCPD and glycidol in edible oils and fats and related foods.

Friday, January 1, 2021
Session Time: 1:00 AM - 2:00 AM

Presentation Time: 1:00 AM - 2:00 AM

Track: Analytical

(4230) Food-borne Toxicants: An Important Class of Compounds for Industry, Government and Consumers

Presenting Author: Michael Granvogl, PhD - University of Hohenheim

Monday, June 29, 2020
Session Time: 8:25 AM - 10:30 AM

Presentation Time: 8:30 AM - 9:15 AM

Track: Analytical@@@Biotechnology

(3974) ISO TC 34/SC 16 Horizontal Methods for Molecular Biomarker Analysis —International Standards for Molecular Biomarker Analysis/Isothermal Nucleic Acid Amplification Methods

Presenting Author: Michael Sussman, PhD - USDA, Agricultural Marketing Service

Harmonized, easy to handle methods of analysis with defined patterns and known nomenclatures bring more customers to the market. The International Organization for Standardization (ISO.org) was formed in 1946. It is an independent, non-governmental voluntary consensus standard body based in Geneva, Switzerland with a membership of 164 national standards bodies. The US ISO member is the American National Standards Institute (ANSI.org) a consortium of US standardization organizations. There are 44 participating countries. The US delegation responsible for developing the US position for standards development in agricultural molecular biomarker analysis was delegated to the American Oil Chemist's Society (AOCS.org) by ANSI. The AOCS US TAG also hosts the TC 34/SC 16 international secretariat. TC 34/SC 16 has published 21 standards with another 14 under development. SC 16 is working on a standard providing general criteria for development and validation of isothermal nucleic acid amplification analytical methods. Isothermal nucleic acid amplification-based methods are now being used to amplify DNA for detection, identification, quantification and analysis of biomarkers in food and food products. Isothermal nucleic acid amplification is a constant temperature polymerase mediated chain reaction. Some advantages are no need for precision thermocycling instruments, single assay printable paper chromatographic format permits for ease of use, in some cases use of unpurified cell extract as substrate and only a few preparation steps. The new ISO standard is applicable to food, feed, plant matrices, and their propagules, plant pathogens and animals and provides guidance for specific isothermal amplification technologies.

Tuesday, June 30, 2020
Session Time: 8:25 AM - 10:10 AM

Presentation Time: 9:20 AM - 9:20 AM

Track: Analytical@@@Biotechnology

(3900) Development of Immune-magnetic Nanoparticles for the Quantification of Aflatoxin B1 in Oilseeds

Presenting Author: Hongshun Yang, PhD - National University of Singapore

The objective of this research was to develop a rapid detection method based on nanoparticles to quantify the aflatoxin B1 (AFB1) in oilseeds. Liquid-solid extraction (LSE) combined with immune magnetic solid-phase extraction (IMSPE) via nanoparticles was developed for the determination of AFB1 in oilseeds. Self-synthesized antibody functionalized magnetic nanoparticles (MNPs) were firstly synthesized and characterized using FT-IR and TEM before being used to extract the AFB1 in oilseeds. Due to the highly selective extraction with the aid of antibody-antigen interaction, the MNPs captured the AFB1 in the samples highly efficiently. After the extraction process, the AFB1 was detected and quantified using HPLC-Fluorescence Detection. The extraction method was optimized based on several crucial factors which affect its

efficiency, namely, the low temperature clean-up duration, the volume of MNPs used in IMSPE and the vortex duration for IMSPE. Based on the results obtained, the optimum conditions were 20 h, 1 mL and 5 min, respectively. Subsequently, a calibration curve constructed with spiked samples of known concentrations yielded a correlation coefficient of 0.9946, showing strong linearity over a concentration range of 0.1-100 ng·g-1. The limits of detection and quantification of the method were estimated to be 0.02 and 0.05 ng·g-1, respectively. The proposed method also showed satisfactory repeatability, with intra and inter-day method recovery rates ranging from 94.92-118.95%. Lastly, real sample analysis was carried out on five oilseed samples. The proposed method was highly accurate, sensitive and reliable. The results show the promising in detecting AFB1 contamination in oilseeds rapidly.

Friday, January 1, 2021
Session Time: 1:00 AM - 2:00 AM

Presentation Time: 1:00 AM - 2:00 AM

Track: Analytical@@@Biotechnology

(3937) Electrochemical Detection of Cannabis Drugs in Human Saliva

Presenting Author: Margaret M. Renaud-Young, PhD - University of Calgary, Department of Chemistry

With concerns about safety related to individuals driving under the influence of cannabis drugs, there is an urgent need for sensitive and selective detection of Δ^9-tetrahydrocannabinol (THC) using a point-of-contact sensor. THC and other cannabinoids are electroactive compounds, making them excellent candidates for electrochemical detection, analogous to the techniques used in blood-alcohol sensors. We have recently demonstrated that we can reproducibly, quantitatively, and sensitively detect THC and its metabolites by aliquot-depositing the compounds onto carbon paper electrodes and performing electrochemical oxidation measurements in a pH 10 electrolyte solution. With this technique, THC and the metabolite 11-hydroxy-tetrahydrocannabinol could be detected at levels as low as 1 pmol of drug (~ 4 nM from a 0.25 mL saliva sample) with a detection efficiency of ~ 0.2 electrons/drug molecule. However, the non-psychoactive metabolite, 11-nor-9-carboxy-tetrahydrocannabinol, was found to be more difficult to detect, with a detection efficiency of only 0.03 electrons/drug molecule. To build upon this previous work, we have characterized the responses of THC, THC metabolites, and other relevant cannabis compounds in solutions of varying pH. Here we observe profound differences in the detection of these molecules, likely attributable to their different functional groups. Additionally, we have developed techniques to circumvent the problems of THC insolubility, enabling us to detect drug directly from aqueous saliva specimens. The results have been very promising, showing that we can obtain reproducible and sensitive detection of THC and its metabolites in artificial saliva, taking us one step closer to a realizable road-side THC sensor.

Friday, January 1, 2021

Session Time: 1:00 AM - 2:00 AM

Presentation Time: 1:00 AM - 2:00 AM

Track: Analytical@@@Biotechnology

(4126) A brief look into the world of cannabis testing in Canada

Presenting Author: John Coleman - Anandia

Into the second year of legalization, we are only scratching the surface of the cannabis plant's potential. This session will take a dive into the regulatory testing requirements for cannabis testing in Canada, and will aim to bring clarity to the benefits of a legal cannabis industry. Using comparisons from mature markets like California and Colorado, we will look into public perception of potency and the impact testing methods have on this. This session will also take a look at the history of cannabis testing in Canada, where that has brought us to today, and what the future may look like for the industry.

Tuesday, June 30, 2020

Session Time: 8:25 AM - 10:10 AM

Presentation Time: 9:45 AM - 9:45 AM

Track: Analytical@@@Health and Nutrition

(4072) Miniaturization of Optical Sensors and their Potential for High-Throughput Screening of Foods

Presenting Author: Luis E. Rodriguez-Saona, PhD - The Ohio State University

Molecular fingerprinting technology has evolved from bulky laboratory benchtop instrumentation to field deployable devices driven by advances in semiconductor and photonic technologies. Ongoing miniaturization of vibrational spectroscopy equipment has revolutionized the food industry by allowing on-site and real-time monitoring of food products and production processes to ensure quality and safety. By producing a characteristic chemical 'fingerprint' with unique signature profiles, miniaturized molecular spectroscopy techniques combined with chemometric analysis have positioned as viable "green" alternatives for field applications allowing phenotyping, quality assurance, authentication, and detection of adulteration. We will present targeted and non-targeted applications for monitoring quality traits and authenticating oils using vibrational spectroscopy signatures combined with chemometrics. Oils were characterized by their fatty acid profile, free fatty acids (FFA), peroxide value (PV), and total polar compounds (TPC). Pattern recognition and partial least squares regression analysis of spectra allowed to identified oil adulteration and excellent correlations ($Rval \geq 0.9$) with reference tests and standard errors of prediction that would allow for quality control applications.

Wednesday, July 1, 2020

Session Time: 9:45 AM - 12:35 PM

Presentation Time: 9:45 AM - 10:10 AM

Track: Analytical@@@Health and Nutrition

(3993) Searching a Comprehensive High Resolution Tandem Mass Spectral Library for Reliable Human Metabolite Identification

Presenting Author: Xiaoyu Yang, PhD - NIST

Liquid chromatography tandem mass spectrometry is becoming a routine and powerful technique for compound identification in lipidomics and metabolomics studies. However, identifying compounds from millions of mass spectra is critical for data analysis. We built and extended a comprehensive and high quality reference tandem mass spectral library and applied it to fast and accurate metabolite identification in human samples. For building the library, tandem mass spectra of each authentic compound were acquired with Orbitrap Elite and Fusion Lumos instruments (IT, FT-IT, and HCD) at different collision energies. A consensus spectrum was generated from multiple spectra at the same instrument condition for the library. The product ions in each spectrum were annotated with MS_Sing and MS Interpreter software programs (free download at chemdata.nist.gov) and validated by manual inspection. Currently, the library contains >1 million spectra from >25,000 compounds. Of these, ~5,000 are human metabolites and ~1,000 are lipids including phospholipids, glycolipids, steroids and related compounds. Human plasma, urine, and milk samples were analyzed on reverse phase LC/MS/MS (Orbitrap Fusion Lumos) in positive and negative modes respectively. Metabolites were identified by searching the library with MS Search program. Over 700 human metabolites were identified with positive and negative precursor ions (e.g. $[M+H]+$, $[M+Na]+$, $[M+2H]2+$, $[M-H]-$). The ions produced in-source during the ionization process from the metabolites by various neutral losses (e.g. ammonia loss from tryptophan) were also identified in about 50% of the identified metabolites. These in-source fragments can help confirm the metabolite identification.

Wednesday, July 1, 2020

Session Time: 9:45 AM - 12:35 PM

Presentation Time: 10:30 AM - 10:30 AM

Track: Analytical@@@Health and Nutrition

(3938) New and rare vitamin E compounds – countercurrent chromatographic isolation followed by structure identification

Presenting Author: Walter Vetter, Dr rer nat - University of Hohenheim

Objective. Vitamin E is mainly composed of four tocopherols (α-, β-, γ- and δ-T) and four tocotrienols (α-, β-, γ- and δ-T3), i.e. tocochromanols. Scarcely, a few further minor compounds were described in the scientific literature. Here, we aimed to develop a method to screen for these minor tocochromanols, to enrich and/or isolate them, and to assign their exact structures by NMR and MS techniques. Methods used. High gram-amounts of edible oils were carefully

saponified (inert gas, addition of antioxidant) and the unsaponifiable matter was separated. Aliquots of ~0.5-1 g unsaponifiable matter or the content from vitamin E capsules were repeatedly fractioned by countercurrent chromatography. Structure-dependent elution volumes of α- to δ-tocopherols and tocotrienols were developed and used to predict the elution range of tocodienols and tocomonoenols, as well as possible co-eluting compounds. After purification, NMR and MS techniques were used for structure determination. Results. Four tocopherols, at least four tocomonoenols, four tocodienols and four tocotrienols were found to be native minor or trace constituents of plant oils. CCC proved to be well-suited for the detection and enrichment/isolation with high purity. Several of these tocochromanols were detected for the first time in the corresponding oil or at all. Structures and positions of double bonds were assigned by NMR and MS methods. Conclusions. Vitamin E minor compounds are more varied than previously anticipated. Their vitamin E activity is widely unknown, but the amounts that were gained will be sufficient to study their role in oils and food.

Wednesday, July 1, 2020
Session Time: 9:45 AM - 12:35 PM

Presentation Time: 11:20 AM - 11:45 AM

Track: Analytical@@@Health and Nutrition

(4069) Chiral separation of alpha-tocopherol in food products

Presenting Author: Jinchuan Yang - Waters

Vitamin E is an essential vitamin that functions as a chain-breaking antioxidant in the body by preventing the spread of free-radical reactions. For the nutritional purpose, vitamin E activity is defined as being limited to the 2R-stereoisomeric forms of alpha-tocopherol.(1) Depending on the stereoisomeric forms of alpha-tocopherol, or the source of the alpha-tocopherol, different conversion factors are used in calculating the vitamin E activities in foods and dietary supplements. (1, 2) So far, there is no established standard method for the determination of the stereoisomers of alpha-tocopherol, recordkeeping of the source of the alpha-tocopherol is required for the estimation of the vitamin E activity in foods or dietary supplement.(2) There is a need for an routine analytical method to verify the form of the alpha-tocopherol in foods and dietary supplements. Separation of the eight stereoisomers of alpha-tocopherol is a complicated process. Normal phase liquid chromatography has been partially successful in separating the stereoisomers using chiral columns. (3) Up to five peaks can be separated for a racemic-alpha-tocopherol, which could be used to verify the source of the alpha-tocopherol, since the natural source only has one stereoisomeric form, the RRR-alpha-tocopherol. The main drawback of the NPLC chiral separation is the long run time. Ultra performance convergence chromatography has been successful in the chiral separation. Much shorter run time is often achieved in UPCC for chiral separations. Here the application of the UPCC to the chiral separation of alpha-tocopherol has been exploited.

Wednesday, July 1, 2020

Session Time: 9:45 AM - 12:35 PM

Presentation Time: 11:45 AM - 12:10 PM

Track: Analytical@@@Health and Nutrition

(4107) Analysis of Fatty Acid Isomers in Human Plasma

Presenting Author: Heather C. Kuiper, PhD - CDC

Fatty acids (FA) serve many important roles in human health, acting as energy reserves, signaling molecules, membrane components, and more. FA are also associated with disease risk, with individual FA associated with increased risk of cardiovascular disease or shown to influence inflammatory diseases and type 2 diabetes. In order to better understand the role of individual FA in human health and disease, it is important to quantitatively assess these FA in biological samples. However, biological samples contain a large variety of FA, including many positional and geometric isomers, with over 65 different FA including trans-FA (TFA) being reported in humans. The large number of cis- and trans-isomers pose analytical challenges, because these isomers have the same molecular mass and very similar chemical structures. Herein we provide an overview of technologies used to assess fatty acid positional and geometric isomers. We combine the CDC isotope dilution-gas chromatography-negative chemical ionization-mass spectrometry (ID-GC-NCI-MS) method with Ag+ ion HPLC to separate and identify over 50 geometric FA isomers in human plasma. We also utilize mCPBA to epoxidize FA double-bonds and analyze the FA-epoxides via GC-MS/MS using multiple reaction monitoring to assess positional FA isomers in human plasma. Fragmenting the epoxides allows us to distinguish C16:1n-7t and C16:1n7c from 10 other C16:1 isomers present when using selected ion monitoring. These approaches allow us to assess both geometric and positional isomers in order to identify unknown FA of interest in blood.

Thursday, July 2, 2020

Session Time: 8:25 AM - 9:45 AM

Presentation Time: 8:55 AM - 9:20 AM

Track: Analytical@@@Health and Nutrition

(3930) ^{1}H NMR–metabolomics for PDO and PGI EVOOs Assessment

Presenting Author: Francesco Paolo Fanizzi, Chemistry Doctor - University of Salento

Being a premium price product, EVOO's authenticity and traceability are important for both consumer health and commercial purposes. In order to protect high-quality foodstuffs from a specific origin, Protected Designations of Origin (PDO), Protected Geographical Indication (PGI) and Traditional Specialty Guaranteed (TSG) were established at European level since 1992 and thereafter regulated [1]. These products meet strict production rules describing, for EVOOs, specific characteristics such as geographical production area, used cultivars, methods of cultivation and processing. Nevertheless, the EVOOs PDO and PGI assessment still remains essentially based on farmers declarations. Mediterranean countries are responsible for over 95%

of globally produced olive oil, including 105 European quality scheme registered from Italy (holding a quality record with over 40 among PDO and PGI EVOOs), Greece, Spain, France, Portugal, Croatia and Slovenia [2]. The aim of this work is to verify, following our studies on Italian PDO [3] and PGI [4] EVOOs, the possibility of assessing these classifications by using ^{1}H NMR metabolic profiles databases and MVA (multivariate analysis), beside farmers declarations. [1] Council Regulation (EC) 2081/92 and 510/2006. Regulation (EU) 1151/2012 and 665/2014. [2] Gonçalves Albuquerque, T.; Costa, H. S. and Oliveira, M. B. P. P. Eur. J. Lipid Sci. Technol. 2019, 121, 1800129. [3] Del Coco, L.; Mondelli, D.; Mezzapesa, G.N.; Miano, T.; De Pascali, S.A.; Girelli, C.R.; Fanizzi, F.P. J Am Oil Chem Soc, 2016, 93, 373-381. [4] Girelli, C.R.; Del Coco L.; Zelasco, S.; Salimonti, A.; Conforti, F.L.; Biagianti, A.; Barbini, D.; Fanizzi, F.P. Metabolites 2018, 8 (4), 60.

Thursday, July 2, 2020
Session Time: 12:10 PM - 1:00 PM

Presentation Time: 12:10 PM - 12:35 PM

Track: Analytical@@@Health and Nutrition

(4000) Identification of major gangliosides species in human milk and infant formula

Presenting Author: Francesca Giuffrida, PhD - Nestle Research Ctr

Gangliosides (GD) are glycosphingolipids, which play an important role in brain and intestinal immune development. GD are synthetized endogenously and found in food such as meat and dairy products; in infancy, GD are provided by human milk. Breastfed babies were found to have a higher GD content in their frontal cortices than formula fed babies, thought to support neuronal function as for example myelination, synaptogenesis and memory formation. GD in breastmilk are also thought to influence the gut microbiota and interfere with pathogen binding in the intestine. The enrichment of infant formula with alpha-lactalbumin, provides a source of GD, due to a specific manufacturing process, with level similar to those of human milk, however, little is known about GD chemical similarity. In this study, we aimed at identifying the most abundant GD species in human milk and infant formula enriched with alpha-lactalbumin. High resolution mass spectrometry was used to elucidate the fragmentation pathway of GD allowing the identification of GD species. In both, human milk and infant formula, GD3 and GM3 were the most abundant GD classes and similar species were identified in the analyzed matrices.

Friday, January 1, 2021
Session Time: 1:00 AM - 2:00 AM

Presentation Time: 1:00 AM - 2:00 AM

Track: Analytical@@@Health and Nutrition

(3924) Beyond 2D-LC: Three Dimensions of Chromatography with Quadruple Parallel Mass Spectrometry, LC3MS4, for Formula and Milk Analysis

Presenting Author: William C. Byrdwell, PhD - USDA ARS BHNRC MAFCL

Two-dimensional liquid chromatography (2D-LC) is becoming less exotic and more mainstream as commercially available instruments proliferate. Despite the impressive capabilities of 2D-LC, there are times when even 2D-LC is not sufficient for an adequate separation. One-dimensional reversed-phase HPLC is not adequate to completely resolve all milk (or infant formula) triacylglycerol (TAG) molecular species, which are highly complex, and contain many isomers of TAGs, especially TAGs with short-chain fatty acids. Even conventional 2D-LC is not completely satisfactory. A new approach was developed, in which two second dimensions were used simultaneously for dual parallel comprehensive 2D-LC with quadruple parallel (x4) mass spectrometry. A pair of contact-closure (CC) controlled UHPLC switching valves were coupled to a timed CC circuit to allow the second ^{2}D separation, ^{2}D(2). The first second dimension, ^{2}D(1), used the commercially available system, in which conventional 2D-LC improved the separation of components, and all components eluted in the first modulation period from a 50 mm C30 column. The ^{2}D(2), which employed a 100 mm C30 column, produced an even better separation of components, but required more than one modulation period to elute, resulting in multi-cycle chromatography or "controlled wraparound" chromatography, analogous to twin column recycling chromatography. Electrospray ionization (ESI) mass spectrometry (MS) was used in parallel with atmospheric pressure photoionization (APPI) MS for LC1MS2 to monitor the first dimension RP-HPLC, while ESI-MS monitored the 50 mm C30 UHPLC (LC1MS1) and ESI-MS also monitored UHPLC using the 100 mm C30 column (LC1MS1), to produce LC1MS2 x (LC1MS1 + LC1MS1) = LC3MS4.

Friday, January 1, 2021
Session Time: 1:00 AM - 2:00 AM

Presentation Time: 1:00 AM - 2:00 AM

Track: Analytical@@@Health and Nutrition

(3964) Development of Cannabis and Hemp Certified Reference Materials to Support Cannabinoid Testing

Presenting Author: Jeremy Melanson - National Research Council Canada

A growing list of countries have thriving medicinal cannabis programs and countries such as Canada have legalized cannabis for recreational purposes. Most regulated markets have stringent testing requirements to ensure safety of consumers. Reports of high variability in Δ^9-tetrahydrocannabinol (THC) results between cannabis testing laboratories have highlighted the need for standards in cannabis testing. The National Research Council Canada is addressing this challenge through the promotion of documentary standards for cannabis testing methods and the development of cannabis certified reference materials (CRMs). These standards will ensure accuracy and consistency of testing results, assist licensed cannabis producers in achieving regulatory requirements, and ultimately promote confidence in regulated cannabis industries. The cannabis flower has a unique morphology with the majority of THC contained within epidermal outgrowths known as trichomes. These trichomes are fragile and can be dislodged from the plant surface during processing and tend to aggregate. This creates THC "hot-spots" that hinders CRM

homogeneity. Poor stability of cannabinoids is also well-known, especially the conversion of acid to neutral forms, so this also creates challenges for reference material production. This presentation will highlight recent developments and challenges faced in the production and certification of a cannabis CRM, and the development of a hemp CRM will also be discussed. The availability of cannabis and hemp CRMs will facilitate method validation for cannabis testing laboratories and allow laboratories to assess their entire method, from extraction to instrument analysis.

Friday, January 1, 2021
Session Time: 1:00 AM - 2:00 AM

Presentation Time: 1:00 AM - 2:00 AM

Track: Analytical@@@Health and Nutrition

(4084) Two- and Three-Dimensional Liquid Chromatography with Quadruple (x4) Parallel Mass Spectrometry for Triacylglycerol Analysis

Presenting Author: William C. Byrdwell, PhD - USDA ARS BHNRC MAFCL

The compositions of triacylglycerols (TAGs) in natural seed oils and biological samples such as cow's milk can be exceedingly complex. One-dimensional HPLC with mass spectrometry (MS) detection is often not sufficient to provide complete resolution of isomers or TAGs having the same equivalent carbon number (ECN), and regioisomers are usually not separated. Recent developments in commercially available two-dimensional liquid chromatography (2D-LC) systems have opened new opportunities for resolution of previously inseparable molecular species. We have used orthogonal separation techniques, specifically non-aqueous reversed-phase (NARP) HPLC, which separates TAGs by ECN, coupled with silver-ion UHPLC, which separates TAGs by degree of unsaturation and its locations in the molecules, allowing physical separation of regioisomers. In such cases, two mass spectrometers with complementary ionization techniques monitored the first dimension and two more mass spectrometers monitored the second dimension for LC1MS2 x LC1MS2 = LC2MS4. For the most complex samples, which contain both short-chain and long-chain TAGs, we employ three dimensions of separation, in which the first dimension partially resolves molecular species, and two parallel second dimensions allow greater separation, for LC1MS2 x (LC1MS1 + LC1MS1) = LC3MS4. The first second dimension separates short-chain and long-chain TAGs very well, and the second (parallel) second dimension employs multi-cycle, or "controlled wraparound", chromatography to separate otherwise unresolved ECN isomers. These experiments not only show the possibilities and benefits of three-dimensional chromatography, but also show the great advantage of multi-cycle chromatography to improve resolution and optimally fill the separation space available in a second dimension.

Friday, January 1, 2021

Session Time: 1:00 AM - 2:00 AM

Presentation Time: 1:00 AM - 2:00 AM

Track: Analytical@@@Industrial Oil Products

(3850) Univariate Vs. Multivariate Analysis of Moisture in Olive Oil: Ft-nir Spectroscopic Calibration Based on the Karl-fischer Primary Reference Method

Presenting Author: Sanjeewa R. Karunathilaka - US Food and Drug Administration

The moisture content of olive oil is a quality parameter according to CODEX and there is interest in developing rapid methods for moisture determination. We recently demonstrated for the first time that the weak near-infrared (NIR) band near 5260 cm-1 is primarily attributed to moisture. To improve accuracy, univariate calibration curves were generated based on plotting the first derivative peak-to-peak (p-p) heights of pure oils vs the gold standard Karl-Fischer (KF) primary reference method values. To enhance the speed of FT-NIR data collection, measurements were carried out in the transmission mode using disposable glass tubes. To further determine the accuracy of the moisture measurement, we also developed and compared a multivariate partial least squares approach to the univariate one and the reference KF method. In addition, to evaluate the precision of the univariate and multivariate procedures, all spectra were collected using three different FT-NIR spectrometers. The developed univariate and multivariate calibration curves for the three FT-NIR instruments were tested with 26 calibration-independent test samples that had moisture concentrations within the calibration range from 0.027-0.088 (%, as determined by KF reference method). High accuracies were found with the three FT-NIR instruments as indicated by the low root mean squared error (RMSE, %) for predicted values obtained with the univariate method (i.e. RMSE = 0.010, 0.008, and 0.010), and the multivariate method (i.e. RMSE= 0.007 for all the three instruments). These simple, rapid and accurate procedures could potentially be used for the rapid quantification of moisture contents in olive oils.

Friday, January 1, 2021

Session Time: 1:00 AM - 2:00 AM

Presentation Time: 1:00 AM - 2:00 AM

Track: Analytical@@@Industrial Oil Products

(4040) Simultaneous determination of free monochloropropanediols and free glycidol in glycerol by GCMS

Presenting Author: Hong Yang, PhD - Wilmar Intl

A method was developed and validated for simultaneous determination of free monochloropropanediols (MCPD) and free glycidol in glycerol by GCMS. These analytes are potentially carcinogenic chemical contaminants formed during glycerol processing. There is no standard method applied in free MCPD and glycidol in glycerol so far. 3-MCPD, 2-MCPD and glycidol are extracted from glycerol using diethyl ether. Glycidol is brominated to 3-bromo-1,2-

propanediol (3-MBPD). Subsequently, the free form analytes are derivatized with phenylboronic acid and subjected to GC-MS analysis. The results showed that the linear range and correlation coefficient of the method were 10-1000 ng /mL and higher than 0.9990 respectively. The range of average recoveries and relative standard deviations (RSDs) across the glycerol matrices at three spiked concentrations are 83-86% (4.78-5.72% RSD) for 3-MCPD, 81-85% (3.95-4.68% RSD) for 2-MCPD, and 81−83% (5.08−5.24% RSD) for glycidol, and the limit of detection is 10 μg/kg, the limit of quantification is 50 μg/kg. The quantitative method is useful for routine quality control of refined glycerol.

Tuesday, June 30, 2020

Session Time: 8:25 AM - 10:10 AM

Presentation Time: 8:55 AM - 9:20 AM

Track: Analytical@@@Lipid Oxidation and Quality

(3556) Machine-Learning-Driven Raman Spectroscopy for Rapidly Detecting Type and Adulteration of Edible Oils

Presenting Author: Hefei Zhao, PhD - University of Nebraska-Lincoln; University of California-Davis

Edible oils are commonly used in home cooking and industrial food manufacturing worldwide but are susceptible to adulteration. Raman spectroscopy has been introduced as a rapid detection method for oil adulteration. However, the analysis of spectra is mentally labor-intensive and time-consuming due to complicated data analysis. Machine learning has shown great advantages in data analysis and brought about breakthroughs in processing spectra and images. Hence, our central hypothesis is that integrating ML into Raman spectroscopy will significantly increase the accuracy of data analysis and therefore its power in detecting type and adulteration of edible oils. Fifteen common edible oils with 8 kinds of plant sources were purchased from supermarkets for the classification study. Avocado oil adulterated by canola oil at different levels, and olive oil by soybean oil, were prepared for the adulteration study. All the oils were subjected to collect Raman spectra. Then half of the spectra (total 357) were applied for learning procedure while the rest were implemented for the accuracy test. Ten machine learning approaches including Logistic Regression, L2 Penalty, Elast Net Penalty, Random Forest, Boosting, 2D-CNN were implemented for the classification of these spectra. The Random Forest method was found having the highest and fastest test accuracy in the classification of various pure edible oils (98.1% in 0.8 s), and in the classification of oil adulteration (94.6% in 0.7 s), followed by Bosting and L2 Penalty. This study demonstrated machine-learning-driven Raman spectroscopy significantly increased the accuracy and speed in detecting type and adulteration of edible oils.

Wednesday, July 1, 2020

Session Time: 8:25 AM - 11:45 AM

Presentation Time: 8:55 AM - 8:55 AM

Track: Analytical@@@Lipid Oxidation and Quality

(3487) Using Adsorbents to Impact Oxidative Stability of Biodiesel and its Influence on Engine Oil Deterioration

Presenting Author: Jerome Desire Aliebakaa D.A Kpan, M.Eng - Coburg University of Applied Sciences, Germany

This study presents the impact of adsorbents in augmenting the oxidative stability of biodiesel. Engine oil performance is affected by the use of biodiesel as it can accumulate in a vehicle's sump and impact engine oil durability. Compared with conventional diesel fuel, biodiesel is less likely to evaporate out of engine oil due to its higher boiling range. Biodiesel therefore, accelerates the degradation of engine oil and this result in a short period of oil drain interval. Neat base oil, 80 % blended with biodiesel, 20 % were thermo oxidatively aged. The adsorbents Magnesium aluminum hydroxycarbonate and 1,3,5-trimethyl-2,4,6-tris(3,5-di-tert-butyl-4-hydroxybenzyl)benzene were used and the formation of oligomers in the base oil-RME mixture was monitored. The two adsorbents were used in ratio of 1: 2 respectively as stated above. Various amounts, 0.225 g, 0.45 g and 0.675 g of the combined adsorbent were added to 30 mL of the oil mixture and aged at 170 °C for 80 h. The total acid number, molecular mass distribution, viscosity, density and changes in molecular structure were monitored. The analysis with FTIR showed about 60 % less the formation of oligomers as compared to the samples aged without the adsorbents. About 90 % reduction in the total acid number was recorded. There was 50% reduction in viscosity increment. The adsorbents therefore, have an enhanced impact on the oxidative stability of biodiesel and its blends.

Wednesday, July 1, 2020

Session Time: 9:45 AM - 12:35 PM

Presentation Time: 12:10 PM - 12:10 PM

Track: Analytical@@@Lipid Oxidation and Quality

(3855) Sample Preparation and Simultaneous Quantification of Major and Minor Cannabinoids in an Aqueous Matrix Using GC-MS

Presenting Author: Liyun Ye, PhD - Dalhousie University

The "entourage effect" has drawn much attention to the individual and synergistic effects of major and minor cannabinoids in recent years. However, due to a lack of harmonized sample preparation and instrumental methods, it has been challenging to conduct well-controlled clinical studies and to compare results across labs. Biosynthesized cannabinoids produced from microorganisms, such as bacteria, yeast, and algae, have further challenged this task with their complex aqueous matrices. The objective of this study was to develop a purification procedure for such complex matrices and a quantification method for major and minor cannabinoids.

Isochrysis galbana was used as the model matrix. After methanol extraction, samples were purified using solid phase extraction, derivatized with N-methyl-N-(trimethylsilyl)trifluoroacetamide, and analyzed using gas chromatography-mass spectrometry (GC-MS) in electron ionization mode. A strong anion-exchange column efficiently recovered the cannabinoids in acid form (olivetolic acid, cannabidiolic acid, tetrahydrocannabinolic acid, and cannabigerolic acid). A graphitized carbon black column was necessary to purify the neutral cannabinoids (olivetol, cannabidiol, and tetrahydrocannabinol). Both columns removed amino acids, sugars, fatty acids, alcohols, and pigments from the algae extract and prepared samples suitable for derivatization and GC-MS analysis. A sensitive and robust GC-MS method was developed to simultaneously quantify these acid-form and neutral-form cannabinoids. The method showed good linearity and reproducibility in the tested range (0.05 to 0.5 ppm in extract). This method has the potential to be a harmonized profiling tool for cannabinoids in complex matrices and to help with comparison among clinical studies.

Thursday, July 2, 2020
Session Time: 8:25 AM - 9:45 AM

Presentation Time: 8:30 AM - 8:55 AM

Track: Analytical@@@Lipid Oxidation and Quality

(3588) Evaluation of Avocado Oils Sold in the US

Presenting Author: Hilary S. Green - University of California, Davis

The demand for avocado oil has increased rapidly as consumers resonate with its potential health benefits. As the avocado oil market continues to grow, it is crucial to monitor and ensure that the oils are of the quality and purity advertised. However, there are currently no standards for avocado oil and consumers are unprotected from fraud (i.e., economic motivated adulteration). This study analyzed avocado oils currently on the market in the US to determine their quality (e.g., free fatty acidity, peroxide value, UV absorbances, vitamin E) and purity (e.g., fatty acids and sterols). It was discovered that many avocado oils not only suffer from poor quality some are also adulterated with other oils. Our results demonstrate that there is an urgent need to develop standards for avocado oil to not only ensure the consumers are getting high quality and authentic products but establishing a fair playing field for the global avocado oil industry.

Thursday, July 2, 2020
Session Time: 8:25 AM - 1:00 PM

Presentation Time: 10:55 AM - 10:55 AM

Track: Analytical@@@Lipid Oxidation and Quality

(3527) Effect of Oil Types and Frying Cycles on Flavor Compounds of French Fries during Deep-frying

Presenting Author: Lirong Xu - Jiangnan University

Flavor is a decisive sensory property to determine the popularity of deep-fried foods, the oil types and frying cycles are paramount factors to influence the manufacture of volatiles by oxidative degradation of fatty acids. In this study, the volatile compounds of French fries were monitored in 11 frying oils, and 48 frying cycles in soybean oil (SO), palm oil (PO), high-oleic rapeseed oil (HORSO). The partial least squares regression analysis investigated that (E,E)-2,4-decadienal, 2,4-decadienal, (E,E)-2,4-nonadienal, pentanal, 2-pentylfuran, 1-pentanol, trans-4,5-diepoxy(E)-2-nonenal were associated with the initial content of linoleic acid and nonanal, decanal were related to the oleic acid. The aroma profile analysis showed rice bran oil, sunflower oil and soybean oil with higher linoleic acid exhibited good deep-fried flavor. The key odor compounds (E,E)-2,4-decadienal and 2,4-decadienal of French fries in SO, PO and HORSO increased to maximum values then decreased slowly with the frying cycles. The principal component analysis results showed that the French fries in the first frying cycle had obvious distinguish with other cycles, and French fries in SO could be discriminated from those in PO and HORSO for its higher volatile compounds. This study will provide a guideline for consumers to choose the appropriated frying oil and achieve the scientific frying for ideal flavor of French fries.

Thursday, July 2, 2020

Session Time: 12:10 PM - 1:00 PM

Presentation Time: 12:10 PM - 12:35 PM

Track: Analytical@@@Lipid Oxidation and Quality

(3946) Rapid analytical approach for quality assurance of pet food products using ultrafast GC analyzer (e-nose)

Presenting Author: Lili T. Towa, PhD - Alpha MOS

One of the major causes of quality deterioration in foods is related to lipid oxidation or rancidity. During this process, lipids oxidize through a complex series of reactions generating undesirable aromas, off-flavors, color fading, browning, and/or color degradation. The products of oxidation also reduce the nutritional value of the food and can pose health risks due to the formation of oxidation products that are suspected to be toxic. Various technologies are used to determine the oxidative state of food products. Alpha MOS e-nose system proposes a fast, flexible and complementary solution for assessing the quality and the oxidative state from raw material to finished pet food products. Data analysis illustrating the use of the e-nose system to rapidly discriminate, identify and quantify off-flavors affecting pet food products will be presented. Predictive models using the e-nose profiles, hexanal concentration, peroxide values and aroma descriptions of pet food products were established.

Tuesday, June 30, 2020

Session Time: 8:25 AM - 10:10 AM

Presentation Time: 10:10 AM - 10:10 AM

Track: Analytical@@@Phospholipid

(4003) Quantitative Determination of Krill Oil Composition by ATR-FTIR, NIR, and ^{31}P NMR Spectroscopy

Presenting Author: Ashraf A. Ismail, PhD - McGill University

Marine oils are rich sources of long-chain omega-3 fatty acids that have important roles in human health, particularly eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA). In krill oil, substantial proportions of these fatty acids are esterified to glycerol in phospholipids rather than triacylglycerols, representing a key difference between the lipid profiles of krill and fish oils that is of significance in relation to health benefits. Whereas analysis of fatty acid composition in fish oil and fish-oil supplements by infrared spectroscopic methods has been reported in the literature, simultaneous determination of the concentrations of EPA, DHA, and phospholipids in krill oil by both attenuated total reflectance-Fourier transform infrared (ATR-FTIR) and near-infrared (NIR) spectroscopy was investigated for the first time in the present study. Individual ATR-FTIR and NIR calibration models were developed by employing partial-least-squares regression (PLSR) to relate the spectral data acquired for 19 samples of krill oil from various commercial sources to concentration values obtained by reference methods. Characterization of these samples by ^{31}P NMR spectroscopy was also undertaken. Cross-validation of the NIR calibration models for the prediction of EPA, DHA, and total phospholipids (PL) yielded root-mean-square errors (RMSECV) of 2.5, 3.5, and 15.8 mg/g, respectively. Slightly higher RMSECV values were obtained for the ATR-FTIR calibration models (i.e., 3.9 mg EPA/g, 3.7 mg DHA/g, and 18.6 mg PL/g). These results support the potential applicability of both NIR and ATR-FTIR spectroscopy in quality control and authentication of krill oil.

Friday, January 1, 2021

Session Time: 1:00 AM - 2:00 AM

Presentation Time: 1:00 AM - 2:00 AM

Track: Analytical@@@Phospholipid

(3911) Lipid Separation and Structural Characterization Using a Travelling Wave Ion Mobility

Presenting Author: Giorgis Isaac, PhD - Waters Corporation

Lipids represent a diverse group of biomolecules that have an essential role in structural, storage and signaling processes in living systems. The analysis and structural characterization of lipids remain challenging due to the chemical structure diversity and isobaric nature. The main objective of this presentation is to add ion mobility (IM) to the lipidomic workflows to enhance isomer separation and structural characterization of lipids. Data were collected on a SYNAPT-

XS and hybrid quadrupole cyclic IM orthogonal acceleration time-of-flight (Q-cIM-oaToF) instrument. Ion mobility separation is achieved using a multi-pass travelling-wave cyclic IM (cIM). MS and CID fragmentation data were obtained on precursor IM separated lipids for structural characterization of lipids. Unsaturated free fatty acid (FA) standards, differing in chain length and number of cis/trans configurations, were chosen to determine the degree of IM separation required to separate lipid isomers. In all direct infusion cIM-MS measurements, FAs with cis-double bond orientations, introduced as two component mixtures, were found to be more compact than those with trans-orientations. Moreover, the cis- and trans-orientations for the monounsaturated FAs were distinguishable. A different number of cycles through the cIM separator, thereby increasing the effective path length/resolution, were required to achieve a similar degree of IM separation for mono-unsaturated FAs of differing chain length. The separation of other lipid classes by cIM is currently under study. The potential of ion mobility for the separation of positional isomers and structural characterization including double bond localization will also be investigated.

Friday, January 1, 2021
Session Time: 1:00 AM - 2:00 AM

Presentation Time: 1:00 AM - 2:00 AM

Track: Analytical@@@Phospholipid

(3997) NMR Analysis of Krill Oil Detection of Counterfeiting and Fraud

Presenting Author: Bernd W.K Diehl - Spectral Service

Krill oil has become an important factor in nutritional supplements in recent years. The composition of the oil is very complex and mainly consists of phospholipids and triglycerides, but it does not contain glycolipids or sugars, such as in soya lecithin. The fatty acid composition is similar to that of fish oils, but is selectively differently between triglycerides and phospholipids. The pattern of the phospholipids is also very specific for krill and differs significantly from plant and animal sources, including those from marine origin such as fish roe. Since krill oil is a very high quality raw material and therefore has a high price, counterfeiting this product is financially lucrative, albeit criminal. For this reason, a clear, specific analysis is necessary that goes beyond the classic parameters such as gas chromatography, acetone-soluble, total phosphorus and other non-specific methods. In principle, these non-specific methods allow counterfeiting of krill oil from mixing vegetable lecithin and fish oil respectively their industrially derived products, such as fish oil ethyl ester. These types of counterfeits have now been brought onto the Asian market. NMR spectroscopy has been the USP's official method for identifying krill oil since 2012. In the study presented here, the distinction between original krill oil and the various counterfeits will be shown in detail.

Friday, January 1, 2021

Session Time: 1:00 AM - 2:00 AM

Presentation Time: 1:00 AM - 2:00 AM

Track: Analytical@@@Phospholipid

(4144) Monitoring Galactolipids Hydrolysis by Pancreatic Lipase-Related Protein 2 Using Infrared Spectroscopy

Presenting Author: Moulay Sahaka - Aix-Marseille Université

Aim/hypothesis: Galactolipids are the major components of plant membranes, in particular the membranes of the photosynthetic machinery (thylakoids) found in plant leaves and green algae. As such, these lipids are considered as the most abundant fatty acid source on the earth. However, this fatty acid source is not exploited by industry due to a high extraction cost. In this work, we explored an enzymatic process for releasing fatty acids from galactolipids. We used transmission infrared (IR) spectroscopy and Thin Layer Chromatography (TLC) to monitor continuously the hydrolysis of synthetic medium chain (C8) galactolipids by guinea pig pancreatic lipase-related protein 2 (GPLRP2) and the release of all products. Research design and Methods: Monogalactosyl di-octanoylglycerol (MGDG) substrate was mixed with sodium taurodeoxycholate (NaTDC) in D2O buffer at pD 8, and 37°C. MGDG hydrolysis reaction by GPLRP2 (at various concentrations) was monitored in a liquid cell by transmission Fourier transform infrared spectroscopy (FTIR). Residual MGDG, monogalactosyl monoacylglycerol (MGMG), monogalactosyl glycerol (MGG) and Octanoic acid (OA) were also quantified by TLC. Results: Changes in the CO stretching region of IR spectra were correlated to variations in the concentrations of MGDG, MGMG and OA using calibration curves made with these compounds, individually mixed with NaTDC in the bulk phase. We were then able to quantify the concentrations of these different compounds and their time-dependent variations during MGDG hydrolysis by GPLRP2 from IR spectrum analysis in the CO stretching region and thus, to estimate the enzyme activity under various enzyme concentration. Overall, we observed the successive hydrolysis of the MGDG substrate and the MGMG intermediate product by GPLRP2, which led to the release of a large proportion of the fatty acids initially present in MGDG. The transient accumulation of MGMG suggests that its hydrolysis by GPLRP2 occurs only after intramolecular acyl chain migration from the sn-2 to sn-1 position in glycerol.

Monday, June 29, 2020

Session Time: 8:25 AM - 10:30 AM

Presentation Time: 9:15 AM - 9:15 AM

Track: Analytical@@@Processing

(3651) Real time process control using spectroscopic techniques

Presenting Author: Jonathon D. Speed, PhD - Keit Spectrometers

Seed oil refining is a technique almost as old as human civilization, but the dawn of the fourth industrial revolution (Industry 4.0) means that we sit on the cusp of truly exciting and industry-

changing developments. These have taken the form on online, real-time measurement of processes that enables control, and ultimately greatly improved efficiency and cost-reduction. Traditional measurement techniques such as pH and conductivity, however, do not work in oil based media; whilst techniques such as colorimetry and refractive index measurements don't tell the whole story. Here we present the calibration and use of a static optics FTIR spectrometer specifically targeted at seed oil refining. We provide a brief overview of the main types of spectrometer, their strengths and weaknesses and use cases. We also explain the calibration process for a spectrometer, and discuss the merits of different calibration use cases – namely partial least squares (PLS) for real-time concentration monitoring or principal component analysis (PCA) for more qualitative measurements. We will also show the performance of monitoring total free fatty acids and fatty acid methyl acids in real time, as well as the ability to discriminate between the chemicals to better understand the makeup of the feedstock seed. We will also explore the other sorts of chemicals that can be measured using this approach (such as phosphorus, water, soap and glycerol). Lastly we hope to share the results on an online trial at a manufacturing facility to investigate the chemicals of interest during production in real world samples.

Friday, January 1, 2021
Session Time: 1:00 AM - 2:00 AM

Presentation Time: 1:00 AM - 2:00 AM

Track: Analytical@@@Processing

(4081) Unusual Long Chain Fatty Acids in Sorghum Wax

Presenting Author: Robert A. Moreau - USDA ARS ERRC, retired

The surfaces of many plant parts are often coated with various types of epicuticular waxes. These waxes provide protection from physical injury, insects, microbes, and desiccation. Both the concentration and chemical composition of epicuticular waxes vary greatly depending on species and on the anatomical plant part (leaf, flower, stem, seed, etc.). The most common chemical components in epicuticular waxes include wax esters (a compound comprised of a fatty acid esterified to a fatty alcohol) and alkanes, but can include many other types of lipids. The epicuticular wax of sorghum kernels contains a complex mixture of many types of lipids but the major components are long chain (C28 and C30) fatty acids and fatty aldehydes. The structure and function of these unusual fatty acids will be discussed and compared to the waxes in other grains.

Friday, January 1, 2021
Session Time: 1:00 AM - 2:00 AM

Presentation Time: 1:00 AM - 2:00 AM

Track: Analytical@@@Processing

(3693) Suitability of biodiesel catalysts as determined by oxygen-17 NMR

Presenting Author: Sarah K. Purdy, PhD - Univ of Saskatchewan

The use of biodiesel is increasing worldwide as a fuel made with renewable resources that produces fewer greenhouse gas emissions than petroleum products. The study and optimization of alkoxide catalyzed biodiesel production are of great interest and previous studies have probed it indirectly using ^{1}H and ^{13}C Nuclear Magnetic Resonance spectroscopy (NMR). The catalysis activity can be directly observed by NMR using the ^{17}O (I = 5/2) nuclei to determine the optimal catalyst species and concentration. In this work, measurements are performed on natural abundance, neat samples with a spin-echo pulse program using a Bruker Avance-II 600 (14.09T) spectrometer, operating at 81.3 MHz. The spin-echo pulse program decreases signal intensity, but significantly increases signal resolution. The resolution is further improved by increasing the temperature of the samples (e.g. 295K for sodium methoxide). Catalyst solutions of varying metal ion (Li, Na, K), alkoxide species (methoxide, isopropoxide, glyceroxide), alkoxide concentrations (0 - 25% v/v), and solvents (methanol, glycerol) are prepared for biodiesel production in the Bioprocessing Pilot Plant at the University of Saskatchewan. The catalyst solutions, liquid glycerol and fatty acid methyl ester products are compared using the ^{17}O-NMR spectra. Since this method is specific to one atom and the signal area is directly proportional to the concentration of the functional group, it offers an excellent method for quantitative analysis. It can be a very powerful tool for the investigation of oxygen functionalities in complex mixtures present in synthetic biofuels.

Friday, January 1, 2021
Session Time: 1:00 AM - 2:00 AM

Presentation Time: 1:00 AM - 2:00 AM

Track: Analytical@@@Processing

(4223) The Future of Specialty Canola Oils

Presenting Author: Curtis. B Rempel - Canola Council of Canada

Canola oil is one of the healthiest vegetable oils in the marketplace with zero trans fat and cholesterol, healthy monounsaturated and polyunsaturated fats, and the lowest amount of saturated fat of all common culinary oils. Today, there are two types of canola oil: commodity or classic, which is sold at retail, and high-oleic or high-stability, which is newer and sold almost exclusively to food companies and food service operations. Both oils have the same low level of saturated fat and positive health attributes. The only difference between them is that high-oleic canola oil, as its name suggests, contains more oleic acid (a monounsaturated fat) and less polyunsaturated fats, namely alpha-linolenic acid (ALA) and linoleic acid (LA). This fat profile

makes high-oleic canola oil more stable, allowing for greater heat tolerance and longer shelf life. High-oleic canola oil, and in some culinary applications classic canola oil, is a great replacement for partially hydrogenated (PH) oils used in food products and food service, which account for about 80 percent of trans fat in North America. Trans fat is artificially formed when liquid vegetable oils are turned into solid fat using a process called partial hydrogenation, hence, the term partially hydrogenated oils. New patents for new fatty acid profiles for canola have or are now being filed, and existing patents are expiring. This has significant implications for the future of canola. Furthermore, technical developments in soy and sunflower also have potential to impact the plant oil marketspace with short- and long-term implications for the canola industry. This presentation will examine the markets, economic value, logistics, and opportunities for growers and consumers for existing and emerging specialty canola oils, including high oleic or high stability canola oil.

Friday, January 1, 2021
Session Time: 1:00 AM - 2:00 AM

Presentation Time: 1:00 AM - 2:00 AM

Track: Analytical@@@Processing

(4022) Method development for the determination of volatile odor compounds in flavored rapeseed oil using headspace solid-phase microextraction arrow coupled with gas chromatography-mass spectrometry

Presenting Author: Chuan Zhou - Wilmar Biotechnology Research & Development Center (Shanghai) Co., Ltd, China

Flavored rapeseed oil is widely appreciated for its unique roasted and spicy flavor. Quantitative analysis of volatile odor compounds contributed to the characteristic odor plays an important role in sensomics approach of flavored rapeseed oil. However, sampling and sample preparation in a quantitative method having good repeatability and precision are challenging tasks to perform due to complex matrix of flavored rapeseed oil. Nowadays, headspace solid-phase microextraction (SPME) Arrow with enlarged sorption phase and arrow-shaped tips are good alternatives to replace SPME fibers for the determination of volatile odor compounds in food. This study applied a novel technique SPME Arrow followed by gas chromatography-mass spectrometry to determine key volatile odor compounds in flavored rapeseed oil. In sampling, the "matrix effects" including surface tension, viscosity, interaction and composition of fatty acid will influence the extraction parameters such as type of coating, sample amount, incubation time, temperature and rotate speed, extraction time, temperature and rotate speed. The optimization of the SPME Arrow extraction condition has a real effect on the release of volatile odor compounds. The external calibration (EC) is simple to perform and calibrate hundreds of volatile compounds according to the relation time and structure with quantitative and semi-quantitative methods. Based on our results, the present method can be considered as the most suitable approach for the determination of volatile odor compounds in flavored rapeseed oil.

Wednesday, July 1, 2020

Session Time: 9:45 AM - 12:35 PM

Presentation Time: 12:10 PM - 12:10 PM

Track: Analytical@@@Protein and Co-Products

(3522) Flax Orbitide Identification Guided by Genome Mining Strategies

Presenting Author: Ziliang Song - University of Saskatchewan

Flaxseed oil contains bitter flavor due to the presence of oil-soluble orbitides naturally produced by flax. Orbitides are a class of N-to-C-linked cyclic peptides with no disulfide bonds. The circular structure confers them high stability relative to linear peptides and thus orbitides can be used as drug scaffolds. Studies showed a broad range of biological activities including anticancer, anti-inflammatory and immunosuppressive effects that have great potential for pharmaceutical applications. The discovery of cyclic peptides has been limited by bottom-up chemical approaches, but recent advancement of whole-genome sequencing technology provides an opportunity to facilitate this process. In flax, 9 orbitides have been identified and these orbitide domains in the precursor proteins are characterized to be flanked by several conserved amino acids. The present study employed this distinct pattern as a probe to search the translated genome for similar sequences as orbitide candidates. Information such as molecular weight and sequence of the peptide candidates is useful for developing proper extraction methods, optimizing the separation by liquid chromatography and guiding the identification by mass spectrometry. To achieve better fragmentation, putative cyclic peptides can be first hydrolysed to linear peptides and subsequently analyzed.

Thursday, July 2, 2020

Session Time: 8:25 AM - 1:00 PM

Presentation Time: 11:20 AM - 11:20 AM

Track: Analytical@@@Protein and Co-Products

(3617) Methods of Protein Analysis for Nutritional Labeling and Protein Claim Verification

Presenting Author: Sneh D. Bhandari, PhD - Consultant

Consumers are concerned about what they eat for want of better nutrition and health benefits from diet. Formulated, protein-based foods, beverages and supplements with new protein sources including plants continue to drive food manufacturers' and consumers' interests. Great numbers of protein enhanced food and dietary products are being developed from a variety of new sources and made available to consumers. This has further raised importance of accurate and precise estimation of protein quantity and their nutritional quality. The protein estimations based on nitrogen content of food products are widely used for nutritional labeling. Protein digestibility-corrected amino acid score (PDCAAS) is a recognized measure of nutritional quality of a protein in diet of adults and children over 1 year of age. The digestible indispensable amino acid scores (DIAAS) is a new measure of protein quality evaluation and may provide more accurate

information about protein quality once it has developed completely. Protein efficiency ratio (PER) is a quality measure for proteins in foods for infants through 12 months of age. A protein needs to meet a minimum nutritional quality threshold before it could be considered as a significant source of protein.

Friday, January 1, 2021
Session Time: 1:00 AM - 2:00 AM

Presentation Time: 1:00 AM - 2:00 AM

Track: Analytical@@@Protein and Co-Products

(4172) Utilization of Microbially Enhanced Plant Proteins for Improved Production and Environmental Sustainability of Farmed Fish

Presenting Author: Phil S. Kerr, PhD - Prairie AquaTech, LLC

The looming challenge of fulfilling the global demand for high-quality dietary protein requires that the production of farmed fish and crustaceans be achieved with the most efficient and environmentally sustainable production practices. To meet this challenge, a proprietary production process was developed that employs microbial and enzymatic enhancement of plant proteins. In addition, an analytical system to support the production and utilization of the product was created. Diets with the protein ingredient from the process display superior macronutrient (dry matter, crude protein, and crude fat) and essential amino acid apparent digestibility compared to that from Menhaden fish meal-based diets in rainbow trout and improved performance in other fined fish species. In addition, phosphorus bioavailability is substantially superior to that from fishmeal, poultry meal, and soy protein concentrate based diets. The superior bioavailability has enabled greater reduction in discharge of phosphorus into the environment from commercial scale US trout farms relative to other diets tested. Enhanced environmental sustainability was achieved while allowing the substantial reduction of animal by-product meals in least-cost formulated rations, and enhanced value in use. Together, this demonstrates that aquaculture production practices that deploy plant-based raw materials made with microbial and enzymatic production technologies can play a crucial role in achieving greater food protein security while simultaneously delivering superior environmental sustainability.