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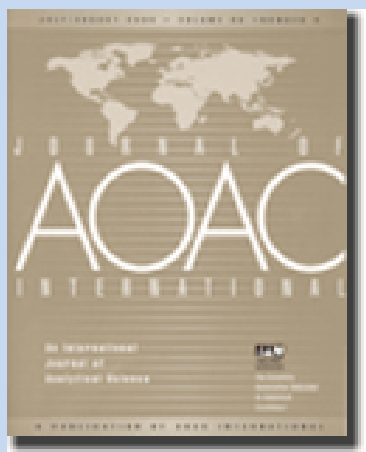

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European Regulations for Labeling Requirements for Food Allergens and Substances Causing Intolerances: History and Future

Popping, Bert; Diaz-Amigo, Carmen

ABSTRACT

Food allergens and intolerances have been diagnosed by doctors for decades, but have received heightened attention in the last two decades because diagnosis and awareness have increased. Consequently, regulators in many jurisdictions have addressed this topic by introducing labeling requirements for substances causing allergies and intolerance reactions in affected individuals. Mandatory labeling of food allergens allows persons suffering from these to make informed choices. However, regulations in some geographic areas have resulted in significant problems for manufacturers as well as consumers. This has been mainly due to frequent changes and amendments, and it has been difficult for all stakeholders to follow and understand the status quo of legislation. The present paper describes the development of European directives and regulations for the labeling of food allergens and intolerances to substances like gluten over the past decades and provides an outlook of what can reasonably be expected to change in the coming years. It also identifies existing gaps, like a lack of threshold levels for adventitious contamination and consequently a proliferation of precautionary allergen labeling, which neither benefits the consumer nor the food industry in its current form.

Japanese Food Allergen Labeling Regulation: An Update

Shoji, Masahiro1; Adachi, Reiko2; Akiyama, Hiroshi2

ABSTRACT

The Japanese food allergen labeling regulation was designed to match real Japanese food allergy circumstances and also to be enforced effectively; thus, (1) regulated food allergens were selected by prevalence and seriousness according to food allergy surveys in Japan; (2) the detection criterion for ELISA monitoring, 10 μg food allergen protein/g (or mL) food, was set up as the threshold value to regulate commercial prepackaged foods; and (3) official food allergen analytical methods, which can determine the threshold value accurately, were developed. These three points are distinctive from other countries. Furthermore, as an on-going project, the regulation has been amended according to food allergy circumstances and requirements of society. This paper presents recent changes regarding the Japanese food allergen labeling regulation. To date, the Japanese food allergen labeling regulation has been enforced for more than 15 years and seems to be working effectively. Now would be an opportune time to review the regulation for its next level of development.

Food Allergen Labeling: A Latin American Approach

Lopez, Maria Cristina

ABSTRACT

Food allergy is a public health concern almost all over the world. Although most of the countries that regulate the declaration of allergens in prepackaged foods include the list recommended by the Codex Alimentarius, some countries have added other allergens to this list due to prevalence data and regional incidence, whereas others have incorporated exceptions for some products derived from allergenic foods. Within this context, the situation in Latin America regarding these regulations is diverse. Data about prevalence of food hypersensitivity are very limited in the region. The countries that have established regulations are Brazil, Colombia, Costa Rica, Guatemala, Honduras, El Salvador, Nicaragua, Chile, Mexico, and Venezuela. Argentina has approved a regulation for the labeling of food allergens in November 2016. It only needs to be published in the Official Bulletin to go into effect. All countries follow the Codex list that includes latex and excludes sulfites, except Brazil. On the other hand, Argentina is the only country that includes exceptions. As for the methodologies for the detection of allergens in foods, this issue is a serious problem for both the food industry and control laboratories. Available methodologies are based mainly on commercial ELISA kits; currently, there are no Latin American companies that produce them, so ELISA kits are expensive and their acquisition is complicated. There is an initiative in Argentina to address all these gaps in the region through the Platform of Food Allergens (PFA), a nonprofit organization that integrates health professionals, patients, representatives of the food industry, government, and scientists. The different actions carried out by the PFA have made it possible to contact different scientific groups from other Latin American countries in order to expand this initiative and thereby promote and strengthen both public and private capacities in the region.

Action Levels for Food Allergens: An Approach for Official Food Control in Germany

Waiblinger, Hans-Ulrich1; Schulze, Gesine2

ABSTRACT

Official food control laboratories in Germany have established internal action values for the assessment of analytical results of food allergens especially obtained from samples without declaration of the specified allergen. A pragmatic approach was chosen considering the current situation for European food information legislation. Accordingly, when a positive result is obtained for an unlabeled allergen, it is not necessarily an irregularity if it can be demonstrated that the result was caused by cross-contamination. Action values take into account current analytical experiences as well as published allergologic reference doses. They are considered as internal de minimis thresholds by food control authorities that are used to support laboratories in the decision-making process and when a written expert opinion is requested by an enforcement authority. If only minor traces are detected at concentrations below the action values, further investigation of the issue and inspections at the location of manufacture can be abandoned. The present report includes a collection of results from official food control laboratories in Germany that have been evaluated in line with the aforementioned system of action levels.

Undeclared Food Allergens and Gluten in Commercial Food Products Analyzed by ELISA

Do, Andrew B.; Khuda, Sefat E.; Sharma, Girdhari M.

ABSTRACT

Undeclared allergen(s) in commercial food products are responsible for many food recalls, as reported by regulatory agencies in various countries, including the United States. Correct allergen labeling practices are essential for the safety of food-allergic consumers. However, this practice may be hindered by the introduction of allergens all along the food supply chain, including unintentionally through cross-contact. To understand the pervasiveness of undeclared allergen(s) in commercial food products, the objective of this review is to summarize the prevalence of undeclared milk, egg, hazelnut, peanut, soy, and gluten as detected by ELISA from previously published surveys. The prevalence of undeclared allergen(s) in products with or without an advisory statement was also summarized and compared. As compiled by this review, there are some food categories that may be at higher risk for containing undeclared allergen(s). However, the data on prevalence and amount of allergen present may vary widely within any particular allergen or food category. Factors, such as food survey product selection, geography, awareness of allergen/gluten issues, and/or the choice of ELISA method, may be responsible for such differences.

The Use of Visual Examination for Determining the Presence of Gluten-Containing Grains in Gluten Free Oats and Other Grains, Seeds, Beans, Pulses, and Legumes

Allred, Laura K.; Kupper, Cynthia; Quinn, Channon

ABSTRACT

Obtaining representative test samples for antibody-based testing is challenging when analyzing whole grains for gluten. When whole grains are ground into flour for testing, confocal microscopy studies have shown that gluten tends to exist as aggregates within the starch background, making single-sample testing inaccurate and complicating the ability to arrive at an accurate average from multiple samples. In addition, whole-grain products present a unique risk to gluten free consumers, in that any contamination is localized to specific servings rather than being distributed across the product lot. This makes parts-per-million values less relevant for whole-grain products. Intact grains, seeds, beans, pulses, and legumes offer an alternative opportunity for gluten detection, in that contaminating gluten-containing grains (GCGs) are visible and identifiable to the trained eye or properly calibrated optical sorting equipment. The purpose of the current study was to determine a Gluten Free Certification Organization threshold level for the maximum number of GCGs within a kilogram of nongluten grains sold as specially processed gluten free product and to determine the feasibility of this threshold by evaluating visual examination data from two major oat processors.

Managing Food Allergens in the U.K. Retail Supply Chain

Walker, Michael J.[1]; Gowland, M. Hazel[2]; Points, John[3]

ABSTRACT

The U.K. food and grocery market is highly significant financially and dominated by 10 retailers within a regulated and extremely economically competitive environment. We summarize the approach of U.K. retailers to allergen risk assessment (RA) and risk management (RM) within the U.K. legal framework and explore public visibility of retailers' allergen policies. RA and RM of allergens appear effective in curtailing retail-triggered severe food allergy reactions. However, allergen recalls remain high, precautionary allergen labeling (PAL) remains an area of confusion, and there is no consistent Web-based provision of information for consumers who have allergies. Resolution of PAL awaits an agreed-on threshold framework, but a key challenge is to engage with patients and gain their trust rather than thrust education at them. It would be helpful for retailers to publish their allergen RA and RM policies. A target should be agreed on between government and retailers for a reduction in the proliferation of PAL wording variants by a given date within the next 3 years. A further hurdle is potentially flawed allergen analysis—development of reference methods and reference materials are acknowledged needs. Laboratories should report allergen results in an informative manner, communicating uncertainty and caveats. Ideally a laboratory representative would be included on any incident control team. Efforts must continue to standardize preparedness for protecting and defending food and drink from deliberate attack.

Allergens: An Enhanced Focus

Spotz, Kristen

ABSTRACT

Food Allergy Awareness Week was created with the purpose of placing a spotlight on the seriousness of food allergies. Recognized in the United States in mid-May every year, Food Allergy Awareness Week serves as a reminder of the over 15 million Americans who suffer from food allergies. The importance of allergies and allergen labeling can be seen when looking at U.S. Food and Drug Administration recall data: of the 764 recalls in 2016, 305 (representing more than 40%) were due to undeclared allergens. However, recalls for undeclared allergens are a complex issue with numerous factors. The implementation of prevention-based systems with the necessary management components and further error-proofing the systems, along with allergen awareness embedded throughout a company's food safety culture, can likely help reduce the number of recalls for undeclared allergens. As a resource to manufacturers, the Grocery Manufacturers Association and the Food Allergy Research and Resource Program have developed several resources to assist with developing robust allergen management programs. By reducing the number of recalls for undeclared allergens, the food industry will likely increase and maintain consumer confidence and trust of the food-allergic community. This enhanced consumer confidence and trust could eventually open the door for further collaboration with the food-allergic community and, potentially, advance allergen-related policies.

Challenges and Path Forward on Mandatory Allergen Labeling and Voluntary Precautionary Allergen Labeling for a Global Company

Yeung, Jupiter[1]; Robert, Marie-Claude[2]

ABSTRACT

For food manufacturers, the label on a food package is a tool meant to alert consumers to the presence of specific allergens, allowing consumers to make informed decisions and not unnecessarily limit their food choices. Mandatory allergen labeling is used when the allergen is an intentionally added ingredient, whereas voluntary allergen labeling is used when the presence of the allergen is unintentional and may be in the finished product as a result of cross-contact. In a globalized economy, ensuring food safety is a growing challenge for manufacturers. When ingredients and technologies are sourced worldwide from multiple business partners, complexity rises, which can increase the chance for errors, leading to potential harm. Threshold science, Voluntary Incidental Trace Allergen Labelling (VITAL) reference doses, fit-for-purpose analytical technology, and common sense enable us to optimize allergen management for the benefit of allergic consumers. This is a good strategy because all stakeholders share the common goal of making foods safe and wholesome for all. Herein, we recommend that (1) senior management make science-based thresholds a priority for both regulatory authorities and the food industry; (2) VITAL 2.0 be adopted as a risk assessment and risk management tool for precautionary allergen labeling (PAL); (3) a standardized message for PAL, i.e., “may contain x,” be used to make it easily understandable to allergic consumers so they can make informed food choices; and (4) validated fit-for-purpose allergen methods be used to meet analytical needs. This is an opportunity for us to speak with one voice and demonstrate that food safety is not a competitive issue, but a shared responsibility. This approach could significantly improve allergic consumers’ lives.

The Allergen Bureau VITAL Program

Taylor, Simon Brooke[1]; Christensen, Georgina[1]; Grinter, Kirsten[2]; Sherlock, Robin[3]; Warren, Lisa[4]

ABSTRACT

This paper sets out the role of the Allergen Bureau and the Voluntary Incidental Trace Allergen Labelling (VITAL) Program from its origin in 2007 to its current iteration, VITAL 2. Herewith are outlined the scientific principles that support the program; the program’s application in the food chain; and the benefits of the program’s use to the food industry, clinicians, and the allergic consumer. VITAL was developed by the Australian and New Zealand food industry in consultation with multiple stakeholders, including consumer organizations, industry bodies, regulators, and retailers, to provide a standardized, science-based risk assessment process for the investigation of the potential presence of food allergens due to cross-contact and to determine whether, for cases in which the allergen is unable to be removed or controlled consistently, precautionary statements are required. The aim of the program is to provide a consistent process, a standardized approach, and a relevant cross-contact statement to allow the allergic consumer to make an informed decision regarding consumption of food.

Integrating Allergen Analysis Within a Risk Assessment Framework: Approaches to Development of Targeted Mass Spectrometry Methods for Allergen Detection and Quantification in the iFAAM Project

Nitride, Chiara1; Lee, Victoria2; Baricevic-Jones, Ivona1; Adel-Patient, Karine3; Baumgartner, Sabine4; Mills, E.N. Clare5

ABSTRACT

Allergen analysis is central to implementing and monitoring food allergen risk assessment and management processes by the food industry, but current methods for the determination of allergens in foods give highly variable results. The European Union-funded "Integrated Approaches to Food Allergen and Allergy Risk Management" (iFAAM) project has been working to address gaps in knowledge regarding food allergen management and analysis, including the development of novel MS and immuno-based allergen determination methods. Common allergenic food ingredients (peanut, hazelnut, walnut, cow's milk [Bos domesticus], and hen's egg [Gallus domesticus]) and common food matrixes (chocolate dessert and cookie) have been used for both clinical studies and analytical method development to ensure that the new methods are clinically relevant. Allergen molecules have been used as analytical targets and allergenic ingredients incurred into matrixes at levels close to reference doses that may trigger the use of precautionary allergen labeling. An interlaboratory method comparison has been undertaken for the determination of peanut in chocolate dessert using MS and immuno-based methods. The iFAAM approach has highlighted the need for methods to report test results in allergenic protein. This will allow food business operators to use them in risk assessments that are founded on clinical study data in which protein has been used as a measure of allergenic potency.

ILSI Europe's Food Allergy Task Force: From Defining the Hazard to Assessing the Risk from Food Allergens

Crevel, René R.W.1; Ronsmans, Stefan2; Marsaux, Cyril F.M.3; Bánáti, Diána3

ABSTRACT

The International Life Sciences Institute (ILSI) Europe Food Allergy Task Force was founded in response to early public concerns about the growing impact of food allergies almost coincidentally with the publication of the 1995 Food and Agriculture Organization-World Health Organization Technical Consultation on Food Allergies. In line with ILSI principles aimed to foster collaboration between stakeholders to promote consensus on science-based approaches to food safety and nutrition, the task force has played a central role since then in the development of risk assessment for food allergens. This ranged from consideration of the criteria to be applied to identifying allergens of public health concern through methodologies to determine the relationship between dose and the proportion of allergic individuals reacting, as well as the nature of the observed responses. The task force also promoted the application of novel, probabilistic risk assessment methods to better delineate the impact of benchmarks, such as reference doses, and actively participated in major European food allergy projects, such as EUROPREVALL, the European Union (EU)-funded project "The prevalence, cost and basis of food allergy across Europe;" and iFAAM, "Integrated approaches to food allergen and allergy risk management," also an EU-funded project. Over the years, the task force's work has evolved as answers to initial questions raised further issues: Its current work program includes a review of analytical methods and how different ones can best be deployed given their strengths and limitations. Another activity, which has just commenced, aims to develop a framework for stakeholders to achieve consensus on acceptable risk.

Assessing Almond and Peanut Allergens Using Commercially Available Immunoanalytical Kits and LC-MS/MS: A Case Study

Daly, Matthew1; Ansari, Parisa2; Häubl, Georg2; Rogers, Adrian1; Brunner, Kurt2

ABSTRACT

With an ever-increasing allergic population and an emerging market for allergen-free foods, accurate detection of allergens in foods has never been more important. Although ELISA-based methods are the most widely used for detection of allergens in food, there is a need for the development of orthogonal approaches. A commercial ELISA detected a relatively high concentration of peanut and almond in an allergen-free product. However, another commercial ELISA declared a low peanut concentration and was negative for almond. Further testing using a commercial almond lateral-flow device confirmed the results from the second ELISA kit and demonstrated that the positive detection of almond was due to cross-reactivity. An MS method was used for final confirmation that the reported results were negative for both almond and peanut.

Commercial ELISA Measurement of Allergens and Gluten: What We Can Learn from Case Studies

Lacorn, Markus; Lindeke, Stella; Siebeneicher, Susanne; Weiss, Thomas

ABSTRACT

During the last decade, results from ELISA test kits have often been criticized as being flawed. Therefore, it may appear counterintuitive that ELISAs are used for most food allergen and gluten analytical needs. One reason, in addition to the nonavailability of comparable alternative methods, is the fact that the methods used underwent a long validation and learning period in the market. This publication presents several case studies on interference, cross-reactivity issues, calibrators, fragmented allergens and gluten, matrix influences, and misunderstood intended-use statements. Afterward, the relevant validation parameter LOD, LOQ, selectivity, and precision are discussed. Finally, a comprehensive list of practical recommendations for ELISA test kit users is presented.

A Targeted LC-MS/MS Method for the Simultaneous Detection and Quantitation of Egg, Milk, and Peanut Allergens in Sugar Cookies

Boo, Chelsea C.1; Parker, Christine H.1; Jackson, Lauren S.2

ABSTRACT

Food allergy is a growing public health concern, with many individuals reporting allergies to multiple food sources. Compliance with food labeling regulations and prevention of inadvertent cross-contact in manufacturing requires the use of reliable methods for the detection and quantitation of allergens in processed foods. In this work, a novel liquid chromatography-tandem mass spectrometry multiple-reaction monitoring method for multiallergen detection and quantitation of egg, milk, and peanut was developed and evaluated in an allergen-incurred baked sugar cookie matrix. A systematic evaluation of method parameters, including sample extraction, concentration, and digestion, were optimized for candidate allergen peptide markers. The optimized method enabled the reliable detection and quantitation of egg, milk, and peanut allergens in sugar cookies, with allergen concentrations as low as 5 ppm allergen-incurred ingredient.

Development and Validation of a Quantitative ELISA for the Detection of Almond Residues in Foods

Slotwinski, Ewa; Almy, Dave; Viator, Ryan; Abouzied, Mohamed; Klein, Frank; Rice, Jennifer

ABSTRACT

Neogen Corp. has developed Veratox for Almond Allergen for use in the quantitative analysis and screening of almond protein residues in food products, such as cookies, crackers, chocolate bars, cereals, beverages, and clean-in-place rinses. Quantitation with Veratox for Almond Allergen ranges from 2.5 to 25 ppm and, with dilution, it can be extended for highly positive samples. This paper describes the findings of internal testing and validation studies designed to establish product claims for the assay of Veratox for Almond Allergen.

Novel Approaches for the MS-Based Detection of Food Allergens: High Resolution, MS3, and Beyond

Brockmeyer, Jens

ABSTRACT

The prevalence of allergic reactions to food is believed to be increasing in industrialized countries worldwide. One of the major tasks in risk management is, therefore, the analytical surveillance of allergen contamination in food and targeted proteomics using MS, which is of hugely growing interest due to its specificity and sensitivity and the possibility to analyze multiple allergens in parallel. Though approximately 200 different foods have been described as having the potential to elicit allergic reactions, current regional labeling requirements are focused on the 5–14 priority allergens that elicit the vast majority of severe reactions or that pose a risk as hidden allergens in food production. MS-based detection methods have been published for the majority of priority allergens, and this review provides an overview of the different methodological approaches, namely multiple-reaction monitoring-, high-resolution MS-, and triple-stage MS-based methods. In addition, requirements for the identification and validation of specific marker peptides and the influence of thermal processing and structural heterogeneity of allergens are discussed.

Simultaneous Analysis of Multiple Allergens in Food Products by LC-MS/MS

New, Lee Sun1; Schreiber, Andre1; Stahl-Zeng, Jianru2; Liu, Hua-Fen3

ABSTRACT

There is currently no cure for food allergies, and sufferers can only rely on the correct labeling of foods to avoid allergens. Hence, it is important that analytical methods are sensitive and accurate enough to screen for the presence of multiple allergens in food products. In this study, we developed an LC-tandem MS method that is able to simultaneously screen or quantify the signature tryptic peptides of multiple allergen commodities. This method is capable of screening and identifying egg white, skim milk, peanut, soy, and tree nuts (almond, Brazil nut, cashew, hazelnut, pecan, pine nut, pistachio, and walnut) at a detection limit of 10 ppm in incurred bread and cookies. It was further tested for the quantitative analysis of whole-egg, whole-milk, peanut butter, and hazelnut commodities, which are incurred or spiked into selected food matrixes as defined in AOAC INTERNATIONAL *Standard Method Performance Requirement* (SMPR®) 2016.002. The method demonstrated excellent sensitivity with a Method quantitative limit of 3 ppm for whole egg and 10 ppm for the remaining three allergen commodities. It also demonstrated good recovery (60-119%) and repeatability (RSD_r <20%), with an analytical range of 10–1000 ppm for each allergen commodity and was able to meet the minimum performance requirements of the SMPR.

Target Selection Strategies for LC-MS/MS Food Allergen Methods

Downs, Melanie L.; Johnson, Philip

ABSTRACT

The detection and quantitation of allergens as contaminants in foods using MS is challenging largely due to the requirement to detect proteins in complex, mixed, and often processed matrixes. Such methods necessarily rely on the use of proteotypic peptides as indicators of the presence and amount of allergenic foods. These peptides should represent the allergenic food in question in such a way that their use is both sensitive (no false-negatives) and specific (no false-positives). Choosing such peptides to represent food allergens is beset with issues, including, but not limited to, separated ingredients (e.g., casein and whey), extraction difficulties (particularly from thermally processed foods), and incomplete sequence information, as well as the more common issues associated with protein quantitation in biological samples. Here, we review the workflows that have been used to select peptide targets for food allergen detection. We describe the use and limitations of both in silico-based analyses and experimental methods relying on high-resolution MS. The variation in the way in which target selection is performed highlights a lack of standardization, even around the principles describing what the detection method should achieve. A lack of focus on the food matrixes to which the method will be applied is also apparent during the peptide target selection process. It is hoped that highlighting some of these issues will assist in the generation of MS-based allergen detection methods that will encourage uptake and use by the analytical community at large.

Assessment of Recovery of Milk Protein Allergens from Processed Food for Mass Spectrometry Quantification

Groves, Kate; Cryar, Adam; Walker, Michael; Quaglia, Milena

ABSTRACT

Assessing the recovery of food allergens from solid processed matrixes is one of the most difficult steps that needs to be overcome to enable the accurate quantification of protein allergens by immunoassay and MS. A feasibility study is described herein applying International System of Units (SI)-traceably quantified milk protein solutions to assess recovery by an improved extraction method. Untargeted MS analysis suggests that this novel extraction method can be further developed to provide high recoveries for a broad range of food allergens. A solution of α-casein was traceably quantified to the SI for the content of α-S1 casein. Cookie dough was prepared by spiking a known amount of the SI-traceable quantified solution into a mixture of flour, sugar, and soya spread, followed by baking. A novel method for the extraction of protein food allergens from solid matrixes based on proteolytic digestion was developed, and its performance was compared with the performance of methods reported in the literature.

Almond or Mahaleb? Orthogonal Allergen Analysis during a Live Incident Investigation by ELISA, Molecular Biology, and Protein Mass Spectrometry

Walker, Michael J.; Burns, Malcolm; Quaglia, Milena; Nixon, Gavin; Hopley, Christopher J.; Gray, Kirstin M.; Moore, Victoria; Singh, Malvinder; Cowen, Simon

ABSTRACT

It is now well known that an incident investigated in the United Kingdom in 2015 of cumin alleged to be contaminated with almond, a risk for people with almond allergy, was caused by the Prunus species, Prunus mahaleb. In the United Kingdom, the Government Chemist offers a route of technical appeal from official findings in the food control system. Findings of almond in two official samples, cumin and paprika, which had prompted action to exclude the consignments from the food chain, were so referred. Herein are described the approaches deployed to resolve the analytical issues during the investigation of the incidents. The cross-reactivity of ELISA to Prunus species was confirmed, and although this is useful in screening for the genus, orthogonal techniques are required to identify the species and confirm its presence. Two novel PCR assays were developed: one specific for P. mahaleb and the other a screening method capable of identifying common Prunus DNA. Peptides unique to almond and mahaleb were identified, permitting LC-tandem MS and criteria were developed for peptide identification to forensic standards. This work enables a staged approach to be taken to any future incident thought to involve Prunus species and provides a template for the investigation of similar incidents.

Highly Sensitive Matrix-Independent Quantification of Major Food Allergens Peanut and Soy by Competitive Real-Time PCR Targeting Mitochondrial DNA

Ladenburger, Eva-Maria1; Dehmer, Markus1; Grünberg, Ruben1; Waiblinger, Hans-Ulrich2; Stoll, Dieter1; Bergemann, Jörg1

ABSTRACT

The development of two competitive real-time PCR assays for the quantitative detection of trace amounts of two major food allergens, peanut and soybean, is reported. In order to achieve very low detection levels for both allergens, we established PCR primers and probes targeting mitochondrial DNA sequences. We were able to demonstrate that this approach led to an increase in detection sensitivity in the range of at least 1 order of magnitude compared with published assays targeting nuclear DNA. Furthermore, we generated corresponding competitor molecules, which were used as internal standards to compete with matrix effects that are evident during DNA extraction and PCR amplification in heterogeneous analytical matrixes like food. According to the recently described competitive quantitative PCR method published by Holzhauser et al. (2014), we performed threshold calibration against milk powder spiked with 10 ppm peanut and soy. Matrix-independent quantitative determination of peanut and soy could be demonstrated for three different calibrated food matrix standards in a range between 1 and 100 ppm. The data presented indicate that both assay concepts are powerful analytical tools for the quantitative detection of trace amounts of peanut and soy in commercial food products.

Stakeholders' Guidance Document for Consumer Analytical Devices with a Focus on Gluten and Food Allergens

Popping, Bert1; Allred, Laura2; Bourdichon, François3; Brunner, Kurt4; Diaz-Amigo, Carmen1; Galan-Malo, Patricia5; Lacorn, Markus6; North, Jennifer7; Parisi, Salvatore8; Rogers, Adrian9; Sealy-Voyksner, Jennifer10; Thompson, Tricia11; Yeung, Jupiter12

ABSTRACT

Until recently, analytical tests for food were performed primarily in laboratories, but technical developments now enable consumers to use devices to test their food at home or when dining out. Current consumer devices for food can determine nutritional values, freshness, and, most recently, the presence of food allergens and substances that cause food intolerances. The demand for such products is driven by an increase in the incidence of food allergies, as well as consumer desire for more information about what is in their food. The number and complexity of food matrixes creates an important need for properly validated testing devices with comprehensive user instructions (definitions of technical terms can be found in ISO 5725-1:1994 and the International Vocabulary of Metrology). This is especially important with food allergen determinations that can have life-threatening consequences. Stakeholders—including food regulators, food producers, and food testing kit and equipment manufacturers, as well as representatives from consumer advocacy groups—have worked to outline voluntary guidelines for consumer food allergen- and gluten-testing devices. These guidelines cover areas such as kit validation, user sampling instructions, kit performance, and interpretation of results. The recommendations are based on (1) current known technologies, (2) analytical expertise, and (3) standardized AOAC INTERNATIONAL allergen community guidance and best practices on the analysis of food allergens and gluten. The present guidance document is the first in a series of papers intended to provide general guidelines applicable to consumer devices for all food analytes. Future publications will give specific guidance and validation protocols for devices designed to detect individual allergens and gluten, as statistical analysis and review of any validation data, preferably from an independent third party, are necessary to establish a device's fitness-for-purpose. Following the recommendations of these guidance documents will help ensure that consumers are equipped with sufficient information to make an informed decision based on an analytical result from a consumer device. However, the present guidance document emphasizes that consumer devices should not be used in isolation to make a determination as to whether a food is safe to eat. As advances are made in science and technology, these recommendations will be reevaluated and revised as appropriate.

Determination of Curcuminoids in Turmeric Raw Materials and Dietary Supplements by HPLC: Single-Laboratory Validation, First Action 2016.16

Mudge, Elizabeth M.; Brown, Paula N.

ABSTRACT

The AOAC Expert Review Panel (ERP) approved a method for the quantitation of curcuminoids for consideration for First Action Official Method SM status. The previously published method summarized a single-laboratory validation of three individual curcuminoinds—curcumin, demethoxycurcumin, and bis-demethoxycurcumin—in raw materials and finished products. Method performance was compared with AOAC Standard Method Performance Requirement 2016.003. With repeatability precision ranging from 0.3 to 5.5% and recoveries from 96.6 to 103.3% in the different product matrixes, the ERP adopted the method and provided recommendations for achieving Final Action status.

First Report for Determination of d-Penicillamine in the Presence of Tryptophan Using a 2-Chlorobenzoyl Ferrocene/Ag-Supported ZnO Nanoplate–Modified Carbon Paste Electrode

Beitollahi, Hadi1; Gholami, Abbas2; Ganjali, Mohammad Reza3

ABSTRACT

In this work, a carbon paste electrode modified with Ag-ZnO nanoplates and 2-chlorobenzoyl ferrocene was prepared and applied for the determination of d-penicillamine in the presence of tryptophan. The morphologies of Ag-ZnO nanoplates were examined by scanning electron microscopy. It was found that under an optimum condition (pH 7.0), the oxidation of d-penicillamine at the surface of such an electrode occurs at a potential about 165 mV less positive than that of an unmodified carbon paste electrode. The diffusion coefficient ($D = 7.6 \times 10^{-6}$ cm2/s) and the electron transfer coefficient ($\alpha = 0.54$) for d-penicillamine oxidation were also determined. The proposed method exhibited a wide linear dynamic range of 0.03–250.0 μM, with an LOD (S/N = 3) of 0.015 μM. Moreover, the modified electrode exhibited good reproducibility and high selectivity, demonstrating its feasibility for the analytical purpose. Lastly, this new sensor was used for the determination of d-penicillamine and tryptophan in real samples.

Staphylococcal Enterotoxin Type R Pseudogene Presence in Staphylococcus aureus Reference and Outbreak Strains

Hait, Jennifer M.; Bennett, Reginald W.; Monday, Steven R.

ABSTRACT

Novel staphylococcal enterotoxins (SEs) expressed by Staphylococcus aureus strains have been described throughout the years, among these being the SE protein SER. To further characterize this toxin, this research used 13 S. aureus strains previously determined to contain the SE type R (ser) gene. These S. aureus isolates were evaluated using serological assays for identification of SEA–SEE and PCR for the detection of newly described SE and SE-like enterotoxin genes seg–seu. PCR-based cloning was performed such that the ser gene could be ligated into the pTrc99A plasmid expression vector. Ligation products were used to transform Escherichia coli (DH10Br) strains so that the ser open reading frame (ORF) could be sequenced and expressed for further characterization. Four of the 13 S. aureus strains tested harbored a ser ORF that yielded a PCR-positive result, but contained a frameshift mutation that subsequently introduced a premature stop codon abrogating expression of a full-sized functional protein. In this study, 30% of the PCR-positive ser strains tested were found to carry genes that coded for a nonfunctional SER protein, a finding that clearly illustrates the limited effectiveness of PCR for reliably evaluating enterotoxin potential for ser and, perhaps, other enterotoxin types.

Differentiation of Bovine, Porcine, and Fish Gelatins by Attenuated Total Reflectance Fourier Transform Infrared Spectroscopy (ATR-FTIRS) Coupled with Pattern Recognition

Aloglu, Ahmet Kemal; Harrington, Peter de B.

ABSTRACT

Bovine, porcine, and fish gelatins have been differentiated based on their spectra collected by attenuated total reflectance FTIR spectroscopy (ATR-FTIRS) coupled with pattern recognition. Three tree-based classification methods, a fuzzy rule-building expert system (FuRES), support vector machine classification trees (SVMTreeG and SVMTreeH), and one reference model, super partial least-squares discriminant analysis (sPLS-DA), were evaluated with and without two preprocessing techniques, namely standard normal variate (SNV) and principal component orthogonal signal correction (PC-OSC). Validation of these methods was obtained with 95% confidence intervals with 10 bootstraps and 4 Latin partitions (10:4). The ATR-FTIR spectra were used with four different ranges: full spectra (4000–650 cm–1), fingerprint region (1731–650 cm–1), specified spectra (4000–800 cm–1), and narrow fingerprint region (1731–800 cm–1). Classification rates for the methods were improved with SNV and PC-OSC when they were used separately or together. The highest classification rates were obtained from the narrow fingerprint region with SNV and PC-OSC at 97.4 ± 1.6% for FuRES, 100 ± 0% for sPLS-DA, and 99.3 ± 0.5% for both SVMTreeG and SVMTreeH. ATR-FTIRS combined with pattern recognition is a potential analytical technique for differentiating the sources of bovine, porcine, and fish gelatins with fast and reliable results.

An HPLC Method for the Determination of Isoflavones and the Evaluation of Their Antioxidant Capacity in Both Homogeneous and Microheterogeneous Systems

Montero, Guillermo; Günther, Germán; Valdés, Karina; Arriagada, Francisco; Morales, Javier

ABSTRACT

In this work, we developed an HPLC method to simultaneously quantify and hence evaluate the stability, distribution, and antioxidant capacity of six isoflavones: genistein, genistin, daidzein, daidzin, glycitin, and biochanin A. Isoflavones have been described as having an important estrogenic activity to treat menopausal symptoms and can reduce postmenopausal bone loss and also participate in the prevention of cardiovascular diseases. These beneficial properties are believed derived from their capacity to act as free-radical scavengers. Isoflavones are formulated in capsules and creams and also can be used as antioxidants in liposomes. HPLC separation was achieved on an Agilent Hypersil ODS C18 column. The mobile phase consisted of 0.02–0.2% orthophosphoric acid in water–acetonitrile with gradient elution. The diode array detector was operated at 260 nm. The hydrophobicity of isoflavones was determined through their distribution in octanol–buffer. These results allowed us to establish a relation between chemical structure, pKa, lipophilicity, and the characteristics of the dispersion medium. Photolysis of hydrogen peroxide was used to measure the HO• scavenging capability of isoflavones. In liposomes, the order of reactivity of the studied compounds was genistein > biochanin A > genistin > daidzein > daidzin > glycitin.

Mass Spectral Characterization and UPLC Quantitation of 3-Deoxyanthocyanidins in Sorghum bicolor Varietals

Stern, Nathan P.; Rana, Jatinder; Chandra, Amitabh; Balles, John

ABSTRACT

A quantitative ultra-performance LC (UPLC) method was developed and validated to successfully separate, identify, and quantitate the major polyphenolic compounds present in different varieties of sorghum (Sorghum bicolor) feedstock. The method was linear from 3.2 to 320 ppm, with an r2 of 0.99999 when using luteolinidin chloride as the external standard. Method accuracy was determined to be 99.5%, and precision of replicate preparations was less than 1% RSD. Characterization by UPLC-MS determined that the predominant polyphenolic components of the sorghum varietals were 3-deoxyanthocyanidins (3-DXAs). High-throughput screening for 3-DXA identified four unique classes within the sorghum varieties. Certain feedstock varieties have been found to have a high potential to not only be plant-based colorants, but also provide significant amounts of bioactive 3-DXAs, making them of unique interest to the dietary supplement industry.

Rapid Determination of Amino Acids in Beer, Red Wine, and Donkey-Hide Gelatin by Gradient Elution of HPLC: From Micellar Liquid Chromatography to High Submicellar Liquid Chromatography

Xie, Yunfei; Luo, Tian; Yang, Jing; Dong, Yuming

ABSTRACT

Amino acids (AAs) in beer, red wine, and donkey-hide gelatin were rapidly determined by gradient LC elution from micellar LC to high submicellar LC. Mobile phase A was a 0.075 M sodium dodecyl sulfate solution containing 20 mM ammonium acetate with a pH value adjusted to 3.5 with acetic acid solution, and mobile phase B was acetonitrile. Optimized chromatographic conditions were as follows: mobile phase B increased from 25 to 60% (v/v) in 30 min and the use of a Venusil XBP C18 column (5 μm, 250 × 4.6 mm) as the stationary phase, with a column temperature of 35°C, flow rate of 1.2 mL/min, and detection wavelength of 266 nm. The results indicated good linearity ($r2 \geq 0.9924$). The intraday precision of the retention time was RSD ≤ 1.1%, whereas interday was RSD ≤ 3.2%; intraday precision of the peak area was RSD ≤ 3.3%, whereas interday was RSD ≤ 4.9%. The range of recovery was 94.6–102.4%. The RSDs of the retention time for the AAs for the different samples were 0.04–0.31%.

Analysis of Vitamin D2 and Vitamin D3 in Infant and Adult Nutritional Formulas by Liquid Chromatography-Tandem Mass Spectrometry: A Multilaboratory Testing Study

Gill, Brendon D.; Indyk, Harvey E.

ABSTRACT

A multilaboratory testing study was conducted on AOAC First Action Method 2016.05 "Analysis of Vitamin D 2 and Vitamin D 3 in Fortified Milk Powders, Infant Formulas, and Adult/Pediatric Nutritional Formulas by Liquid Chromatography-Tandem Mass Spectrometry." Nine laboratories participated in the analysis of duplicate samples of 20 nutritional products. The samples were saponified at high temperature with lipid-soluble components extracted into isooctane; an aliquot was washed and vitamin D derivatized with 4-phenyl-1,2,4-triazoline-3,5-dione to form a high-molecular mass, easily ionizable adduct, extracted into acetonitrile and analyzed by reversed-phase LC-tandem MS. Stable isotope-labeled internal standards were used for quantitation to correct for losses in extraction and variation in derivatization and ionization efficiencies. Acceptable precision as RSD was demonstrated; repeatability ranged from 1.9 to 5.8% RSDr and reproducibility values ranged from 6.4 to 12.7% RSDR, with samples meeting the precision limits specified in the vitamin D Standard Method Performance Requirements and the guidelines recommended for the Horwitz ratio. Method accuracy was assessed using NIST 1849a Standard Reference Material, with a P-value of 0.32, indicating an absence of bias against the certified value. As expected, placebo samples not fortified with vitamin D returned negligible results.

Determination of Carotenoids in Infant, Pediatric, and Adult Nutritionals by HPLC with UV-Visible Detection: Single-Laboratory Validation, First Action 2017.04

Schimpf, Karen J.; Thompson, Linda D.; Pan, Shang-Jing

ABSTRACT

This reversed-phase HPLC method uses C30 chromatography and UV-Vis spectroscopy to determine cis and trans isomers of lutein, β-carotene, and lycopene in infant, pediatric, and adult nutritionals. Samples are saponified with a mixture of potassium hydroxide, tetrahydrofuran, and methanol, and carotenoids are extracted from saponified samples with 75 + 25 hexane–methyl tertiary butyl ether (MtBE). After extraction, a portion of the organic layer is evaporated to dryness, and the residue is dissolved in 75 + 25 10% butylated hydroxytoluene in methanol–MtBE. Prepared samples are injected into a C30 HPLC column where cis and trans isomers of lutein, β-carotene, and lycopene are separated with a methanol–MtBE gradient and detected with UV-Vis spectroscopy at 445 nm. Total carotenoid concentrations are calculated by comparison of sample peak areas with the areas of trans carotenoid standards of known concentration. During a single-laboratory validation of this method, total lutein repeatability and intermediate precision ranged from 1.89 to 14.9 and 1.93 to 14.0%, respectively, in infant and adult nutritional matrixes with concentrations >1 μg/100 g. Total β-carotene repeatability and intermediate precision ranged from 1.81 to 6.77 and 3.07 to 16.2%, respectively, in infant and adult nutritional matrixes with concentrations >1 μg/100 g, and total lycopene repeatability and intermediate precision ranged from 3.01 to 6.37 and 4.29 to 10.3%, respectively, in infant and adult nutritional matrixes with concentrations >1 μg/100 g. Mean overspike recoveries ranged from 90.3 to 95.3, 89.3 to 108, and 97.3 to 109% for lutein, β-carotene, and lycopene, respectively. The method also demonstrated good linearity. For lutein, r averaged 0.99991 over a standard range of approximately 10–250 μg/L trans-lutein. with average calibration errors of <1%. For β-carotene and lycopene, r averaged 0.99993 and 0.9998 over standard ranges of approximately 25–500 and 5–100 μg/L with calibration errors of <1 and <1.5%, respectively. Lutein, β-carotene, and lycopene LOQs in ready-to-feed nutritionals were estimated to be 0.4, 0.1, and 0.3 μg/100 g, respectively. This method met AOAC Stakeholder Panel on Infant Formula and Adult Nutritionals Standard Method Performance Requirements and was approved as a First Action Official Method at the AOAC INTERNATIONAL 2017 midyear meeting.

Using Safranin T as a Charge Transfer-Sensitive Ion-Pairing Reagent in Ultrasound-Assisted Cloud Point Extraction: Determination of Bisphenol A in Selected Beverages

Temel, Nuket Kartal; Gürkan, Ramazan

ABSTRACT

In this study, a new ultrasonic-assisted cloud point extraction method coupled with spectrophotometry was developed to extract from and determine bisphenol A (BPA) in a sample matrix. The method is based on charge transfer-sensitive ion-pairing complex formation between BPA and Safranin T in the presence of nitrate in a micellar interface at pH 5.0. The variables affecting extraction efficiency were optimized. A good linear relationship with a significant sensitivity difference was obtained in the ranges of 2–40 and 2–120 µg/L with LODs of 0.54 and 0.38 µg/L after preconcentration with two different extractants, respectively. From a preconcentration of a 15 mL sample, a preconcentration factor of 60 was obtained. Due to the lack of a certified material compatible with the sample matrix, the method was validated by conducting intraday and interday accuracy and precision studies based on two spiked QC samples (with a recovery rate greater than 94% and an RSD ranging from 3.8 to 5.9%). Moreover, the beverage samples were analyzed by the standard addition method to control for possible matrix effects. BPA was detected in the range of 1.8–7.2 µg/L in beverages with Triton X-114, whereas the levels changed to a range of 1.8–7.3 µg/L with Triton X-45. In such a way not to create a threat, these levels were considerably lower than the specific migration limit set by the European Union.

Determination of Phosphine Residues in Wheat and Yellow Corn with a New Developed Method Using Headspace and SIM Mode GC-MS

Thabit, Tamer M.A.M.1; Elgeddawy, Dalia I.H.2

ABSTRACT

Phosphine is considered the main fumigant material used for long time for controlling insect pests in stored grains. This research was focused on the determination of phosphine residues in cereal matrixes (mainly wheat and corn) after the fumigation process, using a new procedure developed to reduce the number of analytical steps and improve the chromatographic separation, identification, and quantitation of analyte, thus leading to enhanced total efficiency and sensitivity. This method, using gastight vials for extraction with 5% sulfuric acid, a heating extraction sequence, and injection with headspace and GC-MS in selected-ion monitoring mode, gave clean separation and accurate results. Repeatability was achieved for both wheat and corn after spiking samples at the 0.01 mg/kg level, with RSD values of 7.6 and 6.3%, respectively. The LOD and LOQ values were 0.46 and 1.38 µg/kg, respectively. The mean value of phosphine residue in wheat was 8.43 µg/kg, with an RSD of 8.17%, whereas it was 8.09 µg/kg in corn, with an RSD of 7.75%. All residues detected in all the replicates were below the estimated maximum residue limit for wheat and corn (0.1 mg/kg). The highest residue value for wheat was 9.85 µg/kg and the lowest was 7.70 µg/kg, whereas for corn, the highest value was 9.03 µg/kg and the lowest 7.30 µg/kg.

Optimization of the Supercritical Carbon Dioxide Separation of Bergapten from Bergamot Essential Oil

Sicari, Vincenzo

ABSTRACT

The possibility of following traditional cold-press extraction with the post process continuous separation of bergapten from bergamot essential oil was investigated. A fractionation tower was used in an experiment in which cold-pressed bergamot oil was extracted in a continuous countercurrent process by supercritical carbon dioxide under different conditions. Bergapten is fairly soluble in CO2 in its supercritical phase, in particular at a density of 277.90 kg·m–3, corresponding to a pressure of 8 MPa and temperature of 40°C. Under these conditions, an extract with 0.198% bergapten was obtained, a figure slightly below the percentage of bergapten contained in cold-pressed oil (0.21%). However, at densities below 200 kg·m–3, the amount of bergapten in the extracted oil was negligible. Of all tested conditions for separation, the best was found to be at a pressure of 8 MPa and temperature of 70°C, conditions under which bergapten was not detected. The results of the experiment showed that bergapten, and the non-volatile fraction in general, was extracted only in small quantities and was not extracted at all with at a CO2 pressure of 8 MPa.

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Easy and Fast Method for the Determination of Biogenic Amines in Fish and Fish Products with Liquid Chromatography Coupled to Orbitrap Tandem Mass Spectrometry

Kaufmann, Anton; Maden, Kathryn

ABSTRACT

A quantitative method for the determination of biogenic amines was developed. The method is characterized by the virtual absence of sample cleanup and does not require a derivatization reaction. Diluted extracts are centrifuged, filtrated, and directly injected into an ultra-HPLC column, which is coupled to a single-stage high-resolution mass spectrometer (Orbitrap). The chromatography is based on a reversed-phase column and an eluent containing an ion-pairing agent (heptafluorobutyric acid). The high sensitivity of the instrument permits the injection of very diluted extracts, which ensures stable retention times and the virtual absence of signal suppression effects. In addition, the quantification of histamine (a regulated compound) is further aided by the use of an isotopically labeled internal standard. The method was validated for three fish-based matrixes. Both the sample processing and the analytical measurement are very fast; hence, the methodology is ideal for high-throughput work. In addition, the method is significantly more selective than conventional methods (i.e., derivatization followed by LC with UV/fluorescence (FL) detection) for biogenic amines. A comparison showed that LC–UV/FL methods can produce false-positive findings due to coeluting matrix compounds.

Application of Gas Chromatography Coupled to Quadrupole-Orbitrap Mass Spectrometry for Pesticide Residue Analysis in Cereals and Feed Ingredients

Tienstra, Marc; Mol, Hans G. J.

ABSTRACT

A method for residue analysis of pesticides and polychlorinated biphenyls in cereals and feed ingredients based on QuEChERS extraction, programmed temperature vaporizer large-volume injection, and GC with electron ionization (EI) quadrupole Orbitrap full-scan high-resolution MS (60 000 full width at half-maximum at m/z 200) has been developed. In addition to full-scan acquisition, simultaneous full-scan and selected-ion monitoring acquisition was used to improve detectability in incidental cases in which analytes coeluted with intense signals from coextractants. The method was successfully validated down to 10 µg/kg for a single commodity (wheat) using matrix-matched calibration, and for multiple-feed matrixes using standard addition. Identification according to European Union requirements was achieved in >90% of the analyte/matrix combinations, and suggestions for further increasing identification rates have been made. Performance characteristics were compared to an existing method for residue analysis based on GC with EI tandem MS (triple quadrupole).

Coupling Ion Chromatography to Q-Orbitrap for the Fast and Robust Analysis of Anionic Pesticides in Fruits and Vegetables

Rajski, Łukasz1; Díaz Galiano, Francisco José2; Cutillas, Víctor1; Fernández-Alba, Amadeo R.2

ABSTRACT

Ion chromatography coupled to a quadrupole Orbitrap mass analyzer was used to develop a multiresidue method for the determination of highly polar pesticides and their metabolites (chlorate, perchlorate, fosetyl-aluminum, glyphosate, aminomethylphosphonic acid (AMPA), phosphonic acid, N-acetyl AMPA, and N-acetyl glyphosate) in fruits and vegetables. After extraction with methanol, samples were diluted 5× with water. No derivatization was applied. Pesticides were separated in an anion-exchange column. Water was used as the ion chromatography mobile phase. A gradient was created by increasing the concentration of KOH in the mobile phase. Ion chromatography provided good and stable retention and separation for all studied compounds. All investigated pesticides had an LOQ of 0.01 mg/kg and a linear range of 0.01–0.50 mg/kg. The ion ratio of the m/z ions produced was stable and adequate (deviation <30%) in all cases. The obtained mass errors (always in full-scan MS and MS2 mode) were <0.2 mDa. The high resolution (>100 000) provided by the Orbitrap analyzer with the low m/z ions obtained (e.g., m/z 80) was effective in obtaining low background matrix signals. The influence of postcolumn infusion of organic solvent on sensitivity was investigated. Acetonitrile was found to be more effective than methanol, increasing the sensitivity 3× with respect to water. The method was validated for five vegetable-based matrixes. Both the sample processing and the analytical measurement were very fast. Hence, the methodology is ideal for high-throughput work.

LC-HRMS for Characterizing Durum Wheat Pasta Production Variability and Consumer Overall Liking

Lambertini, Francesca1; Cavanna, Daniele2; Catellani, Dante3; Vigni, Mario Li4; Durante, Caterina4; D'Alessandro, Alessandro3; Suman, Michele3

ABSTRACT

Semolina pasta represents one of the most important dishes in Italian cuisine worldwide. Italy is the leader in its production and, recently, the worldwide diffusion of its production has begun to grow tremendously. The perceived quality of a food product, such as pasta, is a key feature that allows a company to increase and maintain the competitive advantage of a specific brand. The overall flavor perception of the consumer, therefore, has become as important as other key quality factors such as texture and color; thus, the food industry needs to meet consumer expectations and needs the tools to objectively "measure" the quality of food products. Untargeted fingerprinting by means of coupling LC with high-resolution MS (HRMS) has been well received within the analytical community, and different studies exploiting this approach for the characterization of high-value food products have recently been reported in the literature. In the present work, a tentative application of the sensomics approach to cluster analysis of semolina pasta obtained using different production conditions was developed to objectively define target molecules that correlate with consumer overall liking of an industrial standard product. Principal component analysis of chemical and physical testing, GC-MS, LC-HRMS, and sensory data were performed with the aim of identifying the main parameters to discern similarities and differences among samples and clustering them according to these features. The correlation between analytical data and compounds related to sensory data was further investigated, and lastly, a partial least-squares regression model for the prediction of consumer overall liking was reported.

Sensitive Detection of Neonicotinoid Insecticides and Other Selected Pesticides in Pollen and Nectar Using Nanoflow Liquid Chromatography Orbitrap Tandem Mass Spectrometry

Moreno-González, David; Alcántara-Durán, Jaime; Gilbert-López, Bienvenida; Beneito-Cambra, Miriam; Cutillas, Víctor M.; Rajski, Łukasz; Molina-Díaz, Antonio; García-Reyes, Juan F.

ABSTRACT

In this work, a new method based on nanoflow LC with high-resolution MS was developed for the determination of eight pesticides in pollen and nectar samples, including neonicotinoid insecticides and other selected pesticides commonly found in bees and beeswax. Detection was undertaken with a hybrid quadrupole-Orbitrap mass spectrometer (Q Exactive™) equipped with a commercial nanospray ion source. The extraction of pesticides from pollen samples was performed by a modified micro-QuEChERS method scaled down to Eppendorf tubes, whereas nectar samples were simply diluted with a water–methanol (95 + 5, v/v) solution. Good linearity (>0.999 in all cases) was obtained between 0.05 and 500 µg/kg and between 0.04 and 400 µg/kg for pollen and nectar, respectively. Recovery rates in pollen ranged from 85 to 97%, with RSDs <12%. Matrix effect was evaluated and showed negligible effects for all studied pesticides. The lowest concentration levels tested and validated were 0.5 and 0.4 µg/kg for pollen and nectar matrixes, respectively. In addition, selected incurred samples were studied, obtaining several positive findings in pollen and nectar samples, demonstrating the sensitivity and applicability of the proposed method.

Pesticide Residue Analysis in Fruit- and Vegetable-Based Baby Foods Using GC-Orbitrap MS

Lozano, Ana[1]; Uclés, Samanta[1]; Uclés, Ana[1]; Ferrer, Carmen[1]; Fernández-Alba, Amadeo R.[2]

ABSTRACT

This paper presents an efficiency evaluation of GC coupled with quadrupole Orbitrap MS for identification and quantitation in the multiresidue pesticide analysis of baby foods in full-scan mode. The identification criteria were studied following SANTE guidelines (retention time, mass accuracy, and ion ratio), comfortably complying with the values established, even at 0.003 mg/kg. Method validation was carried out on 15 selected GC-amenable pesticides covered by Commission Directive No. 2006/125/EC in three different baby food matrixes. Recovery studies were performed at 0.003 and 0.006 mg/kg, with 96% of the cases falling within the 70–120% range and with RSDs <15% for all the pesticides assayed. Linearity over 3 orders of magnitude was verified, with residuals <16% and correlation coefficient values >0.995. In general, matrix effect values were >100%. The LOQ was 0.003 mg/kg for 97% of the cases. The validated method was applied to 20 real baby food samples from Spain and to the European Union Proficiency Test FV-BF01 sample, in which the z-scores obtained were <1, thus demonstrating that this instrumentation has good quantitation capabilities.

Quantitative Determination of Synthesized Genotoxic Impurities in Nifuroxazide Capsules by Validated Chromatographic Methods

Abdelwahab, Nada S.[1]; Ali, Nouruddin W.[1]; Zaki, Marco M.[1]; Abdelkawy, M.[2]; El-Saadi, Mohammed T.[3]

ABSTRACT

Two accurate, selective, and precise chromatographic methods, namely TLC-densitometric and reversed-phase (RP)-HPLC, were developed and validated for the simultaneous determination of nifuroxazide (NIF) and its four synthesized impurities, which are also reported to be its related substances in the range of 10–100 μg/band and 10–100 μg/mL for NIF in the TLC and RP-HPLC methods, respectively. The developed TLC-densitometric method depended on the separation and quantitation of the studied components on silica gel 60 F254 TLC plates. Ethyl acetate–acetone–methanol–ammonia (85 + 25 + 5 + 0.5, v/v/v/v) was used as the developing system, and the separated bands were UV-scanned at 230 nm. On the other hand, the developed RP-HPLC method depended on chromatographic separation using a C8 column at 25°C and an aqueous solution of 0.1% sodium lauryl sulfate–acetonitrile as the mobile phase delivered according to the gradient elution program. Factors affecting the developed methods were studied and optimized. Also, method validation was carried out according to International Conference on Harmonization guidelines. The proposed methods were successfully applied for the determination of the studied drug in its bulk powder and in its pharmaceutical formulation. The developed methods showed no significant difference when compared with the reported RP-HPLC one. Their advantage is being the first stability-indicating methods for NIF and its genotoxic impurities.

Simultaneous Spectrophotometric Determination of Elbasvir and Grazoprevir in a Pharmaceutical Preparation

Attia, Khalid A.M.; El-Abasawi, Nasr M.; El-Olemy, Ahmed; Abdelazim, Ahmed H.

ABSTRACT

Three UV spectrophotometric methods have been developed for the simultaneous determination of two new Food and Drug Administration-approved drugs, elbasvir (EBV) and grazoprevir (GRV), in their combined pharmaceutical dosage form. These methods include dual wavelength (DW), classic least-squares (CLS), and principal component regression (PCR). To achieve the DW method, two wavelengths were chosen for each drug in a way to ensure the difference in absorbance was zero from one drug to the other. GRV revealed equal absorbance at 351 and 315 nm, for which the distinctions in absorbance were measured for the determination of EBV. In the same way, distinctions in absorbance at 375 and 334.5 nm were measured for the determination of GRV. Alternatively, the CLS and PCR models were applied to the spectra analysis because the synchronous inclusion of many unreal wavelengths rather than using a single wavelength greatly increased the precision and predictive ability of the methods. The proposed methods were successfully applied to the assay of these drugs in their pharmaceutical formulation. The obtained results were statistically compared with manufacturing methods. The results conclude that there was no significant difference between the proposed methods and the manufacturing method with respect to accuracy and precision.

A Versatile Liquid Chromatographic Method for the Simultaneous Determination of Metformin, Sitagliptin, Simvastatin, and Ezetimibe in Different Dosage Forms

El-Zaher, Asmaa A.1; Elkady, Ehab F.1; Elwy, Hanan M.2; Saleh, Mahmoud Abo El Makarim2

ABSTRACT

A new LC method is introduced with the concept of its versatile application to widely used drugs from different pharmacological classes. Metformin hydrochloride (MTF), sitagliptin phosphate (SIT), simvastatin (SIM) and ezetimibe (EZB) were simultaneously determined with a simple reversed-phase LC method in which a SIT–SIM binary mixture, present in a dosage form brand, was considered central for its development. Chromatographic separation was achieved with a mobile phase of acetonitrile and 0.02 M potassium dihydrogen phosphate (pH 5.2) (77 + 23, v/v) flowing through a C18 column (BDS Hypersil, 250 × 4.6 mm, 5 μm) at 1.2 mL/min at ambient temperature. UV detection was programmed to be carried out at 210 nm for EZB, SIT, and MTF, whereas SIM was detected at 240 nm. The method was validated according to International Conference on Harmonization guidelines. Linearity, accuracy, and precision were satisfactory over concentration ranges 4–40 μg/mL for EZB and SIM, 0.5–50 μg/mL for SIT, and 5–500 μg/mL for MTF. Coefficients of determination were >0.99 for the four drugs. LOQs found were 0.01 μg/mL for EZB, 0.02 μg/mL for SIT, 0.2 μg/mL for MTF, and 0.02 μg/mL for SIM. The developed method is simple, rapid, accurate, precise, and suitable for the routine QC analysis of the cited drugs in pharmaceutical products by conventional HPLC systems.

Rifaximin Stability: A Look at UV, IR, HPLC, and Turbidimetry Methods

Kogawa, Ana Carolina; Salgado, Hérida Regina Nunes

ABSTRACT

The study of the stability of medicines is mandated by the International Conference on Harmonization and the World Health Organization. Rifaximin, an antimicrobial marketed in the form of tablets, has no record of stability studies. Thus, the objective of the present work was to investigate the behavior and stability of rifaximin tablets for 6 months under simultaneous conditions of temperature and humidity by UV, IR, HPLC, and turbidimetry techniques. After 6 months of stability study, rifaximin tablets were shown to obey zero-order kinetics when analyzed by physicochemical methods and second-order kinetics when analyzed by a microbiological method. However, the UV method was not suitable for the evaluation of rifaximin. IR, HPLC, and turbidimetry methods can already be used to evaluate the stability of rifaximin tablets. It is important to analyze products with more than one type of method before releasing results mainly in the case of antimicrobial products in which the association of physicochemical and microbiological techniques must be a rule. Rifaximin tablets can be considered stable after 6 months under conditions of 40 ± 2°C and 75 ± 5% relative humidity.

Validated Analytical Methods for the Determination of Drugs Used in the Treatment of Hyperemesis Gravidarum in Multiple Formulations

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ABSTRACT

Quantitative multicomponent analysis is considered an analytical goal to save time and cost in analysis. Hence, this work aimed to provide sensitive and selective UV-spectrophotometric, chemometric manipulation, and ultra-performance LC (UPLC) methods for the determination of well-known coformulated antiemetics used in pregnancy, namely pyridoxine HCl (PYR), meclozine HCl, and cyclizine. The developed UV-spectrophotometric methods are dual wavelength in ratio spectra and first derivative of the ratio spectra with which PYR was determined selectively at 290.8 nm, whereas the other drugs in a ternary mixture were determined from their ratio spectra using a spectrum of PYR as a divisor in 0.1 M HCl. An ecofriendly partial least-squares regression chemometric method was applied to raw UV absorbance data for the determination of the ternary mixture in a 218–355 nm range using a three-factor, three-level design with water as the green solvent. A gradient UPLC method was developed and successfully resolved the ternary mixture within 5 min. Different ratios of water (adjusted to pH 3 with phosphoric acid) and methanol were delivered at 0.5 mL/min as the mobile phase into a Hypersil Gold C18 column (50 × 2.1 mm, 1.9 µm). The developed methods were successfully applied to different pharmaceutical formulations containing the aforementioned drugs and validated according to the International Conference on Harmonization guidelines. The results obtained were reproducible and reliable and can be applied for routine analysis and QC in laboratories.

Development and Validation of an HPLC–UV Method to Quantify Tavaborole During in Vitro Transungual Permeation Studies

Tampucci, Silvia; Terreni, Eleonora; Burgalassi, Susi; Chetoni, Patrizia; Monti, Daniela

ABSTRACT

A selective and rapid reversed-phase HPLC–UV method was developed and validated to quantify tavaborole (TAV; AN2690) in biological samples, i.e., in receiving phase and in bovine hoof membrane extract derived from in vitro transungual permeation studies. A simple solid–liquid extraction procedure was used to recover the drug from the bovine hoof slices. TAV chromatographic separation was achieved on a Luna PFP column (150 × 4.6 mm, 5 μm) using a mobile phase consisting of a 70% phosphoric acid solution (10 mM, pH 2.0) with 30% acetonitrile. The detection wavelength was set to 220 nm using a UV detector. The method exhibited good linearity in the calibration ranges, which were 0.5–8.0 and 0.03–2.5 μg/mL for the receiving phase and hoof membranes, respectively. The obtained LOD and LOQ values were 0.023 and 0.069 μg/mL, respectively, for the receiving phase and 0.0024 and 0.007 μg/mL for the bovine hoof membrane extracts. In all cases, the CV for intraday and interday precision was widely below the limit of 2%, demonstrating good precision. The analytical method described was sensitive, precise, linear, and accurate and could be applicable for clinical and bioanalytical studies as an alternative to other analytical methods, which are quite expensive and not always available in research laboratories.

Validation of Modifications to the ANSR for Listeria Method for Improved Internal Positive Control Performance

Alles, Susan1; Meister, Evan1; Hosking, Edan1; Tovar, Eric1; Shaulis, Rebecca1; Schonfeld, Mark1; Zhang, Lei1; Li, Lin1; Biswas, Preetha1; Mozola, Mark1; Donofrio, Robert1; Chen, Yi2

ABSTRACT

A study was conducted to validate a minor reagent formulation change to the ANSR for Listeria method, Performance Tested Method SM 101202. This change involves increasing the master mix volume prelyophilization by 40% and addition of salmon sperm DNA (nontarget DNA) to the master mix. These changes improve the robustness of the internal positive control response and reduce the possibility of obtaining invalid results due to weak-positive control curves. When three foods (hot dogs, Mexican-style cheese, and cantaloupe) and sponge samples taken from a stainless steel surface were tested, no significant differences in performance between the ANSR and U.S. Food and Drug Administration Bacteriological Analytical Manual or U.S. Department of Agriculture-Food Safety and Inspection Service Microbiology Laboratory Guidebook reference culture procedures were observed for any of the matrixes as determined by probability of detection analysis. Inclusivity and exclusivity testing yielded 100% expected results for target and nontarget bacteria. Accelerated stability testing was carried out over a 7 week period and showed no decrease in assay performance over time.

MC-Media Pad SA (Sanita-kun SA) for the Enumeration of Staphylococcus aureus in a Variety of Foods

Teramura, Hajime1; Betts, Gail2; Chen, Yi3; Brodsky, Michael4; Salfinger, Yvonne5

ABSTRACT

MC-Media Pad SA (formerly known as Sanita-kun SA) is a dry rehydratable film medium for the enumeration of Staphylococcus aureus. The performance of the method in a variety of foods was compared with that of ISO 6888-1:1999, Microbiology of Food and Animal Feeding Stuffs - Horizontal Method for the Enumeration of Coagulase-Positive Staphylococci (Staphylococcus aureus and Other Species) - Part 1: Technique Using Baird–Parker Agar Medium. The validated matrixes included pastrami, a sliced cooked chicken roll, cooked prawns, cold-smoked salmon, pasta salad, sandwich spread, fresh uncooked pasta, infant cereal, custard, and raw-milk Brie cheese. In the matrix study, five replicates at each of three contamination levels were tested as paired test portions. Across all matrixes, the difference in mean log10 values ranged from –0.32 to 0.10, which was within the acceptable range of –0.50 to 0.50. Thus, all 10 matrixes met the acceptance criterion at all concentration levels. Further, only two matrixes, cooked prawns and raw-milk Brie cheese, had 95% confidence limits outside the –0.50 to 0.50 criterion, and these were at the lowest concentration level for each matrix. The candidate method sr varied from 0.03 to 0.22 log10 CFU/g. This compares favorably with the reference method SD, which ranged from 0.06 to 0.30 log10 CFU/g. The candidate and reference methods detected 51 of 53 inclusivity strains, with both methods not detecting the same two strains. The candidate method did not detect any of the 32 exclusivity strains, whereas the reference method did not detect 30 of the 32 exclusivity strains; the 2 strains detected by the reference method were S. delphini and S. hyicus, both developing atypical colonies on Baird–Parker plates. The product consistency study demonstrated no significant difference between lots of product and supported the 1 year shelf life. Robustness testing yielded no significant differences when small variations were made in sample volume, incubation temperature, and incubation time. Thus, the data show equivalent or better performance of the Sanita-kun SA/MC-Media Pad SA method compared with the International Organization for Standardization reference method, in support of AOAC Performance Tested Method SM certification.

Detection of Paralytic Shellfish Toxins in Mussels and Oysters Using the Qualitative Neogen Lateral-Flow Immunoassay: An Interlaboratory Study

Dorantes-Aranda, Juan José1; Tan, Jessica Y.C.2; Hallegraeff, Gustaaf M.1; Campbell, Katrina3; Ugalde, Sarah C.1; Harwood, D. Tim4; Bartlett, Jill K.5; Campàs, Mònica6; Crooks, Steven7; Gerssen, Arjen8; Harrison, Keith9; Huet, Anne-Catherine10; Jordan, Timothy B.11; Koeberl, Martina12; Monaghan, Tim13; Murray, Sam4; Nimmagadda, Rama14; Ooms, Corinne15; Quinlan, Rae K.1; Shi, Feng14; Turner, Andrew D.9; Yakes, Betsy Jean16; Turnbull, Alison R.2

ABSTRACT

Paralytic shellfish toxins (PSTs) in bivalve molluscs represent a public health risk and are controlled via compliance with a regulatory limit of 0.8 mg saxitoxin (STX)·2HCl equivalents per kilogram of shellfish meat (eq/kg). Shellfish industries would benefit from the use of rapid immunological screening tests for PSTs to be used for regulation, but to date none have been fully validated. An interlaboratory study involving 16 laboratories was performed to determine the suitability of the Neogen test to detect PSTs in mussels and oysters. Participants performed the standard protocol recommended by the manufacturer and a modified protocol with a conversion step to improve detection of gonyautoxin 1&4. The statistical analysis showed that the protocols had good homogeneity across all laboratories, with satisfactory repeatability, laboratory, and reproducibility variation near the regulatory level. The mean probability of detection (POD) at 0.8 mg STX·2HCl eq/kg using the standard protocol in mussels and oysters was 0.966 and 0.997, respectively, and 0.968 and 0.966 using the modified protocol. The estimated LOD in mussels was 0.316 mg STX·2HCl eq/kg with the standard and 0.682 mg STX·2HCl eq/kg with the modified protocol, and 0.710 and 0.734 mg STX·2HCl eq/kg for oysters, respectively. The Neogen test may be acceptable for regulatory purposes for oysters in accordance with European Commission directives in which the standard protocol provides, at the regulatory level, a probability of a negative response of 0.033 on 95% of occasions. Its use for mussels is less consistent at the regulatory level due to the wide prediction interval around the POD.

Single-Laboratory Validation of the Neogen Qualitative Lateral Flow Immunoassay for the Detection of Paralytic Shellfish Toxins in Mussels and Oysters

Turnbull, Alison R.1; Tan, Jessica Y.C.1; Ugalde, Sarah C.2; Hallegraeff, Gustaaf M.2; Campbell, Katrina3; Harwood, D. Tim4; Dorantes-Aranda, Juan José5

ABSTRACT

Detection of paralytic shellfish toxins (PSTs) in bivalve shellfish by analytical methods is complicated and costly, requiring specific expertise and equipment. Following extensive blooms of Alexandrium tamarense Group 1 in Tasmania, Australia, an investigation was made into commercially available screening test kits suitable for use with the toxin profiles found in affected bivalves. The qualitative Neogen rapid test kit, with a modified protocol to convert gonyautoxins GTX1&4 and GTX2&3 into neosaxitoxin and saxitoxin (STX), respectively, with higher cross-reactivities, was the best fit-for-purpose. This validation study of the test kit and the modified protocol was undertaken following AOAC INTERNATIONAL guidelines for the validation of qualitative binary chemistry methods. The validation used four different PST profiles representing natural profiles found in Australia and in Europe: two in a mussel matrix and two in an oyster matrix. The test kit was shown to have appropriate selectivity of the toxin analogs commonly found in bivalve shellfish. The matrix and probability of detection (POD) study showed that the rapid test kit used with the modified protocol was able to consistently detect PST at the bivalve regulatory level of 0.8 mg STX·2HCl eq/kg, with a POD estimated via the binomial logistic regression of 1.0 at 0.8 mg STX·2HCl eq/kg in all tested profiles in both matrixes. The POD at 0.4 mg STX·2HCl eq/kg was 0.75 and 0.46 for the two toxin profiles in an oyster matrix and 0.96 and 1.0 for the two toxin profiles in a mussel matrix. No significant differences in the PODs of the PSTs at the regulatory level were found between production lots of the test kits. The results suggest the method is suitable to undergo a collaborative validation study.

Extraction and Determination of Trace Amounts of p-Coumaric Acid in Vinegar, Carrot Juice, and Seed Extract from Silybum marianum (L.) Gaertn

Khani, Rouhollah1; Rostami, Zeinab1; Bagherzade, Ghodsieh1; Khojeh, Vahid2

ABSTRACT

In this study, for the monitoring and quantification of p-coumaric acid (p-CA) in vinegar, carrot juice, and seed extract from the plant species Silybum marianum (L.) Gaertn, an efficient and low-cost analytical method has been applied. For this purpose, a dispersive liquid–liquid microextraction (DLLME) method, followed by UV-Vis spectrophotometric detection, was used. To form a cloudy solution, a binary mixture containing ethanol as a disperser solvent and chloroform as an extraction solvent was rapidly injected by syringe into a sample solution containing p-CA. After centrifugation, dilution of the obtained organic phase was done with the proper amount of ethanol, and the phase was transferred into a micro cell for subsequent measurement. Some effective parameters for the DLLME method, such as the volume of disperser solvent and extraction solvent, pH, and salt concentration were inspected by a 24 full factorial central composite design using design Export Software. Under the optimized conditions, linearity was between 10 and 150 ng/mL, and the LOD was 2.3 ng/mL. The results of the proposed method were similar to the obtained results using a GC with flame-ionization detection method.

Optimized Mass Spectrometry-Based Metabolite Extraction and Analysis for the Geographical Discrimination of White Rice (Oryza sativa L.): A Method Comparison Study

Lim, Dong Kyu1; Long, Nguyen Phuoc1; Mo, Changyeun2; Dong, Ziyuan1; Lim, Jongguk2; Kwon, Sung Won3

ABSTRACT

In this study, we examined the effects of different extraction methods for the GC-MS- and LC-MS-based metabolite profiling of white rice (Oryza sativa L.). In addition, the metabolite divergence of white rice cultivated in either Korea or China was also evaluated. The discrimination analysis of each extraction method for white rice from Korea and China and the corresponding discriminatory markers were estimated by unpaired t-test, principal component analysis, k-means cluster analysis, partial least-squares discriminant analysis (PLS-DA), and random forest (RF). According to the prediction parameters obtained from PLS-DA and RF classifiers as well as features that could be identified, the extraction method using 75% isopropanol heated at 100°C coupled with LC-MS analysis was confirmed to be superior to the other extraction methods. Noticeably, lysophospholipid concentrations were significantly different in white rice between Korea and China, and they are novel markers for geographical discrimination. In conclusion, our study suggests an optimized extraction and analysis method as well as novel markers for the geographical discrimination of white rice.

Highly Sensitive GMO Detection Using Real-Time PCR with a Large Amount of DNA Template: Single-Laboratory Validation

Mano, Junichi; Hatano, Shuko; Nagatomi, Yasuaki; Futo, Satoshi; Takabatake, Reona; Kitta, Kazumi

ABSTRACT

Current genetically modified organism (GMO) detection methods allow for sensitive detection. However, a further increase in sensitivity will enable more efficient testing for large grain samples and reliable testing for processed foods. In this study, we investigated real-time PCR-based GMO detection methods using a large amount of DNA template. We selected target sequences that are commonly introduced into many kinds of GM crops, i.e., 35S promoter and nopaline synthase (NOS) terminator. This makes the newly developed method applicable to a wide range of GMOs, including some unauthorized ones. The estimated LOD of the new method was 0.005% of GM maize events; to the best of our knowledge, this method is the most sensitive among the GM maize detection methods for which the LOD was evaluated in terms of GMO content. A 10-fold increase in the DNA amount as compared with the amount used under common testing conditions gave an approximately 10-fold reduction in the LOD without PCR inhibition. Our method is applicable to various analytical samples, including processed foods. The use of other primers and fluorescence probes would permit highly sensitive detection of various recombinant DNA sequences besides the 35S promoter and NOS terminator.

Evaluation of a Commercial Sandwich Enzyme-Linked Immunosorbent Assay for the Quantification of Beta-Casomorphin 7 in Yogurt Using Solid-Phase Extraction Coupled to Liquid Chromatography-Tandem Mass Spectrometry as the "Gold Standard" Method

Nguyen, Duc Doan1; Busetti, Francesco2; Johnson, Stuart Keith3; Solah, Vicky Ann3

ABSTRACT

This study investigated beta-casomorphin 7 (BCM7) in yogurt by means of LC-tandem MS (MS/MS) and enzyme-linked immunosorbent assay (ELISA) and use LC-MS/MS as the "gold standard" method to evaluate the applicability of a commercial ELISA. The level of BCM7 in milk obtained from ELISA analysis was much lower than that obtained by LC-MS/MS analysis and trended to increase during fermentation and storage of yogurt. Meanwhile, the results obtained from LC-MS/MS showed that BCM7 degraded during stages of yogurt processing, and its degradation may have been caused by X-prolyl dipeptidyl aminopeptidase activity. As a result, the commercial sandwich ELISA kit was not suitable for the quantification of BCM7 in fermented dairy milk.

Composition, Distribution, and Antioxidant Activity of Phenolic Compounds in 18 Soybean Cultivars

Zhu, Yi-Lin; Zhang, Hai-Sheng; Zhao, Xin-Shuai; Xue, Huan-Huan; Xue, Jing; Sun, Yu-Han

ABSTRACT

Natural phenols are an important functional compound widely distributed in plants with benefits that promote human health. The content of total phenols, flavonoids, and anthocyanins and their composition distribution in 18 soybean cultivars was investigated. There are four phenolic acid distribution forms in these soybean cultivars, namely free, esterified, glycosided, and insoluble-bound. Total phenols, flavonoids, and anthocyanins from 6 black soybean cultivars were found in higher numbers than those from 12 other yellow soybean cultivars. Free and esterified phenolic acids were the main phenolic acid form in all 18 soybean samples. Chlorogenic acid and caffeic acid were the dominant phenolic acids in eight detected phenolic acids, and daidzin and genistin were the abundant isoflavones in five detected isoflavones. Furthermore, the antioxidant activities of total phenols from the 6 black soybean cultivars were greater than those from the 12 yellow soybean cultivars, and there was a significant positive correlation between antioxidant activity and total phenolic content. Black soybeans could be a potential resource for developing natural antioxidants that may play a crucial role in human health protection.

Influence of Gamma Irradiation on Porcine Serum Albumin Structural Properties and Allergenicity

Zhu, Xudong1; Wang, Wei2; Shen, Juan2; Xu, Xinglian3; Zhou, Guanghong3

ABSTRACT

Pork provides an ideal source of food energy; however, pork can elicit an allergic reaction, and porcine serum albumin (PSA) has been identified as a major allergen. This study examined the impact of gamma irradiation on the allergenicity and structural qualities of PSA; the PSA solution was gamma-irradiated at 1, 2, 4, 6, and 8 kGy. Allergenicity was investigated by immunoblotting and competitive indirect ELISA using serum from patients who are allergic to pork, and conformational changes in irradiated PSA were measured by circular dichroism, sulfhydryl group detection, and fluorescence emission spectra. The secondary and tertiary structures of gamma-irradiated PSA were altered, and the allergenicity of PSA was lowered by boosting the amount of irradiation. In addition, there is high correlation between depletion in the α-helix and immunoglobulin E-binding capability of PSA. The results show a new possibility in using gamma irradiation to reduce the allergenicity of pork products.

Minerals and Trace Elements in Milk, Milk Products, Infant Formula, and Adult/Pediatric Nutritional Formula, ICP-MS Method: Collaborative Study, AOAC Final Action 2015.06, ISO/DIS 21424, IDF 243

Pacquette, Lawrence H.; Thompson, Joseph J.; Malaviole, I.; Zywicki, R.; Woltjes, F.; Ding, Y.; Mittal, A.; Ikeuchi, Y.; Sadipiralla, B.; Kimura, S.; Veltman, H.; Miura, A.

ABSTRACT

AOAC Final Action Official Method SM 2015.06 “Minerals and Trace Elements in Milk, Milk Products, Infant Formula and Adult/Pediatric Nutritional Formula, ICP-MS Method” was collaboratively studied. Note that “milk, milk products” has now been added to the title of the Final Action method because whole milk and several dairy ingredients were successfully incorporated into the collaborative study for the purpose of developing an International Organization for Standardization/International Dairy Federation standard (ISO/DIS 21424; in progress). The method determines sodium, magnesium, phosphorus, potassium, calcium, iron, manganese, zinc, copper, chromium, molybdenum, and selenium by inductively coupled plasma (ICP)-MS after microwave digestion. Ten laboratories participated in the study, and data from five different model ICP-MS units were represented. Thirteen products, five placebo products, and six dairy samples were tested as blind duplicates in this study, along with a standard reference material, for a total 50 samples. The overall repeatability and reproducibility for all samples met Standard Method Performance Requirements put forth by the AOAC Stakeholder Panel on Infant Formula and Adult Nutritionals, with a few exceptions. Comparisons are made to ICP-atomic emission data from a collaborative study of AOAC Official Method 2011.14 carried out concurrently on these same samples.

Method Modification for the Atlas Listeria Environmental LE Detection Assay Using FoodChek Actero Listeria Enrichment Media and Half-Fraser Media for the Detection of Listeria spp. from Environmental Surfaces

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ABSTRACT

Two candidate method modifications for the Atlas Listeria Environmental LE Detection Assay were compared with the U.S. Department of Agriculture (USDA)-Food Safety and Inspection Service Microbiology Laboratory Guidebook 8.09 (MLG 8.09) method for detection of Listeria spp. on stainless steel, polyvinyl chloride (PVC), and sealed concrete surfaces. For LE candidate method 1, samples were enriched in FoodChek Actero Listeria Enrichment Media [ALEM; Performance Tested Method SM (PTM) 111201] at 35 ± 2°C for 18 to 24 h and evaluated for a range of analytical sample volumes. For LE candidate method 2, the current Roka PTM using 90 mL of Half-Fraser broth for enrichment at 35 ± 2°C was evaluated at 24 h with a reduced sample volume. These comparisons were made in multiple studies across the three environmental surfaces. Within each method and study, a total of 5 samples were uninoculated, 20 samples were inoculated with Listeria spp. at a low level to target fractional positivity, and 5 samples were inoculated with Listeria spp. at a high level to approach a probability of detection of 1. Inclusivity and exclusivity studies were also conducted for the LE method in combination with Half-Fraser and ALEM. The Atlas Listeria Environmental LE Detection Assay detected all 50 inclusive organisms, including 25 strains of L. monocytogenes and 5 strains of each of the other five common species of Listeria (L. innocua, L. welshimeri, L. ivanovii, L. seeligeri, and L. grayi) and none of the 30 exclusive organisms across all media and with both 200 and 2000 μL sample volumes. For the LE candidate method 1 studies, no significant differences were observed within the Roka ALEM method at 18, 20, or 24 h and for both the 200 and 2000 μL sample volumes as compared with the paired culture outcome. However, the ALEM method performed significantly better as compared with the unpaired reference method for sealed concrete and stainless steel. For the LE candidate method 2 studies, no significant differences were observed within the Roka HF method at 24 h for the 200 and 2000 μL samples as compared with the paired culture outcomes and unpaired reference method outcomes across the surfaces. The independent laboratory studies observed no significant differences in performance between the USDA/MLG 8.09 reference method and candidate methods 1 or 2, respectively, across the evaluated parameters. Overall, the candidate method 1 modification parameters and candidate method 2 sample parameters for the Atlas Listeria Environmental LE Detection Assay were statistically equivalent to or better than the reference method for detection of Listeria spp. on stainless steel, PVC, and sealed concrete surfaces, providing greater flexibility in method application for end users.

Quantification of Hexavalent Chromium in Surface Water Samples by a Selective Electrochemical Method

Baig, Jameel Ahmed1; Bhutto, Ashfaque Ali1; Uddin, Siraj1; Kazi, Tasneem Gul1; Khan, Muhammad Irfan2

ABSTRACT

The current study aimed to develop a robust, selective, and sensitive voltammetric method for hexavalent chromium (CrVI) at a chemically modified carbon paste electrode. For the preparation of the electrode, a micropipet tip was packed with modified carbon paste mainly consisting of graphite powder and diphenylcarbazone (5 + 1, w/w). Voltammetric mode, type of electrolyte, pH, volume of electrolytes, accumulation time, accumulation potential, and stirring rate were studied in detail. The current response was linearly dependent on the concentration of CrVI from 0.20 to 2.60 µmol/L. The reproducible results were obtained for replicate analyses (n = 11) of three proposed electrodes of the same composition with RSDs of <2.0%. The LODs and LOQs were found to be 0.052 and 0.174 µmol/L, respectively. The noticeable electrode surface passivation was not observed for the detection of CrVI. The proposed methods were successfully applied for CrVI in different surface waters in Sindh, Pakistan.

Determination of Residues of Diazinon and Chlorpyrifos in Lavender and Rosemary Leaves by Gas Chromatography

Rezk, Mamdouh R.1; Abd El-Aleem, Abd El-Aziz B.1; Khalile, Shaban M.2; El-Naggar, Omneya K.2

ABSTRACT

A sensitive gas chromatographic (GC) GC method has been developed for the determination of diazinon and chlorpyrifos residues in lavender and rosemary leaves. The developed method consists of blending weighed samples of chopped leaves with sodium sulfate as the dehydrating agent, extraction with ethyl acetate, filtration, evaporation with a rotary evaporator, and, finally, capillary GC determination of the pesticides. The recoveries of the method were greater than 90%, and the LOQ was less than 0.1 µg/mL. The method was applied to determine the rate of disappearance of diazinon and chlorpyrifos from lavender and rosemary leaves pretreated with the studied pesticides. The half-life values (t 1/2) of diazinon were found to be 5.93 and 6.35 days for lavender and rosemary leaves, respectively, whereas the t 1/2 values of chlorpyrifos were calculated to be 7.86 and 9.52 days for lavender and rosemary leaves, respectively. The safe harvest interval (preharvest interval; PHI) was suggested to be after 21 and 24 days for diazinon and chlorpyrifos, respectively. The PHI refers to the amount of time that must lapse (in days) after a pesticide application before a crop can be cut.

Determination of Selenium and Arsenic Ions in Edible Mushroom Samples by Novel Chloride–Oxalic Acid Deep Eutectic Solvent Extraction Using Graphite Furnace-Atomic Absorption Spectrometry

Zounr, Rizwan Ali1; Tuzen, Mustafa2; Khuhawar, Muhammad Yar3

ABSTRACT

In present study, we proposed the application of a deep eutectic solvent (DES) made up of choline chloride (ChCl) and oxalic acid (Ox) for the dissolution of different edible mushroom samples for the determination of selenium (Se) and arsenic (As) ions. Therefore, an innovative, green, novel, and inexpensive method based on ChCl–Ox as the DES was developed for the determination of Se and As ions in mushroom species by graphite furnace-atomic absorption spectrometry. The important analytical parameters were also optimized. The LODs for Se and As ions were found to be 0.32 and 0.50 μg/L, respectively. The LOQs for Se and As ions were found to be 1.06 and 1.65 μg/L, respectively. The RSD was observed to be less than 5% for both analyte ions. The accuracy of the developed method was confirmed by analyzing mushroom powder Certified Reference Material CS-M-3 (Boletus edulis). The developed technique was effectively useful for the determination of Se and as ions in different species of mushroom samples from Turkey.

Establishment of a Decaplex PCR-Capillary Gel Electrophoresis Method for the Simultaneous Detection of Six Kinds of Genetically Modified Animals

Liu, Xiaofei1; Qiu, Songyin1; Li, Xiaolin1; Liu, Dandan1; Jing, Hongli1; Wang, Qin1; Lin, Xiangmei1; Pan, Dengke2; Shi, Ningning2

This study aimed to establish an event-specific multiplex PCR system using microsatellite markers and fluorescently labeled primers to detect six different genetically modified (GM) animal lines, including human lactoferrin GM cattle, human lysozyme GM cattle, human α-lactalbumin GM cattle, myostatin knockout pigs, phytase GM pigs, and ω-3 fatty acid desaturase gene GM pigs. Four different microsatellite loci for species identification, along with six GM animal-specific fragments, were selected as targets for primer design. The capillary gel electrophoresis results of multiplex PCR showed that the target fragments were amplified successfully. This high-throughput multiplex PCR detection system can be applied for the inspection and quarantine of GM animals.

Mycotoxin Crises: Fit-for-Purpose Analytical Responses in the Developing World

Shephard, Gordon S.

ABSTRACT

In developed market economies, control of mycotoxin exposure in the general population is achieved by legislated regulations governing maximum permitted levels. Such regulations are widely enforced to prevent outbreaks of overt mycotoxicoses. In developing countries, particularly in Africa, the situation is reversed, and individual mycotoxin exposures can be high, especially in rural communities reliant on subsistence or small-holder farming and local markets. Besides the effects of chronic mycotoxin exposure, Africa in recent years has experienced outbreaks of acute toxicity, such as aflatoxicosis. Recognizing and handling mycotoxin-induced health crises requires a range of responses, many of which rely on the provision and availability of fit-for-purpose analytical methods. Although regional laboratories may be able to provide support, rapid responses require in-field test kits reliant on antibody technologies. The future development of aptamers into test systems may be an important component of these analytical responses, as they provide important advantages in terms of stability, shelf-life, and low production costs.

Analytical Methods for Mycotoxin Detection in Southeast Asian Nations (ASEAN)

Lim, Chee Wei; Chung, Gerald; Chan, Sheot Harn

ABSTRACT

Aflatoxins B1 (AFB1) and B2 (AFB2) and G1 and G2 remain the top mycotoxins routinely analyzed and monitored by Association of Southeast Asian Nations (ASEAN) national laboratories primarily for food safety regulation in the major food commodities, nuts and spices. LC tandem fluorescence detection (LC–fluorescence) represents a current mainstream analytical method, with a progressive migration to a primary method by LC tandem MS (MS/MS) for the next half decade. Annual proficiency testing (PT) is conducted by ASEAN Food Reference Laboratories (AFRLs) for mycotoxin testing as part of capability building in national laboratories, with the scope of PT materials spanning from naturally mycotoxin-contaminated spices and nuts in the early 2010s to the recent contamination of corn flour in 2017 for total aflatoxin assay development. The merits of the mainstream LC–fluorescence method are witnessed by a significant improvement ($P < 0.05$) in PT z-score passing rates (≤ 2) from 11.8 to 79.2% for AFB1, 23.5 to 83.3% for AFB2, and 23.5 to 79.2% for total aflatoxins in the last 5 years. This paper discusses the journey of ASEAN national laboratories in analytical testing through AFRLs, and the progressive collective adoption of a multimycotoxin LC-MS/MS method aided by an isotopic dilution assay as a future primary method for safer food commodities.

Detection of Total Ergot Alkaloids in Cereal Flour and in Bread by a Generic Enzyme Immunoassay Method

Gross, Madeleine1; Curtui, Valeriu2; Usleber, Ewald2

ABSTRACT

Four sets of polyclonal antibodies against ergot alkaloids ergometrine, ergotamine, α-ergocryptine, and ergocornine were produced and characterized in a competitive direct or indirect enzyme immunoassay (EIA). Standard curve LODs were 0.03 ng/mL (ergometrine EIA) to 2.0 ng/mL (ergocornine EIA). Three EIAs were highly specific, whereas the ergometrine EIA had a broad specificity pattern and reacted, albeit weakly, with all seven major ergot alkaloids and their epimeric forms. Using the ergometrine EIA, a generic test system was established in which total ergot alkaloids are quantified by a standard curve for a toxin mixture composed of three alkaloids that matched the ergot alkaloid composition in naturally contaminated rye and wheat products. Sample extraction with acetonitrile–phosphate-buffered saline at pH 6.0 without further cleanup was sufficient for EIA analysis. The LODs for total ergot alkaloids were 20 ng/g in rye and wheat flour and 14 ng/g in bread. Recoveries were 85–110% (RSDs of 0.1–11.7%) at a concentration range of 50–1000 ng/g. The total ergot alkaloid EIA was validated by comparison with HPLC–fluorescence detection. Although some under- and overestimation by the total ergot alkaloid EIA was observed, it was suitable for the reliable identification of positive samples at 10–20 ng/g and for the determination of total ergot alkaloids in a concentration range between 100 and 1000 ng/g.

Ultra-High-Performance Supercritical Fluid Chromatography as a Separation Tool for Fusarium Mycotoxins and Their Modified Forms

De Boevre, Marthe1; Van Poucke, Christof1; Ediage, Emmanuel Njumbe1; Vanderputten, Dana2; Van Landschoot, Anita3; De Saeger, Sarah1

ABSTRACT

A simple, reliable method for the detection of free and modified Fusarium mycotoxins in beer using state-of-the-art ultra-high-performance supercritical fluid chromatography (UHPSFC) with low-resolution tandem MS (MS/MS) is presented in this paper. The UHPSFC-MS/MS method was developed for nivalenol, deoxynivalenol, 15-acetyl-deoxynivalenol, 3-acetyl-deoxynivalenol, deoxynivalenol-3-glucoside, HT-2 toxin, T-2 toxin, T-2 toxin-3-glucoside, neosolaniol, diacetoxyscirpenol, zearalenone, α-zearalenol, and β-zearalenol and their internal standards deepoxy-deoxynivalenol and zearalanone. Due to the broad range of the physicochemical properties of the aforementioned, the sample preparation step was minimized to avoid analyte losses. Extraction with acetonitrile–water–acetic acid (79 + 20 + 1, v/v/v) and hexane in combination with solid-phase extraction (C18) was followed by a filtration step. After filtration, the extract was evaporated, and the remaining residue was redissolved in a mobile phase for injection (methanol–water; 90 + 10, v/v). A mobile phase consisting of supercritical CO_2 and a small portion of methanol was used. The developed multimycotoxin method permits the simultaneous determination of multiple fusariotoxins in an one-step chromatographic run using UHPSFC-MS/MS. SFC is a promising strategy; however, the retention mechanism is complex, leading to the unpredictable nature of elution and to some mycotoxins not being retained on the column. This restricts the applicability of UHPSFC in multimycotoxin analyses. The present study is the first report on the use of UHPSFC for the analysis of free and modified Fusarium mycotoxins.

Optimized QuEChERS Method Combined with UHPLC-MS/MS for the Simultaneous Determination of 15 Mycotoxins in Liquorice

Huang, Xiaojing1; Wang, Shaomin2; Mao, Dan2; Miao, Shui2; Hu, Qing2; Ji, Shen2

ABSTRACT

In our study, a reliable and rapid analytical method for the simultaneous determination of 15 mycotoxins (aflatoxin B1, aflatoxin B2, aflatoxin G1, aflatoxin G2, alternariol, agroclavine, citrinin, diacetoxyscirpenol, deoxynivalenol, fumonisin B1, fumonisin B2, ochratoxin A, sterigmatocystin, T-2 toxin, and zearalenone) in liquorice using ultra-HPLC coupled to tandem MS was developed and validated. Due to the complex ingredients in liquorice, we chose a QuEChERS-based extraction procedure as the sample pretreatment. Meanwhile, for the first time, acetate buffer was used to replaced water, which can greatly reduce the concentration of formic acid in acetonitrile, which further reduces the extraction efficiency of impurities. The optimal combination of adsorbents is 150 mg primary secondary amine, 150 mg silica gel, 600 mg octadecylsilane, and 900 mg anhydrous magnesium sulfate. Electrospray ionization in both positive- and negative-ionization modes was applied to detect all the mycotoxins in a single run time of 15 min, with LOQs in the range of 0.125–2.5 µg/kg. The recoveries of determination obtained were in the range of 81.0–104.7%, whereas the analytes could be accurately quantified in the 0.25–625 µg/kg concentration range, with all coefficients being >0.992. Intra- and interday reproducibility were lower than 5.5 and 8.9%, respectively, for all analytical mycotoxins. The validated method was finally applied to screen mycotoxins in 31 batches of real samples collected from drugstores and hospitals in Shanghai, China. Our survey findings show that six mycotoxins were detected, including alternariol, citrinin, deoxynivalenol, fumonisin B1, ochratoxin, and zearalenone, and that the positive rate of mycotoxins was 54.8% in real samples, ranging from 3.37 to 520.6 µg/kg.

Quantitation of Mycotoxins Using Direct Analysis in Real Time Mass Spectrometry (DART-MS)

Busman, Mark

ABSTRACT

Ambient ionization represents a new generation of MS ion sources and is used for the rapid ionization of small molecules under ambient conditions. The combination of ambient ionization and MS allows the analysis of multiple food samples with simple or no sample treatment or in conjunction with prevailing sample preparation methods. Two ambient ionization methods, desorptive electrospray ionization (DESI) and direct analysis in real time (DART) have been adapted for food safety application. Both ionization techniques provide unique advantages and capabilities. DART has been used for a variety of qualitative and quantitative applications. In particular, mycotoxin contamination of food and feed materials has been addressed by DART-MS. Applications to mycotoxin analysis by ambient ionization MS and particularly DART-MS are summarized.

Multimycotoxin Analysis by LC-MS/MS in Cereal Food and Feed: Comparison of Different Approaches for Extraction, Purification, and Calibration

Solfrizzo, Michele1; Gambacorta, Lucia1; Bibi, Rita2; Ciriaci, Martina2; Paoloni, Angela2; Pecorelli, Ivan2

ABSTRACT

Twelve different approaches commonly used for the simultaneous LC tandem MS (MS/MS) determination of mycotoxins (deoxynivalenol, aflatoxins, ochratoxin A, T-2 and HT-2 toxins, fumonisins, and zearalenone) were tested in cereals and feed materials. They comprised different extraction solvents, types of cleanup [solid-phase extraction, QuEChERS, and immunoaffinity (IMA)], and calibration approaches (external or matrix-matched). The percentage of mycotoxins with acceptable recovery, according to Regulation (EC) No. 401/2006, ranged from 9 to 100%. The approach giving the highest percentage of acceptable results was selected and further tested for corn, rice, and feed spiked at three different mycotoxin levels (low, medium, and high). The method is based on extraction with MeOH–water (70 + 30, v/v) and cleanup with two multiantibody IMA columns. For corn and rice spiked at low mycotoxin levels, a significant matrix effect was observed and was compensated by using 13C calibration. At higher mycotoxin levels (medium and high), matrix effects were negligible as no significant differences were observed for the majority of recovery results calculated by 13C calibration and external calibration. Although the proposed method still needs improvement in terms of accuracy and, to a lesser extent, precision, it was successfully tested with four proficiency tests in buckwheat, corn, rice, and feed, giving acceptable z-scores for 97% (34 out of 35) of results.

Development of an LC-MS/MS Determination Method for T-2 Toxin and Its Glucoside and Acetyl Derivatives for Estimating the Contamination of Total T-2 Toxins in Staple Flours

Nakagawa, Hiroyuki1; Matsuo, Yosuke1; McCormick, Susan2; Lim, Chee Wei3

ABSTRACT

A determination method previously validated for trichothecenes and zearalenone by means of liquid chromatography-tandem mass spectrometry (LC-MS/MS) was adapted for the quantification of T-2 toxin (T-2) as well as its glucoside and acetyl derivatives, T-2-3-glucoside (T-2-3G) and 3-acetyl-T-2 (3A-T-2). HT-2 toxin (HT-2) and its acetyl derivative 3-acetyl-HT-2 (3A-HT-2) were also included as the target chemicals. Staple flours (56 samples collected from the Singapore market) were examined for contamination from T-2 and/or HT-2 and their derivatives. Among them, 16 flours were found to be contaminated with T-2 and/or HT-2, whereas none was contaminated with T-2-3G and 3A-HT-2, except for trace 3A-T-2 detected in 2 rye samples. Rye flour samples were frequently contaminated with both T-2 and HT-2. Some of the reference materials (RMs) were further analyzed, and T-2-3G and 3A-T-2 were quantitatively detected in corn and wheat RMs. The ratio of T-2-3G to T-2 in the RMs seemed to be much lower than the ratio of deoxynivalenol-3-glucoside to deoxynivalenol usually reported in former studies. To the best of our knowledge, the natural contamination of 3A-T-2 in staple flour is reported here for the first time.

Development and Interlaboratory Study of a Liquid Chromatography Tandem Mass Spectrometric Methodfor the Determination of Multiple Mycotoxins inCereals Using Stable Isotope Dilution

Ye, Jin1; Wu, Yu1; Guo, Qilei2; Lu, Meiling2; Wang, Songshan1; Xin, Yuanyuan1; Xie, Gang1; Zhang, Yan3; Mariappan, Meena4; Wang, Songxue1

ABSTRACT

An efficient, rapid, accurate, and cost-effective method based on stable isotope dilution and LC tandem MS was developed for the determination of multimycotoxins in cereals. The samples were extracted using acetonitrile–water–acetic acid (70 + 29 + 1, v/v/v), followed by dilution and centrifugation without any further cleanup. The mycotoxins were separated on a C18 column. Interference due to matrix effects was efficiently compensated for with [13C]-labeled stable isotope internal standards. The method demonstrated excellent linear relations, with regression coefficients above 0.999. Spiked recoveries at three different concentrations ranged from 80.9 to 115.9%, and RSDs were below 14% for all mycotoxins. The trueness of the method was also verified by participating in two proficiency tests, and satisfactory z-scores ($|z| < 1.1$) were obtained. In addition, an international interlaboratory study was organized to evaluate the methods. Eight laboratories characterized recovery, repeatability, and reproducibility studies in wheat, maize, and barley. The interlaboratory results were analyzed according to ISO 5725-2. Cochran and Grubbs tests were used to remove outliers. The mean recoveries of all 16 mycotoxins ranged from 87 to 111%. Repeatability, reproducibility, and Horwitz ratio values were 3.5–16.2, 5.4–33.6, and 0.16–1.65%, respectively. The results demonstrate that the method is reliable to determine multimycotoxins in cereals.

Interlaboratory Validation of a Stable Isotope Dilution and Liquid Chromatography Tandem Mass Spectrometry Method for the Determination of Aflatoxins in Milk, Milk-Based Infant Formula, and Feed

Zhang, Kai1; Liao, Chia-Ding2; Prakash, Shristi3; Conway, Michael3; Cheng, Hwei-Fang4

ABSTRACT

An interlaboratory study was conducted to evaluate stable isotope dilution and LC tandem MS (MS/MS) for the determination of aflatoxins B1, B2, G1, G2, and M1 (AFB1, AFB2, AFG1, AFG2, and AFM1) in milk, milk-based infant formula (formula), and feed. Samples were first fortified with five 13C uniformly labeled aflatoxins {[13C]–internal standard (IS)} corresponding to the five native aflatoxins, which were subsequently extracted with acetonitrile–water (50 + 50, v/v), followed by centrifugation, filtration, and LC-MS/MS analysis. In addition to certified milk powder and animal feed, the three participating laboratories also analyzed milk, formula, and feed fortified with the five aflatoxins at concentrations ranging from 0.5 to 50 ng/g. The majority of recoveries ranged from 80 to 120%, with RSDs < 20%. Method LOQs were determined by the three laboratories using the three sample matrixes in replicates (n = 8), and the determined LOQs of AFB1, AFB2, AFG1, AFG2, and AFM1 ranged from 0.1 to 0.91, 0.24 to 0.64, 0.28 to 1.52, 0.19 to 3.80, and 0.12 to 0.45 ng/g, respectively. For detected aflatoxins in the certified materials, all measured concentrations were within ±25% of the certified values. Using [13C]–IS eliminated the need for matrix-matched calibration standards for quantitation, simplified sample preparation, and achieved simultaneous identification and quantitation of the aflatoxins in a simple LC-MS/MS procedure.

Development and Validation of an Analytical Method Readily Applicable for Quality Control of Tabebuia impetiginosa (Taheebo) Ethanolic Extract

Jin, Yan1; Jeong, Kyung Min1; Lee, Jeongmi1; Zhao, Jing2; Choi, Su-Young3; Baek, Kwang-Soo3

ABSTRACT

The dried inner bark of Tabebuia impetiginosa, known as taheebo or red lapacho, has numerous beneficial effects on human health. This study presents the first simple and reliable quantitative method that could serve for the QC of taheebo. The method uses LC–UV spectroscopy to determine the veratric acid (VA; 3,4-dimethoxybenzoic acid) content of taheebo extracts (TEs). Sample preparation entailed the dissolution of TE in methanol (MeOH), facilitated by ultrasonic radiation for 10 min. The optimized conditions included chromatographic separation on an Agilent Eclipse Plus C18 column (4.6 × 150 mm, 5 µm) at 30°C. The mobile phase consisted of 1% acetic acid in water and MeOH, which was eluted under gradient mode at a flow rate of 1.0 mL/min. The detection wavelength was 254 nm. Using these conditions, VA was selectively resolved, and the entire chromatographic analysis time was 27 min. The method was linear in the range of 50–500 µg/mL ($r^2 = 0.9995$), precise (≤3.97% RSD), and accurate (97.10–102.72%). The validated method was applied to three batches of TE samples, yielding an estimated VA content range of 14.92–15.58 mg/g. Thus, the proposed method could serve as an easy and practical method for the QC of TEs or related products containing TEs.

Investigation of the Antifatigue Effects of Korean Ginseng on Professional Athletes by Gas Chromatography-Time-of-Flight-Mass Spectrometry-Based Metabolomics

Yan, Bei1; Liu, Yao1; Shi, Aixin1; Wang, Zhihong2; Aa, Jiye3; Huang, Xiaoping4; Liu, Yi4

ABSTRACT

Ginseng is usually used for alleviating fatigue. The purpose of this paper was to evaluate the regulatory effect of Korean ginseng on the metabolic pattern in professional athletes, and, further, to explore the underlying mechanism of the antifatigue effect of Korean ginseng. GC-time-of-flight-MS was used to profile serum samples from professional athletes before training and after 15 and 30 day training, and professional athletes administered with Korean ginseng in the meanwhile. Biochemical parameters of all athletes were also analyzed. For the athlete control group, strength–endurance training resulted in an elevation of creatine kinase (CK) and blood urea nitrogen (BUN), and a reduction in blood hemoglobin, and a dynamic trajectory of the metabolomic profile which were related to fatigue. Korean ginseng treatment not only lead to a marked reduction in CK and blood urea nitrogen (BUN) in serum, but also showed regulatory effects on the serum metabolic profile and restored scores plots close to normal, suggesting that the change in metabolic profiling could reflect the antifatigue effect of Korean ginseng. Furthermore, perturbed levels of 11 endogenous metabolites were regulated by Korean ginseng significantly, which might be primarily involved in lipid metabolism, energy balance, and chemical signaling. These findings suggest that metabolomics is a potential tool for the evaluation of the antifatigue effect of Korean ginseng and for the elucidation of its pharmacological mechanism.

Simultaneous Determination of Acetylsalicylic Acid, Hydrochlorothiazide, Enalapril, and Atorvastatin in a Polypill-Based Quaternary Mixture by TLC

Maślanka, Anna; Stolarczyk, Mariusz; Apola, Anna; Kwiecień, Anna; Hubicka, Urszula; Opoka, Włodzimierz

ABSTRACT

A new chromatographic-densitometric method has been developed for the qualitative and quantitative determination of the active ingredients in a simulated mixture corresponding to the PolyIran polypill, composed of acetylsalicylic acid, hydrochlorothiazide (HCT), enalapril (ENA), and atorvastatin (ATR), whose efficacy in the treatment and prevention of cardiovascular disease has been documented in clinical trials. Chromatographic separation was performed using TLC silica gel 60 plates with fluorescent indicator F254 as the stationary phase and a mixture of n-hexane–ethyl acetate–methanol–water–acetic acid (8.4 + 8 + 3 + 0.4 + 0.2, v/v/v/v/v) as the mobile phase. Densitometric measurements were carried out at λ = 210 nm when determining ENA and at λ = 265 nm in the case of the other drugs. Peaks of examined substances were well separated in the recorded chromatograms, enabling the evaluation of the results in terms of both qualitative and quantitative analysis. The method was specific for the analyzed components and was characterized by high sensitivity. The LOD was between 0.043 and 0.331 µg/spot, and LOQ was between 0.100 and 0.942 µg/spot. Recovery was in the range of 97.02–101.34%. The linearity range was broad and ranged from 0.600 to 6.000 µg/spot for acetylsalicylic acid, from 0.058 to 1.102 µg/spot for HCT, from 0.505 to 6.560 µg/spot for ENA, and from 0.100 to 1.000 µg/spot for ATR. The method was characterized by good precision, with RSD values that ranged from 0.10 to 2.26%.

Novel Stability-Indicating Chemometric-Assisted Spectrophotometric Methods for the Determination of Chlordiazepoxide and Clidinium Bromide in the Presence of Clidinium Bromide's Alkali-Induced Degradation Product

Nessim, Christine K.1; Michael, Adel M.1; Fayez, Yasmin M.2; Lotfy, Hayam M.3

ABSTRACT

Two simple and accurate chemometric-assisted spectrophotometric models were developed and validated for the simultaneous determination of chlordiazepoxide (CDZ) and clidinium bromide (CDB) in the presence of an alkali-induced degradation product of CDB in their pure and pharmaceutical formulation. Resolution was accomplished by using two multivariate calibration models, including principal component regression (PCR) and partial least-squares (PLS), applied to the UV spectra of the mixtures. Great improvement in the predictive abilities of these multivariate calibrations was observed. A calibration set was constructed and the best model used to predict the concentrations of the studied drugs. CDZ and CDB were analyzed with mean accuracies of 99.84 ± 1.41 and 99.81 ± 0.89% for CDZ and 99.56 ± 1.43 and 99.44 ± 1.41% for CDB using PLS and PCR models, respectively. The proposed models were validated and applied for the analysis of a commercial formulation and laboratory-prepared mixtures. The developed models were statistically compared with those of the official and reported methods with no significant differences observed. The models can be used for the routine analysis of both drugs in QC laboratories.

A New Platform for Profiling Degradation-Related Impurities via Exploiting the Opportunities Offered by Ion-Selective Electrodes: Determination of Both Diatrizoate Sodium and Its Cytotoxic Degradation Product

Riad, Safaa M.1; Abd El-Rahman, Mohamed K.2; Fawaz, Esraa M.2; Shehata, Mostafa A.2

ABSTRACT

Although the ultimate goal of administering active pharmaceutical ingredients (APIs) is to save countless lives, the presence of impurities and/or degradation products in APIs or formulations may cause harmful physiological effects. Today, impurity profiling (i.e., the identity as well as the quantity of impurity in a pharmaceutical) is receiving critical attention from regulatory authorities. Despite the predominant use of spectroscopic and chromatographic methods over electrochemical methods for impurity profiling of APIs, this work investigates the opportunities offered by electroanalytical methods, particularly, ion-selective electrodes (ISEs), for profiling degradation-related impurities (DRIs) compared with conventional spectroscopic and chromatographic methods. For a meaningful comparison, diatrizoate sodium (DTA) was chosen as the anionic X-ray contrast agent based on its susceptibility to deacetylation into its cytotoxic and mutagenic degradation product, 3,5-diamino-2,4,6 triiodobenzoic acid (DTB). This cationic diamino compound can be also detected as an impurity in the final product because it is used as a synthetic precursor for the synthesis of DTA. In this study, four novel sensitive and selective sensors for the determination of both DTA and its cytotoxic degradation products are presented. Sensors I and II were developed for the determination of the anionic drug, DTA, and sensors III and IV were developed for the determination of the cationic cytotoxic impurity. The use of these novel sensors not only provides a stability-indicating method for the selective determination of DTA in the presence of its degradation product, but also permits DRI profiling. Moreover, a great advantage of these proposed ISE systems is their higher sensitivity for the quantification of DTB relative to other spectroscopic and chromatographic methods, so it can measure trace amounts of DTB impurities in DTA bulk powder and pharmaceutical formulation without a need for preliminary separation.

Determination of Total Steroid Saponins in Different Species of Paris Using FTIR Combined with Chemometrics

Yang, Yuangui; Jin, Hang; Zhang, Jinyu; Wang, Yuanzhong

ABSTRACT

The saponins of Paris spp. have antimicrobial, immune-stimulating, and antitumor biological properties. In this investigation, FTIR and ultra-HPLC (UHPLC) were used for the determination of total steroid saponins in different species of Paris from Yunnan Province, China. A 52-sample calibration set and a 26-sample validation set for partial least-squares regression (PLSR) and support vector machine regression (SVMR) combined with FTIR and UHPLC were investigated. The optimal parameters C and γ were screened by a grid search with a sevenfold cross-validation. The results indicate that pretreatment with the combination of standard normal variate, second derivative, and orthogonal signal correction had the best performance. When comparing the SVMR and PLSR models, linear PLSR had better performance than nonlinear SVMR for the determination of total steroid saponins in different species of Paris. The highest total saponin content was found in P. axialis from Baoshan City (40.92 ± 9.06 mg/g). These results demonstrate that this approach would provide a fast and robust strategy for the QC of Paris in further analyses.

Evaluation of mericon E. coli O157 Screen plus and mericon E. coli STEC O-Type Pathogen Detection Assays in Select Foods: Collaborative Study, First Action 2017.05

Bird, Patrick1; Benzinger, M. Joseph1; Bastin, Benjamin1; Crowley, Erin1; Agin, James1; Goins, David1; Armstrong, Marcia2

ABSTRACT

QIAGEN mericon Escherichia coli O157 Screen Plus and mericon E. coli Shiga toxin-producing E. coli (STEC) O-Type Pathogen Detection Assays use Real-Time PCR technology for the rapid, accurate detection of E. coli O157 and the “big six” (O26, O45, O103, O111, O121, O145) (non-O157 STEC) in select food types. Using a paired study design, the assays were compared with the U.S. Department of Agriculture, Food Safety Inspection Service Microbiology Laboratory Guidebook Chapter 5.09 reference method for the detection of E. coli O157:H7 in raw ground beef. Both mericon assays were evaluated using the manual and an automated DNA extraction method. Thirteen technicians from five laboratories located within the continental United States participated in the collaborative study. Three levels of contamination were evaluated. Statistical analysis was conducted according to the probability of detection (POD) statistical model. Results obtained for the low-inoculum level test portions produced a difference between laboratories POD (dLPOD) value with a 95% confidence interval of 0.00 (–0.12, 0.12) for the mericon E. coli O157 Screen Plus with manual and automated extraction and mericon E. coli STEC O-Type with manual extraction and –0.01 (–0.13, 0.10) for the mericon E. coli STEC O-Type with automated extraction. The dLPOD results indicate equivalence between the candidate methods and the reference method.

Application of MALDI-TOF MS Systems in the Rapid Identification of Campylobacter spp. of Public Health Importance

Hsieh, Ying-Hsin1; Wang, Yun F.2; Moura, Hercules3; Miranda, Nancy1; Simpson, Steven1; Gowrishankar, Ramnath3; Barr, John3; Kerdahi, Khalil1; Sulaiman, Irshad M.1

ABSTRACT

Campylobacteriosis is an infectious gastrointestinal disease caused by Campylobacter spp. In most cases, it is either underdiagnosed or underreported due to poor diagnostics and limited databases. Several DNA-based molecular diagnostic techniques, including 16S ribosomal RNA (rRNA) sequence typing, have been widely used in the species identification of Campylobacter. Nevertheless, these assays are time-consuming and require a high quality of bacterial DNA. Matrix-assisted laser desorption ionization (MALDI) time-of-flight (TOF) MS is an emerging diagnostic technology that can provide the rapid identification of microorganisms by using their intact cells without extraction or purification. In this study, we analyzed 24 American Type Culture Collection reference isolates of 16 Campylobacter spp. and five unknown clinical bacterial isolates for rapid identification utilizing two commercially available MADI-TOF MS platforms, namely the bioMérieux VITEK® MS and Bruker Biotyper systems. In addition, 16S rRNA sequencing was performed to confirm the species-level identification of the unknown clinical isolates. Both MALDI-TOF MS systems identified the isolates of C. jejuni, C. coli, C. lari, and C. fetus. The results of this study suggest that the MALDI-TOF MS technique can be used in the identification of Campylobacter spp. of public health importance.

MC-Media Pad ACplus™ for Enumeration of Aerobic Counts in a Variety of Foods

Teramura, Hajime1; Betts, Gail2

ABSTRACT

The MC-Media Pad ACplus™ is a dry, rehydratable film medium for the enumeration of aerobic bacterial colonies. The performance of the method in a variety of foods was compared to that of U.S. reference methods: U.S. Department of Agriculture (USDA) Food Safety and Inspection Service (FSIS) Microbiology Laboratory Guidebook (MLG) Chapter 3.02 "Quantitative Analysis of Bacteria in Foods as Sanitary Indicators" (USDA/FSIS MLG 3.02); Standard Methods for the Examination of Dairy Products (SMEDP) Chapter 6 "Microbiological Count Methods, Standard Plate Count Method" (SMEDP 6); AOAC Official Method SM 966.23 Microbiological Methods; and ISO 4833-1:2013 "Microbiology of the food chain—Horizontal method for the enumeration of microorganisms—Part 1: Colony count at 30 degrees C by the pour plate technique." The validated matrixes included raw chicken breast and raw ground pork for USDA/FSIS MLG 3.02; cream cheese and yogurt drink for SMEDP 6; parsley, vegetable juice, prawns, tuna pate, sandwiches, and pasta salad for AOAC Method 966.23, and raw chicken breast, raw ground pork, cream cheese, yogurt drink, parsley, vegetable juice, prawns, tuna pate, sandwiches, and pasta salad for ISO 4833-1:2013. In each matrix study, five replicates at each of three contamination levels were tested as paired test portions. All 10 matrixes were compared to the appropriate U.S. reference methods under MC-Media Pad ACplus standard-usage conditions (35 ± 1°C for 48 ± 2 h). Across all matrixes, the difference of mean log10 values ranged from –0.43 to 0.44, within the acceptable range of –0.50 to 0.50. The candidate method repeatability SD (sr) varied from 0.03 to 0.23 log10 CFU/g, comparing favorably to the reference method SD, which ranged from 0.06 to 0.30 log10 CFU/g. Seven matrixes were compared to the appropriate U.S. reference methods under MC-Media Pad ACplus rapid-usage conditions (35 ± 1°C for 24 ± 2 h). Of the 21 matrix/concentration combinations, only three instances of difference of mean >0.5 log were observed. The ranges of sr values of the rapid-usage candidate method (0.023–0.324) and the reference method (0.013–0.236) were similar for the seven matrixes tested. All 10 matrixes were compared to the International Organization for Standardization (ISO) reference method under MC-Media Pad ACplus alternate-method conditions (30 ± 1°C for 72 ± 3 h). All 10 matrixes yielded a mean difference between methods of <0.5 log, and the ranges of sr values were similar between the candidate alternate method (0.037–0.378) and the ISO reference method (0.037–0.437). The product consistency study demonstrated no significant difference between lots of product and supported the 2-year shelf life. Robustness testing yielded no significant differences when small variations were made in sample volume, incubation temperature, and incubation time. Thus, the data show equivalent or better performance of the MC-Media Pad ACplus method compared to the relevant reference methods in support of AOAC Performance Tested Method SM certification.

Validation Study of MaxSignal® Histamine Enzymatic Assay for the Detection of Histamine in Fish/Seafood

Gone, Swapna; Kosa, Nicolas; Krebs, Joseph

ABSTRACT

Bioo Scientific Corp. has developed a rapid enzymatic quantitative assay for the determination of histamine in seafood. Fresh/frozen tuna, canned tuna, pouched tuna, and frozen mahi mahi samples were used for the validation study under the specific guidelines of the AOAC Research Institute Performance Tested Methods SM program. Recoveries ranged from 82 to 107% at concentrations ranging from 6 to 72 ppm, with RSDr values between 0.8 and 6.5% (6–72 ppm). The linearity of the assay ranged from 0 to 108 ppm, with R2 values exceeding 0.99. The LOD was 0.9 ppm and the LOQ was 2.6 ppm for frozen tuna, which gave the lowest background level of contaminant. Cross-reactivity of the assay was tested against 14 other biogenic amines and was found to be minimal for all (<0.5%), except for agmatine (4.1%) and putrescine (0.9%). There was no observable interference from any tested biogenic amines. Product consistency was verified by validating lot-to-lot variations and variations within the same lot. Overall recoveries for all tested matrixes were within the acceptable range (80–120%). A 1-year claimed shelf life of the kit at 4°C was verified by accelerated stability study data collected on days 1, 15, and 32 at 25°C and by real-time stability testing at 1-month, 6-month, and 1-year at 4°C. No difference in histamine detection was observed in ruggedness testing, in which minor changes were introduced to the assay protocol. Good agreement was observed between AOAC Official Method SM 977.13 and the MaxSignal® Histamine Enzymatic Assay method. Independent laboratory testing demonstrated that the MaxSignal method works with the same precision in the hands of minimally trained technicians as with the expert method developers. This study validates the performance of Bioo Scientific's rapid enzymatic method.

Development and Method Validation of Analysis of Urushiol in Sumac and Food Ingredients in Korea

Seo, Dongwon1; Kim, Kijin2; Choi, Hee-Don2; Yoo, Miyoung2; Ha, Jaeho3; Lee, Kwang-Won4

ABSTRACT

This study developed an analytical method to determine the urushiol content in sap and several foods. The full process for urushiol analysis consists of extraction, trimethylsilyl silylation, analysis, and identification via GC-MS, with each step optimized to attain the required accuracy and precision. Urushiol was separated from sap via liquid–liquid extraction and was derivatized via silylation. The components were analyzed using a polar capillary column and identified using GC-MS. The deviations of relative retention times and retention time windows were within 0.001 and 0.02 min, which satisfied the criteria of 0.06 and 0.03 min, respectively. The response of the urushiol standards tested was found to be linear in the investigated concentration range, with a correlation coefficient of 0.998. The LODs were between 1.74 and 2.67 μg/mL.

Development and Evaluation of an Enzyme-Linked Immunosorbent Assay Using a Nonpoisonous Extraction System for the Determination of Crustacean Protein in Processed Foods

Koizumi, Daisuke1; Shirota, Kazuya1; Oda, Hiroshi1; Adachi, Reiko2; Sakai, Shinobu2; Akiyama, Hiroshi2; Nishimaki-Mogami, Tomoko2; Teshima, Reiko2

ABSTRACT

Crustacean proteins are food allergens that cause severe allergic reactions in patients with food allergies; therefore, the identification of crustaceans such as shrimp, crab, and lobster as ingredients in processed food products is mandatory in Japan. We previously developed and validated an ELISA method coupled with an extraction process using the surfactant sodium dodecyl sulfate and the reductant 2-mercaptoethanol (2-ME) to quantify crustacean protein. However, 2-ME was designated as poisonous in Japan in 2008. Therefore, in this study, we developed and evaluated an ELISA method for detecting and quantifying crustacean protein that uses sodium sulfite (Na_2SO_3) in place of 2-ME for extraction. The proposed ELISA method showed high sensitivity, with an LOQ of 0.66 µg protein/g food sample. Furthermore, the proposed method showed high specificity for the Decapoda order within the subphylum Crustacea, with recoveries ranging from 83.8 to 100.8% for model processed foods, as well as high reproducibility (intra- and interassay CVs of ≤8.2%) and high correlation with our previously validated ELISA method for processed foods (correlation coefficient of 0.996). The proposed ELISA method does not require the use of poisonous reagents, provides acceptable accuracy, and is useful for the routine monitoring of food products.

Single-Laboratory Validation for Determination of Total Soluble Proanthocyanidins in Cranberry Using 4-Dimethylaminocinnamaldehyde

Sintara, Marsha1; Li, Lin1; Cunningham, David G.2; Prior, Ronald L.3; Wu, Xianli4; Chang, Tony5

ABSTRACT

American cranberry (Vaccinium macrocarpon) is native to Eastern North America. Recent studies have suggested that the A-type proanthocyanidins (PACs) in cranberries are effective in preventing urinary tract infection. To meet the growing interest in the cranberry market, an accurate, reliable, and simple method to determine PAC concentration is needed. In this study, a modified method using 4-dimethylaminocinnamaldehyde to quantify total PACs in cranberry products was validated. Cranberry juice extract powder, cranberry capsules containing juice extract, and cranberry juice concentrate were used as the samples in this study. With the modified method, the calibration curves for proanthocyanidin A2 had correlation coefficients (r^2) of >0.99. The recoveries of two different concentrations after spiking were 97.1 and 99.1%, and the RSDs for repeatability and reproducibility were <2.7 and <1.6%, respectively.

Quantitative Detection of Pork Contamination in Cooked Meat Products by ELISA

Thienes, Cortlandt P.1; Masiri, Jongkit1; Benoit, Lora A.2; Barrios-Lopez, Brianda1; Samuel, Santosh A.1; Cox, David P.1; Dobritsa, Anatoly P.1; Nadala, Cesar1; Samadpour, Mansour3

ABSTRACT

Recent news of many cases of adulteration of meats with pork has bolstered the need for a way to detect and quantify the unwanted contamination of pork in other meats. To address this need, Microbiologique, Inc. has produced a sandwich ELISA assay that can rapidly quantify the presence of pork in cooked horse, beef, chicken, goat, and lamb meats. We carried out a validation study and showed that this assay has an analytical sensitivity of 0.00014 and 0.00040% (w/v) for cooked and autoclaved pork, respectively, and an analytical range of quantitation of 0.05–3.2% (w/v) in the absence of other meats. The assay can measure pork contamination down to 0.1% (w/w) in the presence of cooked horse, beef, chicken, goat, and lamb meats. The assay is quick and can be completed in 1 h and 10 min.

HPLC Determination of Total Tryptophan in Infant Formula and Adult/Pediatric Nutritional Formula Following Enzymatic Hydrolysis: Single-Laboratory Validation, First Action 2017.03

Draher, Jonathan; White, Norman

ABSTRACT

A method for tryptophan (Trp) analysis designed to comply with AOAC Standard Method Performance Requirement 2014.013 is described. Unlike AOAC 988.15, which uses alkaline hydrolysis, this method uses enzymatic hydrolysis to release the Trp from the intact protein. The method achieves an LOQ of 0.18 mg/100 g Trp on a ready-to-feed basis with mean recoveries ranging from 93.8 to 104.9%. Repeatability ranged from 0.2 to 5.0%. Intermediate precision ranged from 1.0 to 6.9%. The analytical range was determined to be 0.18–300 mg/100 g, with linearity over eight calibration standard levels giving an average deviation from theoretical levels of 0.3%. No single calibration point had a deviation of >5.0%. Two standard reference materials (SRMs 1849a and 927e) were analyzed, and the average deviation from the certified value was 98.5% for SRM 1849a and 101.2% for SRM 927e. Sample preparation is very similar to existing methods in terms of time and complexity. The use of an internal standard reduces laboratory error and allows for reproducible results.

Determination of Total Biotin by Liquid Chromatography Coupled with Immunoaffinity Column Cleanup Extraction: Multilaboratory Testing, Final Action 2016.02

Joseph, George1; Devi, Ranjani1; Marley, Elaine C.2; Leeman, David2

ABSTRACT

Single- and multilaboratory testing data have provided systematic scientific evidence that a simple, selective, accurate, and precise method can be used as a potential candidate reference method for dispute resolution in determining total biotin in all forms of infant, adult, and/or pediatric formula. Using LC coupled with immunoaffinity column cleanup extraction, the method fully meets the intended purpose and applicability statement in AOAC Standard Method Performance Requirement 2014.005. The method was applied to a cross-section of infant formula and adult nutritional matrixes, and acceptable precision and accuracy were established. The analytical platform is inexpensive, and the method can be used in almost any laboratory worldwide with basic facilities. The immunoaffinity column cleanup extraction is the key step to successful analysis.

Development of an Accurate and Sensitive Analytical Method for the Determination of Cadmium at Trace Levels Using Dispersive Liquid–Liquid Microextraction Based on the Solidification of Floating Organic Drops Combined with Slotted Quartz Tube Flame Atomic Absorption Spectrometry

Aydin, Ilgin; Chormey, Dotse Selali; Budak, Türkan; Fırat, Merve; Turak, Fatma; Bakirdere, Sezgin

ABSTRACT

A dispersive liquid–liquid microextraction (DLLME) technique based on a solidification-of-floating-organic-drop (SFOD) procedure was developed for the determination of trace amounts of cadmium (Cd) by using a flame atomic absorption spectrometer (FAAS) fitted with a slotted quartz tube (SQT). The extraction of Cd was achieved by forming a complex with diphenylcarbazone. Parameters affecting the formation of complex and extraction outputs were carefully optimized to obtain high-absorbance signals to achieve lower LODs. An SQT was fitted on top of the flame burner head to further enhance the absorbance of the signals recorded by the FAAS. Coupling the DLLME-SFOD procedure with SQT-FAAS produced an enhancement factor of about 183. The LOD of the method was 0.23 µg/L with an RSD of 3.8%. Matrix-matching was used to overcome any low recovery results obtained with tap water and municipal wastewater.

Determination and Safety Assessment of Residual Spirotetramat and Its Metabolites in Amaranth (Amaranthus tricolor) and Soil by Liquid Chromatography Triple-Quadrupole Tandem Mass Spectrometry

Chen, Xiao-Jun; Meng, Zhi-Yuan; Ren, Li; Song, Yue-Yi; Ren, Ya-Jun; Chen, Jian-Shu; Guan, Ling-Jun

ABSTRACT

With the purpose of guaranteeing the safe use of spirotetramat and preventing its potential health threats to consumers, a QuEChERS extraction method coupled with LC triple-quadrupole tandem MS was applied in this study to determine residual spirotetramat metabolites in different tissues of amaranth (Amaranthus tricolor) and in soil. The results indicate that the spirotetramat degraded into different types of metabolites that were located in different tissues of amaranth and in soil. B-keto, B-glu, and B-enol were the three most representative degradation products in the leaf of amaranth, and B-glu and B-enol were the two major degradation products found in the stem of amaranth; however, only B-enol was detected in the root of amaranth. B-keto and B-mono were the two products detected in the soil in which the amaranth grew. The cytotoxicity results demonstrate that spirotetramat and its metabolite B-enol inhibited cellular growth, and the toxicity of spirotetramat and its metabolite B-enol exceeded than that of the metabolites B-keto, B-mono, and B-glu. This investigation is of great significance to the safe use of spirotetramat in agriculture.

Vortex-Assisted Modified Dispersive Liquid–Liquid Microextraction of Trace Levels of Cadmium in Surface Water and Groundwater Samples of Tharparkar, Pakistan, Optimized by Multivariate Technique

Nizamani, Sooraj; Kazi, Tasneem G.; Afridi, Hassan I.; Talpur, Sehrish; Lashari, Ayaz; Lashari, Anjum; Ali, Jamshed

ABSTRACT

A simple vortex-assisted modified dispersive liquid–liquid microextraction procedure is proposed for the enrichment of cadmium (Cd+2) in surface (stored rainwater) and groundwater of the Tharparkar district in Pakistan, before analysis by flame atomic absorption spectrometry. Ammonium pyrrolidinedithiocarbamate was used as a ligand to make a hydrophobic complex of Cd+2, which was extracted in an ionic liquid (1-butyl-3-methylimidazolium hexafluorophosphate), and the nonionic surfactant Triton X-114 was applied as a dispersing medium. The contents of tubes were shaken for different time intervals on a vortex mixer to enhance extraction efficiency. A multivariate strategy was used to simultaneously evaluate seven factors including, concentration of the complexing reagent, pH, amounts of ionic liquid and Triton X-114, vortex shaking time, centrifugation time and extracting solution for their influence on the percentage recovery of the analyte. The important variables were further optimized by central composite design. The preconcentration factor and LOD were observed as 76.9 and 0.048 µg/L, respectively. The Certified Reference Material SRM1643e was used to check the validity of the developed method, and the RSD was found to be 4.02%. The proposed technique was successfully applied for the enrichment of Cd+2 in groundwater and surface water samples from the southeastern part of Pakistan. The observed results revealed that the concentration of Cd+2 in groundwater was higher than the World Health Organization recommended value of 3 µg/L for drinking water. For adults weighing approximately 60 kg, consumption of groundwater for drinking and other domestic purposes would provide levels of Cd+2 that are 2- to 3-fold higher than the provisional maximum tolerable daily intake.

Novel Two-Stage Fine Milling Enables High-Throughput Determination of Glyphosate Residues in Raw Agricultural Commodities

Riter, Leah S.; Wujcik, Chad E.

ABSTRACT

Dramatic process-efficiency gains for residue analysis of glyphosate in raw agricultural commodities (RACs) were achieved by development and validation of a two-stage fine-milling process. This secondary milling produced a uniform and consistent product that could be reproducibly measured with 75 mg analytical test portions. The milligram scale sample size enabled the direct weighing of sample into a liquid-handler-compatible 96-well format. A high-throughput workflow based on this innovative comminution approach for the quantitation of glyphosate, a nonselective herbicide, and its main degradation product, aminomethylphosphonic acid, was validated in various RACs and used to demonstrate the applicability of the two-step milling process. The precision and reproducibility of 75 mg analytical portions taken through this assay was used to demonstrate the feasibility of using a two-stage fine-milling technique for pesticide residue applications. An RSD of less than 10% was achieved in endogenous glyphosate residues in multiple RACs. Comparable recoveries and superior precision were achieved with this new method as compared with a validated 10 g scale method.

Comparison of Spectrophotometric Methods for the Determination of Copper in Sugar Cane Spirit

Soares, Sarah Adriana R.1; Costa, Silvânio Silvério L.2; Araujo, Rennan Geovanny O.3; Teixeira, Leonardo Sena Gomes3; Dantas, Alailson Falcão3

ABSTRACT

Three spectrophotometric methods were developed for the determination of copper (Cu) in sugar cane spirit using the chromogenic reagents neocuproine, cuprizone, and bathocuproine. Experimental conditions, such as reagent concentration, reducer concentration, pH, buffer concentration, the order of addition of reagents, and the stability of the complexes, were optimized. The work range was established from 1.0 to 10.0 μg/mL, with correlation coefficients of >0.999 for all three optimized methods. The methods were evaluated regarding accuracy by addition and recovery tests at five concentration levels, and the obtained recoveries ranged from 91 to 105% (n = 3). Precision was expressed as RSD (relative standard deviation), with values ranging from 0.01 to 0.17% (n = 10). The method using the chromogenic reagent cuprizone presented the greatest molar absorptivity, followed by bathocuproine and neocuproine. The methods were applied for the determination of Cu in sugar cane spirit, and the results were compared with a reference method by flame atomic absorption spectrometry (FAAS). Calibration curve solutions for FAAS analysis were prepared in a 40% (v/v) alcohol medium in a range of concentrations from 0.5 up to 5 μg/mL. Measurements for Cu determination were carried out at a wavelength of 324.7 nm. The concentrations obtained for Cu in sugar cane spirit samples from Brazil were between 1.99 and 12.63 μg/mL, and about 75% of the samples presented Cu concentrations above the limit established by Brazilian legislation (5.0 μg/mL or 5.0 mg/L).

Ultrasonically Dispersed Ionic Liquid-Based Microextraction of Lead in Biological Samples of Malnourished Children Prior to Analysis by Flame Atomic Absorption Spectrometry

Talpur, Sehrish; Kazi, Tasneem Gul; Afridi, Hassan Imran; Talpur, Farah Naz; Nizamani, Sooraj; Lashari, Anjum; Akhtar, Asma; Khan, Mustafa

ABSTRACT

In the present study, a simple ultrasonically dispersed modified liquid-phase microextraction method was developed for the extraction of lead (Pb) from blood and scalp hair samples of malnourished children (MNC). The complexation of Pb was executed by means of the complexing agent, ammonium pyrrolidinedithiocarbamate (APDC), whereas extraction was carried out through the ionic liquid (IL), 1-butyl-3-methylimidazolium hexafluorophosphate. Ultrasound energy was used for the dispersion and extraction of the metal complex into an IL because it enhances the extraction of the metal complex into infinite IL drops at a temperature range of 40–80°C for 1–5 min. After sonication, the enriched analyte phase was separated by centrifugation. Nitric acid (HNO3; 0.5–2 mol/L) was added to the IL-enriched phase to back-extract the analyte into the acidic aqueous phase and analyzed by flame atomic absorption spectrometry. Various experimental parameters that affect the efficiency of the proposed method, such as volume of IL, concentration of the complexing agent, pH, ultrasonication time and temperature, and concentration of HNO3, were optimized. The enhancement factor was calculated as 70. The LOD for Pb ions was found to be 0.19 μg/L, with an RSD of <5%. Accuracy was ensured by applying the procedure to a certified reference material for whole blood and scalp hair. The developed procedure was successfully applied for the analysis of the concentration of Pb ion in whole-blood and scalp hair samples of MNC from different areas of Sindh, Pakistan. The concentration of Pb among MNC was 2-fold higher than the referent.

Determination of Monensin in Bovine Tissues: A Bridging Study Comparing the Bioautographic Method (FSIS CLG-MON) with a Liquid Chromatography-Tandem Mass Spectrometry Method (OMA 2011.24)

Mizinga, Kemmy M.1; Burnett, Thomas J.1; Brunelle, Sharon L.2; Wallace, Michael A.3; Coleman, Mark R.4

ABSTRACT

The U.S. Department of Agriculture, Food Safety Inspection Service regulatory method for monensin, *Chemistry Laboratory Guidebook* CLG-MON, is a semiquantitative bioautographic method adopted in 1991. *Official Method of Analysis* SM (OMA) **2011.24**, a modern quantitative and confirmatory LC-tandem MS method, uses no chlorinated solvents and has several advantages, including ease of use, ready availability of reagents and materials, shorter run-time, and higher throughput than CLG-MON. Therefore, a bridging study was conducted to support the replacement of method CLG-MON with OMA **2011.24** for regulatory use. Using fortified bovine tissue samples, CLG-MON yielded accuracies of 80–120% in 44 of the 56 samples tested (one sample had no result, six samples had accuracies of >120%, and five samples had accuracies of 40–160%), but the semiquantitative nature of CLG-MON prevented assessment of precision, whereas OMA **2011.24** had accuracies of 88–110% and RSD_r of 0.00–15.6%. Incurred residue results corroborated these results, demonstrating improved accuracy (83.3–114%) and good precision (RSD_r of 2.6–20.5%) for OMA **2011.24** compared with CLG-MON (accuracy generally within 80–150%, with exceptions). Furthermore, χ^2 analysis revealed no statistically significant difference between the two methods. Thus, the microbiological activity of monensin correlated with the determination of monensin A in bovine tissues, and OMA **2011.24** provided improved accuracy and precision over CLG-MON.

Quantitation and Confirmation of Chloramphenicol, Florfenicol, and Nitrofuran Metabolites in Honey Using LC-MS/MS

Veach, Brian T.; Anglin, Renea; Mudalige, Thilak K.; Barnes, Paula J.

ABSTRACT

This paper describes a rapid and robust method utilizing a single liquid–liquid extraction for the quantitation and confirmation of chloramphenicol, florfenicol, and nitrofuran metabolites in honey. This methodology combines two previous extraction methods into a single extraction procedure and utilizes matrix-matched calibration standards and stable isotopically labeled standards to improve quantitation. The combined extraction procedure reduces the average extraction time by >50% when compared with previously used procedures. The drug residues were determined using two separate LC-tandem MS conditions. Validation of all the analytes was performed, with average quantitation ranging from 92 to 105% for all analytes and the RSDs for all analytes being ≤12%.

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Biennial Review of Planar Chromatography: 2015–2017

Sherma, Joseph

ABSTRACT

The most important advances in planar chromatography published between November 1, 2015, and November 1, 2017, are reviewed in this paper. Included are an introduction to the current status of the field; student experiments and reviews; apparatus and techniques for sample preparation and TLC separations; detection and identification of separated zones; quantitative analysis; preparative layer chromatography; and thin-layer radiochromatography. Selected applications are given in the various sections of the review.

Antibiotic Resistance in Escherichia coli from Farm Livestock and Related Analytical Methods: A Review

Caruso, Giorgia

ABSTRACT

The indiscriminate use of antibiotics for the treatment of human and animal infections has led to the rise of resistance in pathogens and in commensal bacteria. In particular, farm animals may act as vectors for the dissemination of drug-resistant genes because of the intensive use of antibiotics in animal production, enabling resistance to a wide range of antimicrobial agents, including those normally used in human medicine. Escherichia coli, being a widespread commensal, is considered a good indicator of antibiotic use. Ultimately, it is emerging as a global threat, developing dramatically high levels of antibiotic resistance to multiple classes of drugs. Its prevalence in food animals is hence alarming, and more studies are needed in order to ascertain the spread dynamics between the food chain and humans. In this context, great attention should be paid to the accurate detection of resistance by conventional and molecular methods. In this review, a comprehensive list of the most widely used testing methods is also addressed.

Food Crises and Food Safety Incidents in European Union, United States, and Maghreb Area: Current Risk Communication Strategies and New Approaches

Chammem, Nadia1; Issaoui, Manel2; De Almeida, Ana Isabel Dâmaso3; Delgado, Amélia Martins4

ABSTRACT

Globalization has created a dynamic market, which has dramatically intensified interchanges of goods and information as well as the flow of people among nations. This international phenomenon offers the consumer a choice between a wide variety of foods from diverse locations. However, there are challenges to improving food security and safety on a global scale; the major question is how food safety can be guaranteed while increasing the complexity of food supply chains. A food produced in a certain location usually contains ingredients, additives, and preservatives from different and distant origins. Although countries take several food control measures, their institutional and regulatory frameworks diverge widely, as do the definitions of food crisis, food incidents, and risk management approaches. The present review discusses some past food safety issues and lessons learned. Convergences and differences in the regulatory framework of food control agencies in different regions of the world are herein revealed. Emerging risks are also discussed, particularly the spread of antibiotic resistance in the food chain and the environment, as well as the rise of new antibiotic-resistant pathogenic strains with broader tolerance to environmental factors.

Vitamin D Dietary Supplementation: Relationship with Chronic Heart Failure

Dattilo, Giuseppe1; Casale, Matteo1; Avventuroso, Emanuela2; Laganà, Pasqualina2

ABSTRACT

It is estimated that over 1 billion people worldwide have a deficiency of vitamin D, also known as hypovitaminosis D, which the World Health Organization has defined as a public health problem. Beyond its historical homeostasis regulatory function of calcium and phosphorus, in relation to the preservation of the skeletal system, several studies show today a close connection between hypovitaminosis D and the genesis of rheumatic, autoimmune, neoplastic, and cardiovascular diseases. With exclusive reference to cardiovascular aspects, multiple heart diseases such as hypertension, myocardial ischemia, and heart failures might have deficiency in vitamin D as an important causative factor. Because of the influence of concomitant pathologies caused by antibiotic-resistant agents, the function of this vitamin should be critically evaluated. However, the role of vitamin D remains to be established; only a few studies have tested the effects of its supplementation in patients with chronic heart failure diseases, and reported results are unclear. It is important to implement studies in this field in order to assess the real benefits induced by vitamin D supplementation in cardiovascular patients and, in particular, in patients with heart failure. Should the research confirm actual clinical improvement after treatment with vitamin D, such a supplementation might represent a new low-cost therapeutic approach to improving quality of life.

Antimicrobial Substances for Food Packaging Products: The Current Situation

Pellerito, Alessandra1; Ameen, Sara M.2; Micali, Maria3; Caruso, Giorgia4

ABSTRACT

Antimicrobial substances are widely used in many anthropic activities, including sanitary and military services for the human population. These compounds are also known to be used in food production, agricultural activities, and partially correlated industrial sectors. However, there are concerns regarding the link between the abuse of antimicrobial agents in these ambits and the possible detection of antibiotic-resistant microorganisms. Modern food and beverage products are generally found on the market as prepackaged units, with several exceptions. Consequently, positive and negative features of a specific food or beverage should be considered as the result of the synergic action of different components, including the container (or the assembled sum of packaging materials). At present, the meaning of food container also includes the creation and development of new packaging materials that are potentially able to interact with the contained food. "Active" packaging systems can be realized with antimicrobial substances. On the other hand, a careful evaluation of risks and advantages correlated with antimicrobial agents is needed because of possible negative and/or unexpected failures.

Studies on Antimicrobial Activity and Kinetics of Inhibition by Plant Products in India (1990–2016)

Sharma, Ramesh Kumar1; Rana, Bhupendra Kumar2

ABSTRACT

The antimicrobial activity of herbal extracts or plant isolates has usually been evaluated in India using different antimicrobial susceptibility testing methods generally based on diffusion and dilution. There are different analytical approaches for the reliable evaluation of antimicrobial activity ascribed to medicinal plants against selected pathogenic microorganisms. Obtained results may provide scientific bases for the selective use of these natural plants as healing drugs, crop-protecting pesticides, or shelf-life-extending solutions. In general, antimicrobial susceptibility methodologies involve in vivo and in vitro studies; at present, the in vitro evaluation of antibacterial activity appears more popular. Diffusion methods have some limitations, although they are extensively used to determine the susceptibility of organisms isolated from specimen samples to applied antimicrobials and vice versa. Dilution methods are preferred in the case of more precise antimicrobial activity estimation, in terms of minimum inhibitory concentration. With regard to the inherent antimicrobial nature of herbal compositions, herbs, and herbal extracts, Indian researchers have evaluated the reliability of these antimicrobial agents against selected pathogens and have shown them to be effective. Researchers have also tried to establish linear regression correlation analyses on the basis of available inhibition results. This research is still evolving, and interesting results may be expected in the future.

Antimicrobial Potential of Sicilian Honeys against Staphylococcus aureus and Pseudomonas aeruginosa

Coniglio, Maria Anna1; Laganà, Pasqualina2; Faro, Giuseppina1; Marranzano, Marina1

ABSTRACT

The purpose of this study was to investigate the antibacterial effect of 71 organic Sicilian honeys of different botanical origins against Staphylococcus aureus [American Type Culture Collection (ATCC) 9144™] and Pseudomonas aeruginosa (ATCC 27853™). The antimicrobial activity was determined by means of an agar diffusion assay from the estimation of the diameter of the inhibition zone produced by the honeys. S. aureus was more inhibited than P. aeruginosa (chi-square value 11.2698, P value 0.000788). In particular, a statistically significant inhibition growth against S. aureus was exhibited by the polyfloral (chi-square value 6.1714, P value 0.012983) and the eucalyptus honeys (chi-square value 4, P value 0.0455). Results partially suggest Sicilian organic polyfloral and eucalyptus honeys as possible alternatives to antimicrobial agents when S. aureus resistance causes failure of initial conventional antibiotic treatment.

Identification and Assessment of the Behavior of Methicillin-Resistant Staphylococci in Cheese

Steinka, Izabela

ABSTRACT

This study was carried out with the aim of identifying and assessing methicillin-resistant Staphylococcus aureus (MRSA) during lactic acid cheese storage. The study involved 30 assortments of lactic acid cheese and 21 cheeses with S. aureus TWP11616 (MRSA). Results showed low MRSA contamination levels in lactic acid cheese. The majority of cow and goat lactic acid cheese samples (more than 72%) were characterized by a low level of MRSA (≤10 CFU/g). With regard to cow and sheep lactic acid cheese, methicillin-resistant Staphylococcus spp. contamination levels of ≥100 CFU/g were found in 88 and 100% of samples, respectively. The microbial dynamics of MRSA changes in lactic acid cheese suggest a significant reduction in contamination levels after 4 days of product storage, and this decrease is likely not dependent on the type of packaging method.

Determination of Alkaloids in Mitragyna speciosa (Kratom) Raw Materials and Dietary Supplements by HPLC-UV: Single-Laboratory Validation, First Action 2017.14

Mudge, Elizabeth M.; Brown, Paula N.

ABSTRACT

The AOAC Expert Review Panel (ERP) approved a method for the quantitation of alkaloids in Mitragyna speciosa for consideration as First Action Official Method SM status. The previously published method summarized a single-laboratory validation of two alkaloids, mitragynine and 7-hydroxymitragynine, in raw materials and finished products. The methods performance was compared with the AOAC Standard Method Performance Requirement 2015.008. With repeatability precision (RSDr) ranging from 0.51 to 0.95% and recoveries from 93.6 to 98.9% in the different product matrices, the ERP adopted the method and provided recommendations for achieving Final Action status.

Ultrasound-Assisted Extraction of Anthocyanins from Red Rose Petals and New Spectrophotometric Methods for the Determination of Total Monomeric Anthocyanins

Özgür, Mahmure Üstün; Çimen, Emrah

ABSTRACT

In this study, four extraction technologies for the extraction of anthocyanins (Acyns) from red rose petals (RRPs) were investigated and compared, including ultrasound-assisted extraction (UAE), reflux extraction, Soxhlet extraction, and marinated extraction. UAE was the most suitable for the extraction of Acyns from RRPs because of its high extraction efficiency and short extraction time. The results showed that the best conditions for UAE are an extraction solution of ethanol–0.1 N HCl (80 + 20, v/v), a solid-to-liquid ratio of 1:40 g/mL, a temperature of 30°C, and an extraction time of 15 min performed three times. Using such conditions, 320.4 mg Acyns/100 g RRPs was extracted. UAE was followed by two new difference spectrophotometric (DS) methods, which were developed for the fast and simple determination of Acyns in RRPs. Under the optimum experimental conditions, a linear response was observed for Acyns in the range of 12.5–62.5 μg/mL for the two proposed methods, with correlation coefficients (r) ranging from 0.9988 to 0.9995. The mean recovery values of Acyns for the DS methods were in the range of 99.8–101.5%, and the RSD was 0.5%. The respective LOD and the LOQ values were 1.4 and 4.8 for DS1 and 1.1 and 3.6 μg/mL for DS2. The stability of Acyns was also studied.

Resolution and Quantitation of Triamcinolone Acetonide and Its Coformulated Drug in the Presence of Its Impurities and Degradation Products by HPTLC and HPLC

Abbas, Samah S.1; Hegazy, Maha A.1; Hendawy, Hassan A.M.2; Weshahy, Soheir A.1; Abdelwahab, May H.2

ABSTRACT

Two specific, sensitive, and precise stability-indicating chromatographic methods have been developed for the determination of triamcinolone acetonide (TMC) and its coformulated drug, econazole nitrate (ECZ), in the presence of TMC impurities and degradation products. The first method was based on HPTLC-spectrodensitometry in which resolution and quantitation was achieved by using silica gel 60 F254 HPTLC plates and an ethyl acetate–tetrahydrofuran–ammonia mobile phase (10.0 + 7.0 + 0.1, v/v/v). The second method was a reversed-phase HPLC method in which separation was achieved using an acetonitrile–methanol–0.05 M potassium dihydrogen phosphate mobile phase, pH 3.0 (25.0 + 15.0 + 60.0, v/v/v). In both methods, the separated components were detected at 225 nm. Validation of both methods was conducted in compliance with International Conference on Harmonization (ICH) guidelines, and system suitability was confirmed. The linearity ranges were 0.20–28.00 and 0.50–55.00 µg/band for TMC and ECZ by HPTLC, whereas for HPLC, the range was 0.05–30.00 and 1.00–40.00 µg/mL for both drugs, respectively. The methods were successfully applied for the analysis of a pharmaceutical formulation and were compared with the reported method with no significant difference.

Validated HPLC–DAD Method for the Simultaneous Determination of Six Selected Drugs Used in the Treatment of Glaucoma

Baker, Mostafa M.1; Belal, Tarek S.2

ABSTRACT

This work presents a simple, sensitive, and generic HPLC–diode-array detection method for the simultaneous determination of six drugs prescribed for the treatment of open-angle glaucoma and ocular hypertension. The investigated drugs include brimonidine tartarate (BMN), acetazolamide (AZA), brinzomaide (BZA), dorzolamide HCl (DZA), levobunolol HCl (LVB), and timolol maleate (TIM). Efficient chromatographic separation was achieved using a Thermo Hypersil BDS C18 column (4.6 × 250 mm, 5 µm) with a mobile phase consisting of phosphate buffer pH 5 and acetonitrile in a ratio of 78 + 22. The flow rate was 1 mL/min, and quantification was based on measuring peak areas at 298 nm for TIM and 254 nm for the other drugs. Peaks were perfectly resolved, with retention times at 3.06, 3.87, 4.53, 5.78, 7.31, and 10.78 min for BMN, AZA, DZA, TIM, LVB, and BZA respectively. The developed method was validated according to International Conference on Harmonization guidelines with respect to system suitability, linearity, ranges, accuracy, precision, robustness, and LODs and LOQs. The proposed method showed good linearity in the ranges of 2–80, 2.5–100, 2.5–100, 5–200, 3.75–150, and 1.75–70 µg/mL for BMN, AZA, DZA, TIM, LVB, and BZA, respectively. LODs were 0.20–1.01 µg/mL for the analyzed compounds. Applicability of the proposed method to real-life situations was assessed through the analysis of five different pharmaceutical formulations, and satisfactory results were obtained.

Multivariate Validated Models for Simultaneous Determination of Ibuprofen and Famotidine in the Presence of Related Substances

Elzanfaly, Eman S.; Zaazaa, Hala E.; Soudi, Aya T.; Salem, Maissa Y.

ABSTRACT

Two multivariate validated spectrophotometric methods, namely partial least-squares (PLS) and principal component regression (PCR), were developed and validated for the determination of ibuprofen and famotidine in presence of famotidine degradation products and ibuprofen impurity (4-isobutylacetophenone). A calibration set was prepared in which the two drugs together with the degradation products and impurity were modeled using a multilevel multifactor design. This calibration set was used to build the PLS and PCR models. The proposed models successfully predicted the concentrations of both drugs in validation samples, with low root mean square error of cross validation (RMSECV) percentage. The method was validated by the estimate of the figures of merit depending on the net analyte signal. The results of the two models showed that the simultaneous determination of both drugs could be performed in the concentration ranges of 100–500 µg/mL for ibuprofen and 5–25 µg/mL for famotidine. The proposed multivariate calibration methods were applied for the determination of ibuprofen and famotidine in their pharmaceutical formulation, and the results were verified by the standard addition technique.

Chemometric-Assisted Spectrophotometric Method for the Simultaneous Quantitative Determination of Ezetimibe and Simvastatin in Their Combined Dosage Forms

Heringer de Souza, Fernanda; Todeschini, Vítor; Sangoi, Maximiliano da Silva

ABSTRACT

The multivariate method, partial least-squares (PLS), was used as a calibration procedure for the simultaneous UV spectrophotometric determination of ezetimibe and simvastatin in their pharmaceutical forms. The method was developed and satisfactorily validated according to International Conference on Harmonization guidelines with respect to specificity, linearity, precision, accuracy, and robustness. In this study, the PLS algorithms are based on the absorption spectra of 25 different mixtures of drugs obtained by a multilevel factorial design. The method was linear in the concentration range of 2–8 µg/mL for ezetimibe and 4–16 µg/mL for simvastatin ($r2 > 0.99$; $n = 7$) at wavelengths of 238 and 247 nm, respectively. The LOD and LOQ were 0.28 and 0.93 µg/mL for ezetimibe and 0.16 and 0.53 µg/mL for simvastatin, respectively. Precision and accuracy data, evaluated by RSD, were lower than 2%. The method, which proved to be robust, was performed with a 2 n full-factorial design. The validated method is simple and low cost, has a low use of polluting reagents, and is environmental friendly. Therefore, the proposed method was successfully applied for the simultaneous quantitative analysis of ezetimibe and simvastatin in commercial formulations.

Development and Validation of a Novel Stability-Indicating Reversed-Phase Ion-Pair Chromatographic Method for the Quantitation of Impurities in Marbofloxacin Tablets

Maheshwari, Priyanka1; Shukla, Neelima1; Dare, Manish Kumar2

ABSTRACT

A stability-indicating isocratic reversed-phase ion-pair chromatographic method was designed for the separation of impurities in the presence of degradation products. Marbofloxacin tablets and a placebo were exposed to the stress conditions of oxidative, acid, base, humidity, thermal, and photolytic degradation. Significant and moderate degradation was observed in acidic and oxidative stress conditions, respectively. The degradation products were well resolved from the main peak and its impurities, thus proving the stability-indicating analytical method. The method was developed by using an XTerra RP18 3.5 µm (150 × 4.6 mm) column, with the mobile phase containing a mixture of buffer (pH 2.5)–methanol–glacial acetic acid (77 + 23 + 0.5, v/v). The flow rate of the mobile phase was 1.2 mL/min, with a column oven temperature of 40°C and a detection wavelength of 315 nm. The proposed method met Veterinary International Conference on Harmonization requirements and was successfully used for impurity quantitation in marbofloxacin tablets.

A Validated High-Performance Thin-Layer Chromatographic Method for the Simultaneous Determination of Zofenopril Calcium and Hydrochlorothiazide in the Presence of the Hydrochlorothiazide Impurities: Chlorothiazide and Salamide

Rezk, Mamdouh R.; Fayed, Ahmed S.; Marzouk, Hoda M.; Abbas, Samah S.

ABSTRACT

The chromatographic analysis of either process-related impurities or degradation products is very important in the pharmaceutical industry. In this work, a simple, selective, and sensitive HPTLC method was developed and validated for the simultaneous determination of zofenopril calcium (ZOF) and hydrochlorothiazide (HCT) in the presence of the HCT impurities: A) chlorothiazide (CT) and B) salamide, in raw materials and in pharmaceutical formulation. The separation was carried out on HPTLC silica gel 60 F254 using ethyl acetate–glacial acetic acid–triethylamine (10 + 0.1 + 0.1, v/v/v) as a developing system. The separated bands were scanned densitometrically at 270 nm. Polynomial equations were used for the regression. Calibration curves were constructed for ZOF, HCT, CT, and salamide in the ranges of 0.5–10, 0.2–4, 0.05–1.4, and 0.05–1.0 µg/band, respectively. Different parameters affecting the suggested method, including developing systems of varying composition/ratios and different detection wavelengths, were studied to achieve the best resolution and precision with good sensitivity. System suitability parameters were also tested. The proposed method was validated as per the International Conference on Harmonization guidelines and was successfully applied for the quantification of the studied drugs in their pharmaceutical formulation, with no interference from excipients observed. The results obtained by the developed HPTLC method were compared statistically with those obtained by the reported HPLC method using Student's t and F ratio tests, and no significant difference was obtained, indicating the ability of the proposed method to be used for routine analysis of drug product.

Evaluation of the iQ-Check® Salmonella II Assay in Select Foods: Collaborative Study, First Action 2017.06

Bird, Patrick1; Benzinger, M. Joseph1; Bastin, Benjamin1; Crowley, Erin1; Agin, James1; Goins, David1; Clark, Mike2; Tourniaire, Jean-Philippe2; Pierre, Sophie2; Lauer, Wendy2

ABSTRACT

The iQ-Check Salmonella II Real-Time PCR test kit utilizes Salmonella-specific oligonucleotide probes and primers for the rapid and specific detection of Salmonella species in select food types. The alternative method was evaluated by using 375 g test portions in an unpaired study design for two matrices, milk chocolate and dry dog food. Each matrix was compared with the U.S. Food and Drug Administration Chapter 5 Salmonella reference method. Fourteen technicians from 12 laboratories, including academia and industry, located within the United States and Canada participated in the collaborative study. Three levels of contamination were evaluated for each matrix: an uninoculated control level (0 CFU/test portion), a low inoculum level (0.2–2 CFU/test portion), and a high inoculum level (2–5 CFU/test portion). The statistical analysis was conducted according to the Probability of Detection (POD) statistical model. The results obtained for the low inoculum level test portions produced a difference in the candidate presumptive and confirmatory results (dLPOD) value with a 95% confidence interval of −0.05, (−0.15, 0.06) for the milk chocolate and 0.10, (−0.01, 0.21) for the dry dog food. The dLPOD results indicate an equivalence between the candidate method and reference method for the matrices evaluated, and the method demonstrated acceptable interlaboratory reproducibility as determined in the collaborative evaluation. False positive and false negative rates were determined for each matrix and produce values of <2%. Based on the data generated, the method demonstrated acceptable interlaboratory reproducibility data and statistical analysis.

Kinetic Profiling of the Hydrolytic Reaction of Benazepril: Metabolic Pathway Simulation

Hemdan, A.; Michael, Adel M.

ABSTRACT

A simple, specific, and rapid kinetic study of benazepril (BNZ) hydrolysis was developed and validated using HPLC. BNZ was degraded using 0.1 N sodium hydroxide at room temperature to produce benazeprilat, which is an active metabolite of BNZ and acts as an angiotensin-converting enzyme inhibitor. Analysis was carried out using an Athena C18 column (4.6 × 250 mm, 5 µm particle size). The mobile phase consists of a mixture of phosphate buffer (pH 4.5) and acetonitrile (53 + 47, v/v) at a flow rate of 1 mL/min. UV detection was accomplished at 242 nm using moexipril as the internal standard. The method was validated according to International Conference on Harmonization guidelines, and the calibration curve was linear over the range 10–100 µg/mL, with acceptable accuracy and precision. Kinetic profiling of the hydrolysis was shown to follow pseudo-first-order kinetics. The method was applied to the assay of BNZ in combined dosage form with no interference from other ingredients. The obtained results were statistically compared with those of the official method, showing no significant difference.

Evaluation of the Thermo Scientific RapidFinder™ Salmonella Species, Typhimurium, and Enteritidis Multiplex PCR Kit

Scopes, Emma1; Screen, Jessica1; Evans, Katharine1; Crabtree, David1; Hughes, Annette1; Kaupinen, Mikko2; Flannery, Jonathan3; Bird, Patrick3; Benzinger, M. Joseph3; Agin, James3; Goins, David3; Chen, Yi4; Brodsky, Michael5; Fernandez, Maria Cristina6

ABSTRACT

The Thermo Scientific RapidFinder™ Salmonella Species, Typhimurium, and Enteritidis Multiplex PCR Kit (candidate method) is a real-time PCR assay for the detection and differentiation of Salmonella spp., and the serovars S. Typhimurium, and S. Enteritidis from poultry, pork, and environmental samples. The method was validated in comparison to the U.S. Department of Agriculture Food Safety and Inspection Service and the U.S. Food and Drug Administration reference methods. Thermo Fisher Scientific (Basingstoke, United Kingdom) tested all matrixes. In addition, two matrixes were analyzed independently by Q Laboratories, Inc. (Cincinnati, OH). Few statistically significant differences were found between the candidate and reference methods when analyzed by probability of detection. When differences were observed, these were in favor of the candidate method. All 200 inclusivity strains and none of the 45 exclusivity strains were detected, which demonstrated that the RapidFinder Salmonella Species, Typhimurium, and Enteritidis Multiplex PCR Kit was able to detect all the major groups of Salmonella, the less common subspecies of S. enterica, and the rarely encountered S. bongori. None of the exclusivity isolates analyzed were detected. Robustness testing demonstrated that the assay gave reliable performance, with specific method deviations outside the recommended parameters. Accelerated stability testing was conducted, validating the assay shelf life.

Determination of Ethanol in Kombucha, Juices, and Alcohol-Free Beer by EnzytecTM Liquid Ethanol: Single-Laboratory Validation, First Action 2017.07

Lacorn, Markus; Hektor, Thomas

ABSTRACT

EnzytecTM Liquid Ethanol is an enzymatic test for the determination of ethanol in kombucha, juices, and alcohol-free beer. The kit contains two components in a ready-to-use format. Quantification is based on the catalytic activity of alcohol dehydrogenase, which oxidizes ethanol to acetaldehyde and converts NAD+ to NADH. Measurement is performed in 3 mL cuvettes at 340 nm within 20 min. Samples with alcohol contents around 0.5% alcohol by volume need to be diluted 1:20 or 1:50 with water before measurement. Acetaldehyde interferes at concentrations higher than 3000 mg/L, whereas sulfite interferes at concentrations higher than 300 mg/L. The linear measurement range is from 0.03 up to 0.5 g/L ethanol, whereas LOD and LOQ are 1.9 and 3.3 mg/L ethanol, respectively. Kombucha with concentrations between 2.85 and 5.82 g/L showed relative repeatability standard deviation around 1%, whereas juices were below 2%. Results from a reproducibility experiment revealed that at a concentration of 0.1 g/L, the RSDR was at 2.5%, whereas at higher concentrations between 0.2 and 0.3 g/L, coefficients around 1% were obtained. Trueness was checked by using Cerilliant aqueous ethanol solutions and beer with concentration of 0.4 and 4 g/L (BCR-651 and BCR-652). Spiking of kombucha and juice samples resulted in recoveries between 95% and 104%. Acceptable stability was found for the whole test kit under accelerated conditions at 37°C for 2 weeks. The kit is also not susceptible to short freezing–thawing cycles and harsh transport conditions.

Vitamin B12 (cyanocobalamin) in Infant Formula Adult/Pediatric Nutritional Formula by Liquid Chromatography with Ultraviolet Detection: Collaborative Study, Final Action 2014.02

Giménez, Ester Campos; Martin, Frédéric

ABSTRACT

To determine the repeatability and reproducibility figures of the AOAC First Action Official Method SM 2014.02 (Vitamin B12 in Infant Formula and Adult/Pediatric Formula by Liquid Chromatography with UV Detection), a collaborative study was organized. Twenty-one laboratories located in 13 different countries agreed to participate. The study was divided into two parts. During the first part, the laboratories analyzed two samples in duplicate by using the method described in the protocol. The laboratories that provided results within the expected range were qualified for part two, during which they analyzed 10 samples in blind duplicates. Eighteen laboratories managed to provide results on time for reporting. The results were compared with the Standard Method Performance Requirement (SMPR® 2011.005) established for vitamin B12. The precision results met the requirements stated in the SMPR except for one sample. Repeatability and reproducibility relative standard deviation ranged from 1.1 to 6.5% and from 6.0 to 23.8%, respectively, with only one matrix showing reproducibility values higher than the required 11%. Horwitz ratio values were all well below 2 (0.17–0.78). The AOAC Expert Review Panel (Stakeholder Panel for Infant Formula and Adult Nutritional Expert Review Panel) determined that the data presented met the SMPR and, hence, recommended the method to be granted Final Action status in September 2016.

Clostridium difficile: Epidemiology, Pathogenicity, and an Update on the Limitations of and Challenges in Its Diagnosis

Alyousef, Abdullah A.

ABSTRACT

The bacterium originally named Bacillus difficilis was later renamed Clostridium difficile because of the difficulty associated with its isolation in the laboratory. C. difficile causes human-associated diarrhea, which is now known as C. difficile infection (CDI), a major cause of nosocomial infection mainly occurring in developed countries. Changes in antibiotic patterns in its strains produce toxins that are responsible for the high mortality rates associated with CDI; therefore, the epidemiology and severity of CDI have recently changed. Apart from CDI, C. difficile also causes opportunistic infections of the human gut usually when the normal gut flora are disrupted by broad-spectrum antibiotics. By disrupting normal gut flora, spores of C. difficile germinate and traverse the gut mucosa through flagellar binding to the mucosal epithelium where several proteins are involved in the binding of C. difficile. Proper diagnostic techniques have to be applied to ensure early identification of CDI and prompt treatment administered because false results may lead to inappropriate treatment and increase risk of cross-infection. This review discusses the epidemiology and pathogenicity of this bacterium with concern for its changing pattern over the years. Further details on the diagnosis of CDI are elaborated upon, mainly focusing on the limits of and challenges in molecular diagnosis.

From Commensal to Consumer: Staphylococcus aureus Toxins, Diseases, and Detection Methods

Nguyen, Angela T.; Tallent, Sandra M.

ABSTRACT

Staphylococcus aureus is a Gram-positive bacterium capable of causing a wide array of infections. Generally a commensal organism, S. aureus encodes several virulence mechanisms that contribute to disease progression. This review highlights toxins as a secreted virulence factor by S. aureus, the diseases that manifest as a result, and the methods used to detect them. In particular, the advantages and limitations of current toxin detection methods are discussed.

Rapid Detection of Staphylococcus aureus and Related Species Isolated from Food, Environment, Cosmetics, a Medical Device, and Clinical Samples Using the VITEK MS Microbial Identification System

Sulaiman, Irshad M.[1]; Banerjee, Pratik[2]; Hsieh, Ying-Hsin[1]; Miranda, Nancy[1]; Simpson, Steven[1]; Kerdahi, Khalil[1]

ABSTRACT

Staphylococcus spp. is considered as one of the most common human-pathogenic bacteria, causing illnesses ranging from nonthreatening skin infections to lethal diseases, including sepsis, pneumonia, bloodstream infections, and food poisoning. The emergence of methicillin-resistant Staphylococcus aureus strains has increased morbidity and mortality and resulted in a major healthcare burden worldwide. Single and multilocus sequence typing have been extensively used in the identification of Staphylococcus species. Nevertheless, these assays are relatively time-consuming and require high-quality DNA. Matrix-assisted laser desorption ionization-time-of-flight has been used recently for the rapid identification of several bacterial species. In this study, we have examined 47 Staphylococcus isolates recovered from food, environment, clinical samples, cosmetic products, and a medical device and 3 American Type Culture Collection Staphylococcus reference isolates using bioMérieux VITEK MS and VITEK 2 systems to determine isolate identity. Sequencing of the 16S ribosomal RNA gene was performed to confirm and compare the species identification data generated by VITEK 2 and VITEK MS systems. Although the VITEK 2 system could not identify one of the isolates, VITEK MS identified all 50 Staphylococcus spp. isolates tested. Results of this study clearly suggest that VITEK MS can be used in the rapid identification of Staphylococcus isolates of public health importance.

Collective Ion-Pair Single-Drop Microextraction Attenuated Total Reflectance Fourier Transform Infrared Spectroscopic Determination of Perchlorate in Bioenvironmental Samples

Chandrawanshi, Swati1; Verma, Santosh K.2; Deb, Manas K.1

ABSTRACT

Perchlorate (ClO_4^-) is an environmental pollutant that affects human health. Perchlorate acts as a competitive inhibitor of iodine uptake in the thyroid gland (sodium–iodide symporter inhibitor); thus, its determination is important for public health concerns. Water and milk constitute a significant portion of the human diet. Because regular intake leads to an increase in perchlorate concentration in the human body, the estimation of perchlorate is of great concern. In this work, ion-pair single-drop microextraction (SDME) combined with attenuated total reflectance (ATR)-FTIR spectroscopy has been developed for the determination of perchlorate in bioenvironmental (soil, water, dairy milk, breast milk, and urine) samples. Perchlorate was extracted in a single drop of methyl isobutyl ketone as an - with the cationic surfactant cetyltrimethylammonuim bromide under optimized conditions. The strongest IR peak (at 1076 cm^{-1}) was selected for the quantification of perchlorate among three observed vibrational peaks. Eight calibration curves for different concentration ranges of perchlorate were prepared, and excellent linearity was observed for absorbance and peak area in the range of 0.03–100 ng/mL perchlorate, with r values of 0.977 and 0.976, respectively. The RSDs ($n = 8$) for the perchlorate concentration ranges of 0.03–100, 0.03–0.5, 0.5–10, and 10–100 ng/mL were in the range of 1.9–2.7% for the above calibration curves. The LOD and LOQ in the present work were 0.003 and 0.02 ng/mL, respectively. The extracted microdrop was analyzed directly by ATR-FTIR spectroscopy. The parameters affecting SDME, i.e, effect of pH, stirring rate, reagent concentration, microdrop volume, and extraction time, were optimized, and the role of foreign species was also investigated. F- and t-tests were performed to check the analytical QA of the method. A noteworthy feature of the reported method is the noninterference of any of the associated ions. The results were compared with those of the ion chromatography MS method, and a high degree of acceptability was found. The method was successfully applied for the determination of perchlorate in bioenvironmental samples.

Screening of 485 Pesticide Residues in Fruits and Vegetables by Liquid Chromatography-Quadrupole-Time-of-Flight Mass Spectrometry Based on TOF Accurate Mass Database and QTOF Spectrum Library

Pang, Guo-Fang1; Fan, Chun-Lin1; Chang, Qiao-Ying1; Li, Jian-Xun1; Kang, Jian1; Lu, Mei-Ling2

ABSTRACT

This paper uses the LC-quadrupole-time-of-flight MS technique to evaluate the behavioral characteristics of MS of 485 pesticides under different conditions and has developed an accurate mass database and spectra library. A high-throughput screening and confirmation method has been developed for the 485 pesticides in fruits and vegetables. Through the optimization of parameters such as accurate mass number, time of retention window, ionization forms, etc., the method has improved the accuracy of pesticide screening, thus avoiding the occurrence of false-positive and false-negative results. The method features a full scan of fragments, with 80% of pesticide qualitative points over 10, which helps increase pesticide qualitative accuracy. The abundant differences of fragment categories help realize the effective separation and qualitative identification of isomer pesticides. Four different fruits and vegetables—apples, grapes, celery, and tomatoes—were chosen to evaluate the efficiency of the method at three fortification levels of 5, 10, and 20 μg/kg, and satisfactory results were obtained. With this method, a national survey of pesticide residues was conducted between 2012 and 2015 for 12 551 samples of 146 different fruits and vegetables collected from 638 sampling points in 284 counties across 31 provincial capitals/cities directly under the central government, which provided scientific data backup for ensuring pesticide residue safety of the fruits and vegetables consumed daily by the public. Meanwhile, the big data statistical analysis of the new technique also further proves it to be of high speed, high throughput, high accuracy, high reliability, and high informatization.

Choline Chloride–Oxalic Acid as a Deep Eutectic Solvent–Based Innovative Digestion Method for the Determination of Selenium and Arsenic in Fish Samples

Panhwar, Abdul Haleem1; Tuzen, Mustafa2; Kazi, Tasneem Gul3

ABSTRACT

An innovative and effective digestion method based on choline chloride (ChCl)–oxalic acid (Ox) deep eutectic solvent (DES) was proposed for the determination of Se and As in fish samples via electrothermal atomic absorption spectrometry (ETAAS). The impacts of different variables, including the composition and volume of ChCl–Ox, temperature, and acid addition, on analyte recovery were studied for optimization. In this procedure, an 80 mg sample was dissolved in a 1:2 molar ratio of ChCl–Ox at 105°C for 40 min, with the subsequent addition of 4.0 mL HNO_3 (1.0 M) and further heating at the same temperature for about 5 min. Next, centrifugation was applied, and the supernatant solution was filtered, diluted to a known volume, and measured by ETAAS. The accuracy of the developed method was tested using a Standard Reference Material (NIST SRM 1946 Lake Superior Fish Tissue). The proposed DES-based digestion method was successfully applied to the simultaneous extraction of Se and As from fish samples.

Selective Determination of Diazinon and Chlorpyrifos in the Presence of Their Degradation Products: Application to Environmental Samples

Rezk, Mamdouh R.1; Abd El-Aleem, Abd El-Aziz B.1; Khalile, Shaban M.1; El-Naggar, Omneya K.2

ABSTRACT

An accurate, sensitive, and selective HPLC method was developed and validated for the determination of diazinon and chlorpyrifos. These pesticides were subjected to different stress conditions, such as acidic, alkaline, oxidative, thermal, and photolytic hydrolysis. The proposed method used a C18 Eclipse Plus column (100 × 4.6 mm, 3.5 μm) and a mobile phase consisting of acetonitrile–water (70 + 30, v/v) in an isocratic separation mode. The flow rate was 1.5 mL/min, with UV detection at 247 and 230 nm for diazinon and chlorpyrifos, respectively. The proposed method was linear over the range of 0.40–50.00 μg/mL for diazinon and 0.40–40.00 μg/mL for chlorpyrifos. The proposed method was validated per International Conference on Harmonization guidelines and subsequently applied for the successful determination of the studied pesticides in bulk form in their commercial samples in the presence of their degradation products. The developed method was used for the determination of the residues of these pesticides in lavender and rosemary leaves that were pretreated with the recommended doses of these pesticides.

Comparison of Assigned Values from Participants' Results, Spiked Concentrations of Test Samples, and Isotope Dilution Mass Spectrometric Results in Proficiency Testing for Pesticide Residue Analysis

Yarita, Takashi1; Otake, Takamitsu1; Aoyagi, Yoshie1; Takasaka, Noriko2; Suzuki, Tatsuya2; Watanabe, Takaho2

The Hatano Research Institute (HRI) at the Food and Drug Safety Center has recently organized a series of proficiency-testing (PT) programs called the "External Quality Control for Food Hygiene" in order to evaluate the analytical capability of testing laboratories that inspect food samples in accordance with the Food Sanitation Act. In one of these programs, Pesticide I, consensus values calculated from the participants' analytical results were used as assigned values, and the spiked concentrations of the prepared test samples were used for evaluating variation among individual participants. In the present study, the values obtained in the 2013–2015 rounds have been assessed by comparing the analytical results of the target pesticides obtained by using two different isotope-dilution MS (IDMS) methods. These two IDMS methods are based on a combination of different pretreatment protocols and different GC separation columns. The weighted means of the observed analytical results were higher than the corresponding assigned values, but showed good agreement with the spiked concentrations. These results indicate that the spiking concentrations of the test sample from HRI are reliable, and therefore, these values can be used to evaluate the trueness of the participants' analytical method.

Validation and Measurement Uncertainty Assessment of a Microbiological Method Using Generalized Pivotal Quantity Procedure and Monte-Carlo Simulation

Sossé, Saad Alaoui; Saffaj, Taoufiq; Ihssane, Bouchaib

ABSTRACT

Recently, a novel and effective statistical tool called the uncertainty profile has been developed with the purpose of graphically assessing the validity and estimating the measurement uncertainty of analytical procedures. One way to construct the uncertainty profile is to compute the β-content, γ-confidence tolerance interval. In this study, we propose a tolerance interval based on the combination of the generalized pivotal quantity procedure and Monte-Carlo simulation. The uncertainty profile has been applied successfully in several fields. However, in order to further confirm its universality, this newer approach has been applied to assess the performance of an alternative procedure versus a reference procedure for counting of Escherichia coli bacteria in drinking water. Hence, the aims of this research were to expose how the uncertainty profile can be powerfully applied pursuant to ISO 16140 standards in the frame of interlaboratory study and how to easily make a decision concerning the validity of the procedure. The analysis of the results shows that after the introduction of a correction factor, the alternative procedure is deemed valid over the studied range because the uncertainty limits lie within the acceptability limits set at ±−0.3 log unit/100 ml for a $\beta = 66.7\%$ and $\gamma = 90\%$.

Are LOD and LOQ Reliable Parameters for Sensitivity Evaluation of Spectroscopic Methods

Ershadi, Saba1; Shayanfar, Ali2

ABSTRACT

The limit of detection (LOD) and the limit of quantification (LOQ) are common parameters to assess the sensitivity of analytical methods. In this study, the LOD and LOQ of previously reported terbium sensitized analysis methods were calculated by different methods, and the results were compared with sensitivity parameters [lower limit of quantification (LLOQ)] of U.S. Food and Drug Administration guidelines. The details of the calibration curve and standard deviation of blank samples of three different terbium-sensitized luminescence methods for the quantification of mycophenolic acid, enrofloxacin, and silibinin were used for the calculation of LOD and LOQ. A comparison of LOD and LOQ values calculated by various methods and LLOQ shows a considerable difference. The significant difference of the calculated LOD and LOQ with various methods and LLOQ should be considered in the sensitivity evaluation of spectroscopic methods.

Improvement of Versatility and Analytical Range of AOAC Official Method 2015.06 for Selenium

Hieda, Naoto1; Nagatoshi, Mariko1; Ikeuchi, Yoshihiro1; Iga, Yoshinori1; Goto, Tetsuhisa2

ABSTRACT

AOAC Official Method 2015.06 is not applicable for infant formula without selenium addition because of lack of sensitivity. In addition, Method 2015.06 specifies hydrogen gas as the cell gas of inductively coupled plasma (ICP)-MS instruments. There are only a few manufacturers who have formally adopted hydrogen gas. To expand the applicability of Method 2015.06 for infant formulas with lower selenium content and for ICP-MS instruments that do not use hydrogen gas as the cell gas, we modified the conditions of Method 2015.06. The results exhibited a good linearity (coefficient of determination >0.999) when the range of standard concentration was set from 0.4 to 16.0 µg/L and the cell gas was replaced with helium gas. The measurement precision was improved to an intermediate precision RSD value of 3.49%, and the recovery factor was 103.1%. This study demonstrates that helium gas can be used as the cell gas (easing restrictions in selecting an ICP-MS instrument) and expands the applicability of this method to infant formula samples with lower selenium content by modifying the sample preparation method.

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Fluorescent Nanodiamonds in Biomedical Applications

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ABSTRACT

Nanoparticles have an extended surface and a large surface area, which is the ratio of the size of the surface area to the volume. A functionalized surface can give rise to more modifications and therefore allows this nanomaterial to have new properties. Fluorescent molecules contain fluorophore, which is capable of being excited via the absorption of light energy at a specific wavelength and subsequently emitting radiation energy of a longer wavelength. A chemically modified surface of nanodiamond (ND; by carboxylation) demonstrated biocompatibility with DNA, cytochrome C, and antigens. In turn, fluorescent nanodiamonds (FNDs) belong to a group of new nanomaterials. Their surface can be modified by joining functional groups such as carboxyl, hydroxyl, or amino, after which they can be employed as a fluorescence agent. Their fluorescent properties result from defects in the crystal lattice. FNDs reach dimensions of 4–100 nm, have attributes such as photostability, long fluorescence lifetimes (10 ns), and fluorescence emission between 600 and 700 nm. They are also nontoxic, chemically inert, biocompatible, and environmentally harmless. The main purpose of this article was to present the medical applications of various types of modified NDs.

Determination of Cobalamin and Related Compounds in Foods

Watanabe, Fumio; Bito, Tomohiro

ABSTRACT

Cobalamin, also known as the red-colored vitamin B12, is found in animal-based foods such as meat, milk, and fish. Various cobalamin compounds are extracted from foods and converted into cyanocobalamin, which is most stable, to be analyzed by various methods. Traditionally, the cobalamin content of foods is determined by microbiological assay with Lactobacillus delbrueckii subsp. lactis American Type Culture Collection 7830. However, this lactic acid bacterium can substitute deoxyribosides or deoxynucleotides (known as an alkali-resistant factor) for cobalamin. Therefore, cobalamin contents determined by this microbiological assay are often incorrect in some foods. The difficulty of evaluating whether certain foods contain cobalamin or inactive corrinoids (or both) may be easily resolved by the use of bioautography with a cobalamin-dependent Escherichia coli after separation of the sample by silica gel TLC. LC/electrospray ionization–tandem mass spectrometry is also used to analyze corrinoid compounds, and various inactive corrinoid compounds have been identified in foods.

Inedible Azo Dyes and Their Analytical Methods in Foodstuffs and Beverages

Li, Yongxin; Yang, Yi; Yin, Shuo; Zhou, Chen; Ren, Dongxia; Sun, Chengjun

ABSTRACT

Edible colorants, as an important part of food additives, can not only enhance the sensorial attributes of foodstuffs, but can also increase one's appetite. They hold a very important position in food processing. In the past decades, the illegal addition of the inedible colorants has become one of the major issues of food safety. Industrial dyes, especially some azo dyes, are illegal additives frequently found in foodstuffs. They cannot provide any nutrients for the human body and even have toxicity, carcinogenic, or mutagenic effects, which may cause serious damage to consumers' health. There is an increasing demand to detect the inedible azo dyes in foodstuffs for health and safety reasons. In this review, the types and the physicochemical properties of the inedible azo dyes adulterated in foodstuffs and beverages are summarized, and the emphases are focused on the sample pretreatment methods and analytical techniques for monitoring of these dyes in foodstuffs and beverages.

Mass Spectrometric Analysis of Synthetic Organic Pigments

Sugaya, Naeko1; Takahashi, Mitsuko1; Sakurai, Katsumi1; Tanaka, Nobuko1; Okubo, Ichiro1; Kawakami, Tsuyoshi2

ABSTRACT

Though synthetic organic colorants are used in various applications nowadays, there is the concern that impurities by-produced during the manufacturing and degradation products in some of these colorants are persistent organic pollutants and carcinogens. Thus, it is important to identify the synthetic organic colorants in various products, such as commercial paints, ink, cosmetics, food, textile, and plastics. Dyes, which are soluble in water and other solvents, could be analyzed by chromatographic methods. In contrast, it is difficult to analyze synthetic organic pigments by these methods because of their insolubility. This review is an overview of mass spectrometric analysis of synthetic organic pigments by various ionization methods. We highlight a recent study of textile samples by atmospheric pressure solid analysis probe MS. Furthermore, the mass spectral features of synthetic organic pigments and their separation from other components such as paint media and plasticizers are discussed.

Enhanced Treatment Ability of Membrane Technology by Integrating an Electric Field for Dye Wastewater Treatment: A Review

Li, Chen1; Zhang, Meihan1; Song, Chengwen1; Tao, Ping1; Sun, Menghan1; Shao, Mihua1; Wang, Tonghua2

ABSTRACT

The increasing environmental awareness and stricter regulations have prompted the developments of various treatment technologies for dye wastewater. Membrane separation receives extensive attention as a promising technology because of many advantages. However, higher removal performance requirements and membrane fouling issues make a single separation method inadequate for the removal of dyes from industrial wastewater. Exerting an electric field on membrane separation system for dye wastewater treatment has already been proposed and newly developed in recent years because each technology complements the advantages and overcomes the challenges of the other. Although the amount of literature in this field is limited, this integrated technology has exhibited good performance on dye removal and is believed to have a bright prospect. This review mapped out the previous studies and current trends as well as provided a prospective outlook for advances in various membrane-combining technologies with an electric field, especially with the electric advanced oxidation processes. The different combination patterns, performance evaluations, removal mechanisms, and treatment parameters are gathered and discussed.

Chromatographic Analysis of Textile Dyes

Simion Beldean-Galea, Mihail1; Copaciu, Florina-Maria2; Coman, Maria-Virginia2

ABSTRACT

The textile industry uses many raw materials (natural and synthetic dyes and fibers) and different dyeing techniques that can be considered important pollutants with a negative impact on the environment (toxic working conditions, discharged wastewater, and contamination). Although synthetic dyes are intensively used, offer a wide range of colors and hues and properties of adhesion, longevity, and resistance to sunshine and chemical processes, and are cost-effective, they have begun to be restricted by many textile producers because they are nonbiodegradable and have toxic, carcinogenic, and mutagenic effects that generate some imbalances in plant, animal, and human life. Natural dyes of plant and animal origin exhibit very good tolerance to washing, rubbing, and light and are biodegradable and nontoxic; these properties have led to a call for the renewed use of these dyes. Modern analytical techniques (solid-phase extraction, spectrophotometry, HPLC, HPTLC, capillary electrophoresis) with different spectroscopy (UV-Vis, diode-array detection, pulsed amperometric detection) and/or MS/tandem mass spectrometry detectors have an important role in the textile industry in obtaining essential information about dyeing techniques, material origin, historical trade routes of ancient textiles, and environmental pollution. For this purpose, isolation, separation, and quantification methods of natural and synthetic textile dyes from various matrices (ancient and modern fabrics, water, biota, etc.) are presented.

Dye Removal from Water and Wastewater Using Various Physical, Chemical, and Biological Processes

Piaskowski, Krzysztof; Świderska-Dąbrowska, Renata; Zarzycki, Paweł K.

ABSTRACT

Synthetic dyes or colorants are key chemicals for various industries producing textiles, food, cosmetics, pharmaceutics, printer inks, leather, and plastics. Nowadays, the textile industry is the major consumer of dyes. The mass of synthetic colorants used by this industry is estimated at the level of 1 ÷ 3 × 105 tons, in comparison with the total annual consumption of around 7 × 105 tons worldwide. Synthetic dyes are relatively easy to detect but difficult to eliminate from wastewater and surface water ecosystems because of their aromatic chemical structure. It should be highlighted that the relatively high stability of synthetic dyes leads to health and ecological concerns due to their toxic, mutagenic, and carcinogenic nature. Currently, removal of such chemicals from wastewater involves various techniques, including flocculation/coagulation, precipitation, photocatalytic degradation, biological oxidation, ion exchange, adsorption, and membrane filtration. In this review, a number of classical and modern technologies for synthetic dye removal from industry-originated wastewater were summarized and discussed. There is an increasing interest in the application of waste organic materials (e.g., compounds extracted from orange bagasse, fungus biosorbent, or green algal biomasses) as effective, low-cost, and ecologically friendly sorbents. Moreover, a number of dye removal processes are based on newly discovered carbon nanomaterials (carbon nanotubes and graphene as well as their derivatives).

A Generalized Approach to Forensic Dye Identification: Development and Utility of Reference Libraries

Groves, Ethan; Palenik, Skip; Palenik, Christopher S.

ABSTRACT

While color is arguably the most important optical property of evidential fibers, the actual dyestuffs responsible for its expression in them are, in forensic trace evidence examinations, rarely analyzed and still less often identified. This is due, primarily, to the exceedingly small quantities of dye present in a single fiber as well as to the fact that dye identification is a challenging analytical problem, even when large quantities are available for analysis. Among the practical reasons for this are the wide range of dyestuffs available (and the even larger number of trade names), the low total concentration of dyes in the finished product, the limited amount of sample typically available for analysis in forensic cases, and the complexity of the dye mixtures that may exist within a single fiber. Literature on the topic of dye analysis is often limited to a specific method, subset of dyestuffs, or an approach that is not applicable given the constraints of a forensic analysis. Here, we present a generalized approach to dye identification that (1) combines several robust analytical methods, (2) is broadly applicable to a wide range of dye chemistries, application classes, and fiber types, and (3) can be scaled down to forensic casework-sized samples. The approach is based on the development of a reference collection of 300 commercially relevant textile dyes that have been characterized by a variety of microanalytical methods (HPTLC, Raman microspectroscopy, infrared microspectroscopy, UV-Vis spectroscopy, and visible microspectrophotometry). Although there is no single approach that is applicable to all dyes on every type of fiber, a combination of these analytical methods has been applied using a reproducible approach that permits the use of reference libraries to constrain the identity of and, in many cases, identify the dye (or dyes) present in a textile fiber sample.

A Validated Quantification of Sudan Red Dyes in Spicery using TLC and a 16-bit Flatbed Scanner

Milz, Barbara; Schnurr, Philip; Grafmüller, Jannis; Oehler, Kevin; Spangenberg, Bernd

ABSTRACT

We present a video-densitometric quantification method for Sudan red dyes in spices and spice mixtures, separated by TLC. Application was done band-wise in small dots using a 5 μL glass pipette. For separation, the RP-18 plates (20 × 20 cm with fluorescent dye; Merck, Germany, 1.05559) were developed in a vertical developing chamber without vapor saturation from the starting point to a distance of 70 mm by using acetonitrile, methanol, and aqueous ammonia solution (25%; 8 + 1.8 + 0.2, v/v) as mobile phase. The quantification is based on direct measurements using an inexpensive 16-bit flatbed scanner for color measurements (in red, green, and blue). Evaluation of only the green channel makes the measurements very specific. For linearization, an extended Kubelka-Munk expression for data transformation was used. The range of linearity covers more than two magnitudes and lies between 20 and 500 ng. The extraction from a 2 g sample with acetonitrile, evaporation, and reconstitution to 200 μL with methanol and the band-wise application (7 mm) of a 10 μL sample allows a statistically defined LOD of less than 500 ppb of Sudan red dyes. To perform the analysis, a separation chamber, RP-18 plates, 5 μL glass pipettes, and a 16-bit flatbed scanner for 105 € are needed; therefore, the separation method is inexpensive, fast, and reliable.

Development of Lateral Flow Immunochromatographic Strips for Micropollutants Screening Using Colorants of Aptamer Functionalized Nanogold Particles Part I Methodology and Optimization

Zhao, Shuai1; Zhang, Shan1; Wang, Sai2; Liu, Jiahui1; Dong, Yiyang1

ABSTRACT

A methodology of lateral flow immunochromatographic strip based on aptamer was developed for on-site detection of the small molecule micropollutants. In the present study, we try for the first time to investigate the feasibility of developing a strip assay for the analysis of micropollutants as methodological prototypes by combining the high selectivity and affinity of aptamers with the unique optical properties of nanogolds. This quantitative method was based on the competition for the aptamer between targets and DNA probes. Crucial parameters that might influence the sensitivity, such as the size of nanogolds, amount of aptamer, type and pH of streptavidin, type of nitrocellulose (NC) membrane, blocking procedure, and reading time, were systematically investigated to obtain the optimum assay performance. With the optimized conditions [nanogolds 25 nm, 50 μM aptamer, pH 8 of GSA (a type of streptavidin named "SA Gold," which is a sulfhydrylization streptavidin), Millipore HFC 135 NC membrane, 1% bovine serum albumin as the blocking agent and added in the running buffer and sample pad soakage agents, and 20 min reading time] the aptamer-based lateral flow assay will show a low visual limit of detection and scanning reader LOD. The strip for on-site screening using colorants of aptamer functionalized nanogold particles did not require any complicated equipment and was a potential portable tool for rapid identification of micropollutants.

Activities of Versatile Peroxidase in Cultures of Clonostachys rosea f. catenulata and Clonostachys rosea f. rosea during Biotransformation of Alkali Lignin

Rybczyńska-Tkaczyk, Kamila; Korniłłowicz-Kowalska, Teresa

ABSTRACT

The aim of this study was the evaluation of activities of versatile peroxidase (VP) in cultures of Clonostachys sp. (Gliocladium sp.) strains during biotransformation of 0.2% alkali lignin (AL). The principal component analysis (PCA) method was applied to determine the main factors and correlation between biotransformation of 0.2% AL, activity of VPs, pH value, and Clonostachys sp. strains. The biotransformation of 0.2% AL in cultures of microscopic fungi included decolorization (medium lightening) and colorization (darkening of the medium). The versatile peroxidase synthesized by the microscopic fungi tested showed activity for oxidation of Mn(II) and guaiacol, but the activity for oxidation of guaiacol in the presence of Mn(II) was significantly higher. The PCA analysis indicated a strong correlation between biotransformation of 0.2% AL and pH and between oxidation of Mn(II), guaiacol without and in the presence of Mn(II) ions, strains, and pH.

Development of Lateral Flow Immunochromatographic Strips for Micropollutant Screening Using Colorants of Aptamer-Functionalized Nanogold Particles, Part II: Experimental Verification with Aflatoxin B1 and Chloramphenicol

Zhang, Shan1; Zhao, Shuai1; Wang, Sai2; Liu, Jiahui1; Dong, Yiyang1

ABSTRACT

Lateral flow immunochromatographic strips based on colorants of aptamer-functionalized nanogold particles were developed for the detection of micropollutants aflatoxin B1 (AFB1) and chloramphenicol (CAP). The lateral flow immunochromatographic strip was based on a competitive reaction of thiolated-aptamer between micropollutants and bio-DNA probe-streptavidin as capture material immobilized at the test line. General crucial parameters that might influence the sensitivity have been systematically investigated. To test the effectiveness and applicability of the optimized conditions, two structurally unrelated micropollutants, that is, AFB1 and CAP, were chosen for detection. In the present study, lateral flow immunochromatographic strips for AFB1 and CAP analysis by combining the high selectivity and affinity of aptamers with the unique optical properties of nanogold in municipal water samples were reported for the first time. With the optimized conditions, the immunochromatographic strip showed a visual LOD of 10 ppb and a quantitative LOD of 1.05 ppb using an immunochromatographic reader for AFB1 detection and a quantitative LOD of 63.4 ppb using an immunochromatographic reader for CAP detection. Furthermore, the sensitive strip provided a good linear detection range of approximately 0–50 ppm for AFB1 detection and a wider liner detection range of approximately 0–160 ppm for CAP detection. Moreover, the immunochromatographic strip provided recovery rates for water samples of 90–110% in the AFB1 analysis and 84–108% in the CAP analysis. The results demonstrated that the immunochromatographic strip has excellent potential for wide applicability and verified that the strip methods for the optimized conditions are applicable to a variety of micropollutants. The lateral flow immunochromatographic strip could be used as a simple, rapid, and efficient screening tool for rapid on-site detection of a variety of micropollutants.

Determination of Three Chromium Textile Azo Dyes in Wastewater by SPE-LC-ESI(–)-MS/MS

Copaciu, Florina-Maria1; Simedru, Dorina2; Coman, Maria-Virginia1

ABSTRACT

In the present work, a procedure to determine three textile azo dyes, chromium-complexes [Nylosan Dark Brown (NDB), Lanasyn Dark Brown (LDB), and Lanasyn Red (LR)], from wastewater using solid-phase extraction (SPE) followed by LC-electrospray ionization negative mode tandem mass spectrometry (LC-ESI(–)-MS/MS) has been developed. The extraction/concentration and recovery degree of these dyes from liquid matrices were done on Strata WAX/NH2 cartridges. The chromatographic separation was performed using a Luna C18 (2) 100Å column by isocratic elution with a methanol–acetonitrile–water (0.2% formic acid and 2 mM ammonium formate) mixture. The linearity, the LOD, and the LOQ were determined for each textile dye. The accuracy, the precision (intra- and inter-day), and the matrix effect were also performed for the validation of the developed procedure. These chromium-complex azo dyes often used in the dyeing process by a textile factory in Romania were monitored in the influent and effluent wastewater from a treatment plant situated in its area. Applying the developed SPE-LC-ESI(–)-MS/MS procedure, the following textile dyes were detected (ng/L) in the collected wastewater samples during a 24-h period: NDB 150.1, LDB 200.6, and LR 89.0–244.0 in influents and NDB 22.8, LDB 78.6, and LR 74.0 in effluents.

Preliminary Studies of Synthetic Dye Adsorption on Iron Sludge and Activated Carbons

Świderska-Dąbrowska, Renata; Piaskowski, Krzysztof; Zarzycki, Paweł K.

ABSTRACT

There is great interest in the search for multifunctional waste-based materials that may be applied as environmentally friendly adsorbents. Iron-rich sludge from ground drinking-water treatment plants may be considered a potential adsorbent for various water contaminants. This material is generated during ground water purification because of the excess of metal ions in water (Fe, Mn). In practice, this sludge is frequently disposed of as waste material and, so far, is not commonly applied as the adsorption base. Our research aims to explore the adsorption potential of iron sludge for selected synthetic dyes, including malachite green, ponceau 4R, and brilliant blue FCF. Experimental data were performed using iron sludge collected from the Groundwater Treatment Plant in Koszalin, Poland, and comparing it with adsorption properties of commercial activated carbons (Norit SA Super and Norit CA 1). The kinetics, adsorption isotherms, and temperature influence on the removal of target dyes were investigated and discussed. Preliminary experimental data have revealed that iron sludge can be considered an adsorbent for the removal of cationic dyes.

Toward the Understanding of Micro-TLC Behavior of Various Dyes on Silica and Cellulose Stationary Phases Using a Data Mining Approach

Pereira, Jorge C.1; Marques, Jorge M.C.1; Włodarczyk, Elżbieta2; Fenert, Bożena2; Zarzycki, Paweł K.2

ABSTRACT

Planar chromatography and related techniques [micro-planar chromatography, micro-TLC, or paper-based microfluidic devices (μPADs)] present several advantages in analytical applications, such as simplicity, low cost of analysis, and the ability to work with raw complex samples without the involvement of time-consuming prepurification steps. By using commonly applied planar chromatographic systems and μPADs devices, stationary phases (silica and cellulose based), different solvent mixtures (methanol–water and dichloromethane–methanol), and proportions varying from 0 to 100% (v/v), micro-TLC migration profiles of several dyes described in terms of characteristic of chromatographic parameters (retardation factor, peak base width, and asymmetry factor) were investigated. Combining these results with some quantum mechanics calculated properties for each solute (dipole moment, polarizability), and by using the data mining approach, we modeled this overall chromatographic behavior in order to describe experimental data. With this approach, we were able to predict with reasonable confidence some chromatographic properties. This effort its crucial in order to (1) optimize solute elution, (2) increase mixture resolution, and (3) identify some molecular properties of analytes for designing simple micro-TLC. It is hoped that the presented nonhypothesis-driven data-mining approach can be helpful for understanding the chromatographic behavior of dyes on silica and cellulose adsorbents using the simplest mobile phases. This should be helpful for further designing the micro-TLC separation systems or μPADs quantification devices based on cellulose and related biopolymers and considering dye compounds as analytes for separation and sensing molecules.

Identification and Characterization of Asulam Impurities in Self Made Bulk Batch Synthesis and Quantification by RP-HPLC Method

Authors: Mahaboob Basha, D.1; Venkata Reddy, G.1; Gopi Krishna, Y.2; Kumara Swamy, B.E.3; Vijay, Rajani4

ABSTRACT

The first approach of this research paper explores the simultaneous characterization and determination of the Asulam active ingredient and its associated nine impurities in bulk batch production by the gradient reverse-phase high-performance liquid chromatographic (RP-HPLC) method. The best separation from its potential impurities and reproducible method was achieved by selecting the Cosmosil C-18 (250 × 4.6 mm, 5 µm particle size) analytical column with a run time of 40 min. The pumping chromatographic mobile phase was composed of 0.1% formic acid in milli-Q water (pH ~2.72) and methanol (80 + 20, v/v). An ambient column-oven temperature and UV detection at 260 nm were used. For this broad resolution, a gradient program was employed at a flow rate of 1.20 mL/min. All potential related substances in Asulam bulk manufacturing were ascertained by mass, proton nuclear magnetic resonance, and infrared spectroscopy. The developed HPLC method was validated with respect to linearity (25.64–151.83 mg/L for Asulam and 0.71–16.29, 1.02–12.26, 1.01–20.29, 0.60–10.01, 1.04–16.65, 0.94–22.47, 0.93–16.60, 1.00–12.45, 1.00–12.45, and 0.71–12.17 mg/L for Impurities A to I with a correlation coefficient 0.999 for Asulam and all the impurities), precision (RSD, % for active analyte Asulam and impurities were <2%), accuracy (percent recovery for Asulam at two levels ranged from 99.28 to 99.35%, and for Impurities A to I, it was 93.44 to 101.41%), and specificity. Hence, this simple and reliable HPLC method was able to determine the purity of Asulam active analyte and the level of impurities in bulk batch synthesis. By using this quantified procedure, five self-made production batches were analyzed simultaneously.

Vitamin C in Acerola and Red Plum Extracts: Quantification via HPLC, in Vitro Antioxidant Activity, and Stability of their Gel and Emulsion Formulations

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ABSTRACT

Background: The fruits acerola and red plum are known to be good sources of antioxidants, particularly vitamin C. Antioxidants are compounds that protect organisms from biomolecular damage, such as accelerated aging, caused by free radicals. Objective: The objective of this study was to extract vitamin C from acerola and red plum, incorporate these extracts into different topical formulations, and evaluate the physicochemical stabilities of these formulations under stress conditions. Methods: Vitamin C was extracted from acerola and red plum via dynamic maceration for 2 h at 50 ± 2°C and was quantified via HPLC. In vitro antioxidant activities were evaluated using DPPH assays. The extracts were then incorporated into emulsion and gel formulations in two types of packaging, and stability studies were carried out. Results: Red plum and acerola extracts were orange and red and contained vitamin C concentrations of 2732.70 ± 93.01 mg/100 g and 2.60 ± 1.2 mg/100 g, respectively. In vitro antioxidant activity resulted in over 90.0% inhibition of free radicals at 0.01 mL/mL acerola extract and 0.1 mL/mL red plum extract. In the stability study, pH values decreased for both acerola formulations when stored in the oven or in transparent glass containers. Formulations containing red plum extract were stable under all conditions. Acerola extracts contained a higher concentration of vitamin C than red plum extracts. Both extracts possessed antioxidant activity, although the acerola-based formulation was unstable when stored at high temperatures or in transparent glass containers. Highlights: Extracts from red plum and acerola contained vitamin C; antioxidant activity of the extracts resulted in over 90.0% inhibition of free radicals. Formulations containing red plum were stable under all tested conditions, and formulations containing acerola were unstable when stored in the oven or in transparent glass containers.

Determination of Total Phenolic Content Using the Folin-C Assay: Single-Laboratory Validation, First Action 2017.13

Kupina, Steve1; Fields, Chris2; Roman, Mark C.3; Brunelle, Sharon L.4

ABSTRACT

A single-laboratory validation of a method using Folin & Ciocalteu's phenol reagent (Folin-C reagent) for determination of total phenolic content of selected dietary supplement extracts was performed. The method is composed of a water extraction of dried extracts with sonication followed by reaction with the Folin-C reagent. The resulting colorimetric reaction is measured at 765 nm and compared with a standard curve generated with gallic acid standard solutions. The validation results were compared with Standard Method Performance Requirement (SMPR®) 2015.009, developed by the Stakeholder Panel on Dietary Supplements. The method demonstrated acceptable within-day RSDr of 1.96–7.47% for the five matrixes studied (grape seed extract, grape skin extract, black tea extract, green coffee extract, and cocoa extract). When gallic acid was spiked into maltodextrin (a surrogate dietary supplement carrier) at 30 or 70%, the recovery ranged from 91 to 104%, within the acceptable range established by SMPR 2015.009. Selectivity testing with glucose, fructose, and sucrose demonstrated no positive interference by these compounds. Finally, ruggedness studies demonstrated no significant effects due to changes in the heating apparatus, test material weight, read time after reaction, amount of Folin-C reagent, reaction time, reaction temperature, and amount of Na_2CO_3. The single-laboratory validation results support adoption of the method as First Action Official Method SM 2017.13 and further evaluation in a collaborative study.

Quantification and Discrimination of in Vitro Regeneration Swertia nervosa at Different Growth Periods using the UPLC/UV Coupled with Chemometric Method

Li, Jie1; Zhang, Ji2; Zuo, Zhitian1; Huang, Hengyu1; Wang, Yuanzhong2

ABSTRACT

Background: Swertia nervosa (Wall. ex G. Don) C. B. Clarke, a promising traditional herbal medicine for the treatment of liver disorders, is endangered due to its extensive collection and unsustainable harvesting practices. Objective: The aim of this study is to discuss the diversity of metabolites (loganic acid, sweroside, swertiamarin, and gentiopicroside) at different growth stages and organs of Swertia nervosa using the ultra-high-performance LC (UPLC)/UV coupled with chemometric method. Methods: UPLC data, UV data, and data fusion were treated separately to find more useful information by partial least-squares discriminant analysis (PLS-DA). Hierarchical cluster analysis (HCA), an unsupervised method, was then employed for validating the results from PLS-DA. Results: Three strategies displayed different chemical information associated with the sample discrimination. UV information mainly contributed to the classification of different organs; UPLC information was prominently responsible for both organs and growth periods; the data fusion did not perform with apparent superiority compared with single data analysis, although it provided useful information to differentiate leaves that could not be recognized by UPLC. The quantification result showed that the content of swertiamarin was the highest compared with the other three metabolites, especially in leaves at the rooted stage (19.57 ± 5.34 mg/g). Therefore, we speculated that interactive transformations occurred among these four metabolites, facilitated by root formation. Conclusions: This work will contribute to exploitation of bioactive compounds of S. nervosa, as well as its large-scale propagation. Highlights: The roots formation may influence the distribution and accumulation of metabolites.

Analysis of the VIDAS® Staph Enterotoxin III (SET3) for Detection of Staphylococcal Enterotoxins G, H, and I in Foods

Hait, Jennifer M.1; Nguyen, Angela T.2; Tallent, Sandra M.1

ABSTRACT

Background: Staphylococcal food poisoning (SFP) frequently causes illnesses worldwide. SFP occurs from the ingestion of staphylococcal enterotoxins (SEs) preformed in foods by enterotoxigenic strains of Staphylococcus species, primarily S. aureus. SEG, SEH, and SEI induce emesis and have been implicated in outbreaks. Immunological-based methods are deemed the most practical methods for the routine analysis of SEs in foods given their ease of use, sensitivity, specificity, and commercial availability. These kits are routinely used to test for SEA-SEE. However, only recently has a kit been developed to detect SEG, SEH, and SEI. Objective: Our research examined the performance of the novel VIDAS® Staph Enterotoxin III (SET3) for the detection of staphylococcal enterotoxins SEG, SEH, and SEI in foods. Methods: Here we assess the sensitivity and specificity of SET3 using duplicate test portions of six foods at varying concentrations of inclusivity and exclusivity inocula: pure SEG, SEH, SEI, S. aureus strain extracts positive for seg, seh, and sei, as well as SEA, SEB, SEC, SED, and SEE. Results: The overall detection limit was less than 2.09 ng/mL for foods inoculated with SEG, SEH, and SEI, with no cross reactivity observed. Highlights: Integrating concurrent testing to detect the presence of SEA–SEE and SEG–SEI utilizing the SET3 along with the VIDAS SET2, Ridascreen® SET total, or other comparable kits will be instrumental for the future food assessments in our laboratory and may become the new standard for SE analysis of foods.

Romer Labs RapidChek® Listeria monocytogenes Test System for the Detection of L. monocytogenes on Selected Foods and Environmental Surfaces

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ABSTRACT

The Romer Labs RapidChek® Listeria monocytogenes test system (Performance Tested Method 011805) was validated against the U.S. Department of Agriculture-Food Safety and Inspection Service Microbiology Laboratory Guidebook (USDA-FSIS/MLG), U.S. Food and Drug Association Bacteriological Analytical Manual (FDA/BAM), and AOAC Official Methods of Analysis (AOAC/OMA) cultural reference methods for the detection of L. monocytogenes on selected foods including hot dogs, frozen cooked breaded chicken, frozen cooked shrimp, cured ham, and ice cream, and environmental surfaces including stainless steel and plastic in an unpaired study design. The RapidChek method uses a proprietary enrichment media system, a 44–48 h enrichment at 30 ± 1°C, and detects L. monocytogenes on an immunochromatographic lateral flow device within 10 min. Different L. monocytogenes strains were used to spike each of the matrixes. Samples were confirmed based on the reference method confirmations and an alternate confirmation method. A total of 140 low-level spiked samples were tested by the RapidChek method after enrichment for 44–48 h in parallel with the cultural reference method. There were 88 RapidChek presumptive positives. One of the presumptive positives was not confirmed culturally. Additionally, one of the culturally confirmed samples did not exhibit a presumptive positive. No difference between the alternate confirmation method and reference confirmation method was observed. The respective cultural reference methods (USDA-FSIS/MLG, FDA/BAM, and AOAC/OMA) produced a total of 63 confirmed positive results. Nonspiked samples from all foods were reported as negative for L. monocytogenes by all methods. Probability of detection analysis demonstrated no significant differences in the number of positive samples detected by the RapidChek method and the respective cultural reference method.

Performance Validation of the Microbiologique MicrofilmTM Test System for AOAC Research Institute Performance Tested Method SM Certification

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ABSTRACT

The Microfilm™ Test System is intended for quantitative microbiology and consists of three types of Microfilms for aerobic plate count (Microfilm APC), total coliform and Escherichia coli count (Microfilm TCEc), and yeast and mold count (Microfilm YMC). This study evaluated the performance of the Microfilm Test System against International Organization for Standardization (ISO) methods on 20 food matrixes and 2 environmental surfaces. Ruggedness, robustness, and stability were also determined, while inclusivity and exclusivity studies were performed on Microfilm TCEc and YMC. An independent laboratory evaluated the performance on four food matrixes and one environmental surface. No significant differences and high correlation coefficients were observed between the Microfilm Test System and the corresponding ISO methods (ISO 4833-1:2013 for APC, ISO 4832:2006 for total coliform count, ISO 16649-2: 2001 for E. coli, and ISO 21527 Part 1 and Part 2 for YMC) in spiked food matrixes and environmental samples. These results were corroborated by the independent laboratory. Inclusivity and exclusivity studies for Microfilm TCEc showed expected results for all the E. coli strains tested (blue-violet or violet color), while the related coliforms showed the expected blue-green colonies on the Microfilm. Similarly, all 100 fungal strains tested showed typical growth on Microfilm YMC. Exclusivity testing on Microfilm TCEc and YMC showed no growth of nontarget organisms. Robustness and ruggedness studies showed no significant differences in mean difference counts at varying incubation temperatures and times. Stability studies on three lots of the Microfilm Test System showed that it is stable at 2–25°C for 12 months and at 45°C for 6 weeks.

The Validation of the Dai Nippon Medi·Ca SA Method for AOAC Research Institute Performance Tested Methods SM Certification

Shimizu, Mai; Takenaka, Kentaro; Suzuki, Takeo; Miyasaka, Aya; Matsuda, Taiki; Iwase, Tatsuhiko; Kyotani, Hitoshi

ABSTRACT

A ready-made dry medium method for Staphylococcus aureus count, the Medi·Ca SA method incubated at 35 or 37°C, was compared with the Baird-Parker method (AOAC Official Method SM 975.55) for 11 food matrices: raw beef, raw ground beef, raw lamb, cooked ham, raw salmon, frozen prawn, fresh chilled pasta, pasteurized milk, natural cheese, cream puff, and potato salad. The mean difference between the two methods at each contamination level for each matrix was <0.5 log10, and the 95% confidence intervals on the mean differences fell within the range of −0.50 to 0.50. Standard deviation of repeatability and RSDr values of the Medi·Ca SA method were generally the same level as those of the Baird-Parker method, and r2 ranged from 0.98 to 1.00. Product consistency and stability studies showed little variability between productions lots and a shelf-life of 16 months. Incubation time within the range of 22–26 h and variations to the sample volume did not adversely affect the results. These results showed that the Medi·Ca SA method is a reasonable alternative to the reference method for selected food matrices and makes it possible to simultaneously detect and enumerate S. aureus in only 24 h.

Effect of Silver Nanoparticles on Toxigenic Fusarium spp. and Deoxynivalenol Secretion in Some Grains

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ABSTRACT

Background: Deoxynivalenol (DON) is one of the most important fungal mycotoxins excreted by different Fusarium species in many types of grains and food commodities. It has high damage impact on human and animal immune systems. Objective: This in vitro study aimed to evaluate the influence of silver nanoparticles (Ag-NPs) as an inhibitor for the DON toxin excreted from some Fusarium spp., which were isolated from barely, wheat, and corn grains. Methods: Ag-NPs were estimated on Minimum Inhibitory Concentration, using levels of 5, 25, 50, 75, and 100 ppm, while the effect on DON was conducted with ELISA. Tri13 and Tri7 primers were used to evaluate the impact of Ag-NPs on the DNA of tested toxigenic Fusarium isolates. Results: Results revealed that the relative density values (Rd, %) of the isolated Fusarium from barley, wheat, and corn grains were 41.27, 26.47, and 30.76%, respectively. The predominant fungus was F. graminearum and F. culmorum in wheat and barley, respectively. The maximum inhibition diameters used for concentrations were 0.5, 2.8, 3.2, 3.3, and 3.31 mm, respectively. The impact of Ag-NPs on genomic structure was limited. Results demonstrated that Ag-NPs have the ability to reduce the linear growth of Fusarium spp. and eliminate the DON toxin to 34.44, 34.60, and 34.89% at 50, 75, and 100 ppm. Conclusions: Ag-NPs are considered nontransgenic substances, and their impact on Fusarium DNA under tested concentrations has been neglected. Ag-NPs may work as an alternative to fungicides to reduce fungal growth and eliminate DON mycotoxins.

Determination of Biogenic Amines in Cheese by Ion Chromatography with Tandem Mass Spectrometry Detection

Ščavničar, Andrej1; Rogelj, Irena2; Kočar, Drago1; Köse, Sevim3; Pompe, Matevž1

ABSTRACT

A new method for determination of underivatized biogenic amines in cheese based on ion exchange chromatography coupled with tandem mass spectrometric detection was proposed. The method was applied to the analysis of 10 biogenic amines (trimethylamine, putrescine, cadaverine, histamine, 2-phenylethylamine, spermine, spermidine, tryptamine, agmatine, and tyramine) in different types of cheese. The amines were extracted only with water without any additional derivatization step or sample cleanup. This is a great advantage in terms of simplicity of sample pretreatment procedure compared with other currently existing methods in the literature. Biogenic amines were separated using cation exchange column, under gradient elution conditions by mixing formic acid (1.00 M) and deionized water. Detection was achieved using tandem MS/MS, with the instrument set into multiple reaction monitoring mode to ensure high specificity. The detection and quantification limits were in the ranges of 12–46 µg/L and 40–153 µg/L, respectively. The exceptions were spermidine and spermine, with detection limits of 0.8 and 5.4 mg/L, respectively. The linearity for most of the biogenic amines was from 10 µg/L up to 10 mg/L. The best recoveries were observed for trimethylamine, tyramine, and cadaverine, and were 89, 94, and 102%, respectively. The results showed that this method can be used for routine determination of biogenic amines in different types of cheeses as well other food matrices. It must be stressed that the proposed method is capable of determining 10 biogenic amines, including tyramine, which is reported to cause food intoxication commonly associated with cheeses.

The Validation of the RIDA®QUICK Gliadin for AOAC Research Institute

Lacorn, Markus1; Weiss, Thomas1; Klass, Nicole2; Bird, Patrick2; Benzinger, M. Joseph2; Agin, James2; Goins, David2

ABSTRACT

RIDA®QUICK Gliadin is an immuno-chromatographic test for the detection of gluten in foods, on surfaces, and in Cleaning-in-Place (CIP) waters. This test kit has been adopted as Final Action AOAC INTERNATIONAL Official Methods of Analysis SM 2015.16 for gluten in corn products. The assay is based on the monoclonal antibody R5, which recognizes gluten in wheat, barley, and rye. Four different surfaces were contaminated with a gliadin material and analyzed by a direct swabbing of the surface with the dip-stick. The outcome was an LOD95% concentration of the assay between 1.6 and 3.0 µg/100 cm2 gluten. For CIP waters that contain cleansing reagents, 100% positive results were obtained for minimum gluten concentration between 50 and 100 ng/mL. If the CIP water does not contain these reagents, the minimum detectable gluten level is 10 ng/mL. The independent validation study consisted of a method comparison study of recovery from a CIP solution and from a stainless-steel surface. The test kit was evaluated at six different concentration levels for both matrices, with 20 or 30 replicates per concentration level. The probability of detection was calculated for each contamination level. Additionally, the LOD95% concentration was estimated for each matrix analyzed.

Detection of Peanut Allergen Ara h 6 in Commercially Processed Foods using a Single-Walled Carbon Nanotube–Based Biosensor

Sobhan, Abdus1; Oh, Jun-Hyun1; Park, Mi-Kyung2; Lee, Jinyoung1

ABSTRACT

Background: The peanut protein Arachis hypogaea (Ara h) 6 is one of the most serious food allergens that contributes to food-related, life-threatening problems worldwide. The extremely low allergic dose demands for more selective and rapid methods for detecting Ara h 6. Objective: The goal of this study was to develop a single-walled carbon nanotube (SWCNT)-based biosensor for the rapid detection of Ara h 6 in commercial food products. Methods: The detection principle of this biosensor was based on the binding of Ara h 6 to the anti-Ara h 6 antibody (pAb) through 1-pyrenibutanoic acid succinimidyl ester. The resistance difference (ΔR) was calculated via linear sweep voltammetry using a potentiostat. Results: The ΔR increased as the Ara h 6 concentrations increased above the range of 100–107 pg/L. A specificity analysis showed that the anti-Ara h 6 pAb selectively interacted with Ara h 6 molecules in the buffer solution (pH 7.4). Conclusions: This research proposes that an SWCNT-based biosensor in self-assembly with antibodies could be an effective tool for the rapid detection of allergen proteins in food. Highlights: The developed biosensor exhibited higher sensitivity and selectivity. Application studies resulted in precise Ara h 6 detection in peanut-containing processed food.

Quantification of Whey Protein Content in Milk-Based Infant Formula Powders by Sodium Dodecyl Sulfate–Capillary Gel Electrophoresis (SDS-CGE): Multilaboratory Testing Study, Final Action 2016.15

Feng, Ping1; Fuerer, Christophe2; McMahon, Adrienne3

ABSTRACT

A multilaboratory testing study was conducted on AOAC First Action Official Method SM 2016.15: Quantification of Whey Protein Content in Infant Formula Powders by Sodium Dodecyl Sulfate–Capillary Gel Electrophoresis (SDS-CGE). Nineteen laboratories participated in the analysis of duplicate blind-coded samples of 15 formula powder products for infants and young children. Electrophoregrams were recorded at UV220 nm and integrated. The normalized peak areas of whey and casein proteins were summed separately to calculate total whey protein content. Apart from one sample [NIST Standard Reference Material (SRM) 1849a], relative standard deviation of repeatability (RSDr) and reproducibility (RSDR) ranged from 0.83 to 2.11% and from 2.18 to 4.22%, respectively, and Horwitz ratios ranged from 1.02 to 1.85, meeting the precision limits specified in the whey protein Standard Method Performance Requirements and in the guidelines recommended for the Horwitz ratio. In these samples, the measured whey protein content was between 98 and 108% of the declared value. NIST SRM 1849a showed atypical results, with elevated RSDr (3.51%), RSDR (5.94%), Horwitz ratio (2.62), and recovery (134%). There is no clear reason for this. The percent whey protein value for NIST is calculated from the formulation and is not a reference or certified value. Multiple instrument models and makes, as well as capillary sources, were used in this collaborative study, demonstrating the robustness of the method. The method is fit-for-purpose for the quantification of whey protein content in milk-based formula powder products for infants and young children. It is not applicable to the analysis of hydrolyzed or plant protein–based infant formulas.

A Rapid Method for the Determination of Biotin and Folic Acid in Liquid Milk, Milk Powders, Infant Formula, and Milk-Based Nutritional Products by Liquid Chromatography–Tandem Mass Spectrometry

Gill, Brendon D.; Saldo, Sheila; Wood, Jackie E.; Indyk, Harvey E.

ABSTRACT

Background: Biotin and folate are B-group vitamins that play a critical role in numerous metabolic reactions, and they are supplemented to infant and adult nutritional formulas as free biotin and folic acid. Objective: We describe a rapid method for the analysis of biotin and folic acid that is applicable to liquid milk, milk powders, infant formula, and milk-based nutritional products. Methods: Samples are autoclaved, centrifuged, filtered, and analyzed by HPLC–MS/MS, with quantitation accomplished by the internal standard technique. Results: The method was shown to be accurate, with acceptable spike recovery (biotin: 96.5–108.2%; folic acid: 92.6–104.4%), and no bias ($\alpha = 0.05$) against either a certified reference material (biotin: $P = 0.70$; folic acid: $P = 0.23$) or established analytical method (biotin: $P = 0.10$; folic acid: $P = 0.48$) was found. Acceptable precision was confirmed with repeatability relative standard deviation (RSDr) and Horwitz ratio (HorRat) values (biotin: RSDr = 0.5–5.6%, HorRatr = 0.1–0.6; folic acid: RSDr = 2.0–3.1%, HorRatr = 0.3–0.5). Method detection limit and ruggedness experiments further demonstrated the suitability of this method for routine compliance testing. Conclusions: This rapid method is intended for use in high-throughput laboratories as part of the routine product compliance release testing of biotin and folic acid in the manufacturing of infant formulas and adult nutritional products.

Confirmation and Identification of Listeria monocytogenes, Listeria spp. and Other Gram-Positive Organisms by the Bruker MALDI Biotyper Method: Collaborative Study, First Action 2017.10

Bastin, Benjamin[1]; Bird, Patrick[1]; Crowley, Erin[1]; Benzinger, M. Joseph[1]; Agin, James[1]; Goins, David[1]; Sohier, Daniele[2]; Timke, Markus[2]; Awad, Marian[2]; Kostrzewa, Markus[2]

ABSTRACT

The Bruker MALDI Biotyper® method utilizes matrix-assisted laser desorption/ionization time-of-flight (MALDI-TOF) MS for the rapid and accurate confirmation and identification of Gram-positive bacteria from select media types. This alternative method was evaluated using nonselective and selective agar plates to identify and confirm Listeria monocytogenes, Listeria species, and select Gram-positive bacteria. Results obtained by the Bruker MALDI Biotyper were compared with the traditional biochemical methods as prescribed in the appropriate reference method standards. Sixteen collaborators from 16 different laboratories located within the European Union participated in the collaborative study. A total of 36 blind-coded isolates were evaluated by each collaborator. In each set of 36 organisms, there were 16 L. monocytogenes strains, 12 non-monocytogenes Listeria species strains, and 8 additional Gram-positive exclusivity strains. After testing was completed, the total percentage of correct identifications (to both genus and species level) and confirmation from each agar type for each strain was determined at a percentage of 99.9% to the genus level and 98.8% to the species level. The results indicated that the alternative method produced equivalent results when compared with the confirmatory procedures specified by each reference method.

A Novel Genetic Determination of a Lectin Gene in Iraqi Acinetobacter baumannii Isolates and Use of Purified Lectin as an Antibiofilm Agent

Muslim, Sahira Nsayef; Al-Kadmy, Israa M.S.; Auda, Ibtesam Ghadban; Mohammed Ali, Alaa Naseer; Al-Jubori, Sawsan Sajid

ABSTRACT

Background: Lectin was initially called hemagglutinin or agglutinin because of its capacity to agglutinate human as well as human erythrocytes. They are a heterogeneous group of proteins or glycoproteins of nonimmune origin. Because of their chemical properties, they have become a useful tool in several fields such as immunology, cell biology, molecular biology, membrane structure, pharmacology, cancer research, clinical chemistry, and genetic engineering. Objective: The wide applications of lectins users urged the need to isolate lectins from a new strain of bacteria can produce new and high yield of lectin because the current production of lectin from Pseudomonas spp. is very expensive. The goal of this study was to screen the ability of Acinetobacter baumannii isolates to produce lectin and detection of its phenotypic and genotypic profiles and detection of lectin ability to inhibit ofbiofilm formation. Methods: Fifty-one isolates from different sources were collected and detected genetically by using the recA gene. Phenotypic detection of lectin by using semi-quantitative analysis and quantitative analysis in microtiter plate. Genotypic detection of lectin by designed lec gene and used PCR technique. The lectin was extracted by using glass beads and purified by chromatographyic technique followed by sodium dodecyl sulfate-polyacrylamide gel electrophoresis for determination the molecular size of lectin and finally detection the spectrum of biofilm inhibition by the purified lectin toward biofilm producers. Results: Of 51 A. baumannii isolates, 17 (33.3%) have been found to produce lectin. Ten of 17 were sequenced, of which 2 were submitted and tested by the gene bank National Center for Biotechnology Information (NCBI), and accession numbers (KX766405.1 and KX766406.1) were obtained. These 17 isolates were phenotypically and genotypically positive for lectin and showed different lec gene expression in semi-quantitative and quantitative analysis. The activities ranged between 4–128 U/mL. Lectin purified by ammonium sulfate precipitation was used to inhibit biofilm formation. We found reduction at three different types of bacteria ranging from 26% for Klebsiella pneumonia, 46.7% for P. stutzeri and 53% for A. baumannii. These results suggested that lectin has a promising application as an antibiofilm agent to combat the growing number of multidrug-resistant pathogen-associated infections. Conclusions: Lectin has been detected recently in A. baumannii, but the genetic property of this lectin has not yet been fully studied. In our study, we determined the presence of the lectin gene (lec gene) in A. baumannii by using PCR technique, and lec PCR products were identified with various source of isolation and sequenced to screening for epidemiology and submitted to the gene bank NCBI under accession number (KX766405.1 and KX766406.1). Highlights: A. baumannii has an ability to produce lectin protein; Lec gene was detected in A. baumannii, and the sequence was recorded under accession number KX766405.1 and KX766406.1.; Lectin was extracted by glass beads and purified by chromatographyic technique; Lectin had strong effect against biofilm formation.

A Simple Vortex-Assisted Magnetic Dispersive Solid Phase Microextraction System for Preconcentration and Separation of Triazine Herbicides from Environmental Water and Vegetable Samples Using Fe3O4@MIL-100(Fe) Sorbent

Nasrollahpour, Atefe; Moradi, Seyyed Ershad

ABSTRACT

A vortex-assisted magnetic dispersive solid phase microextraction coupled with high-performance liquid chromatography has been developed for the extraction and determination of triazine herbicides by using magnetic metal organic frameworks [Fe3O4@MIL-100(Fe)] in environmental water and vegetable samples. The Fe3O4@MIL-100(Fe) composite has been characterized by using X-ray diffraction spectroscopy, tunneling electron microscopy, thermogravimetric measurement, and Brunauer-Emmett-Teller analysis. The method is based on the sorption of triazine herbicides on Fe3O4@MIL-100(Fe) because of the complex formation between iron oxide nanoparticles and triazine herbicides beside π-π interactions between organic parts of Fe3O4@MIL-100(Fe) and triazine herbicides. The experimental parameters for the preconcentration of triazine herbicides, such as the type and volume of the eluent, pH, time of the sorption and desorption, and the amount of the sorbent, were optimized. Under the optimized conditions, the method was linear over the concentration range of 0.0061 to 70 ng/mL for each triazine herbicide, and the correlation coefficients ranged from 0.9988 to 0.9997. The limit of detection of the method at a signal-to-noise ratio of 3 was 2.0 to 5.3 ng/mL. The relative standard deviations for inter- and intraday assays were in the range of 5.8 to 10.2% and 3.8 to 6.3%, respectively.

Ultrasound-Assisted Emulsification Microextraction in an Online System for Determination of Cadmium in Water and Tea Samples

Nunes, Leane Santos; Lemos, Valfredo Azevedo

ABSTRACT

In this work, a method using ultrasound-assisted emulsification microextraction (USAEME) without the use of ligands in an online system for preconcentration and determination of cadmium was developed. The method was based on the preconcentration of cadmium by USAEME, employing trichloroethylene as the extraction solvent, and subsequent retention of the rich phase in a mini-column packed with silica gel. The extracted metal was determined by flame atomic absorption spectrometry. The parameters that affect the extraction of cadmium were optimized using the univariate method. Under optimized conditions, the method presented a limit of detection of 0.17 µg/L, a limit of quantification of 0.57 µg/L, an enrichment factor of 56, and a consumptive index of 0.18 mL/min. The accuracy of the method was tested by analyzing the certified reference material, National Institute of Standards and Technology (NIST) 1573 (tomato leaves). The determination of Cd in water (drinking water, bottled water, river water, and seawater) and tea (black and green tea) samples was also performed using the proposed method. The method is simple, efficient, and eco-friendly because it requires low consumption of an organic solvent.

Single-Laboratory Validation of Rapid and Easy DNA Strip for Porcine DNA Detection in Beef Meatballs

Riztyan; Takasaki, Kazuto; Yamakoshi, Yukiko; Futo, Satoshi

ABSTRACT

Detection of meat from an animal species is required to avoid misleading food labels to consumers. Recently, we developed an easy-to-use molecular detection method by combining isothermal amplification and a DNA strip, referred to as DNA Strip. Here, we report our single-laboratory validation of DNA Strip to detect porcine DNA in beef meatballs. Our results showed that DNA Strip could specifically amplify the target of porcine DNA, with detection limit to 0.01% admixture of pork in beef meatballs. DNA Strip method was also robust because the use of heat block and laboratory water bath showed no significant differences and were comparable to the reference instrument. DNA Strip can detect porcine DNA within ca 1 h, including DNA extraction, DNA amplification, and detection. These results suggest that DNA Strip is applicable because it is easy to use and capable of detecting pork in beef meatballs with a greater detection limit.

Loop-Mediated Isothermal Amplification for Detection of Endogenous Sad1 Gene in Cotton: An Internal Control for Rapid Onsite GMO Testing

Singh, Monika; Bhoge, Rajesh K.; Randhawa, Gurinderjit

ABSTRACT

Background: Confirming the integrity of seed samples in powdered form is important prior to conducting a genetically modified organism (GMO) test. Rapid onsite methods may provide a technological solution to check for genetically modified (GM) events at ports of entry. In India, Bt cotton is the commercialized GM crop with four approved GM events; however, 59 GM events have been approved globally. GMO screening is required to test for authorized GM events. The identity and amplifiability of test samples could be ensured first by employing endogenous genes as an internal control. Objective: A rapid onsite detection method was developed for an endogenous reference gene, stearoyl acyl carrier protein desaturase (Sad1) of cotton, employing visual and real-time loop-mediated isothermal amplification (LAMP). Methods: The assays were performed at a constant temperature of 63°C for 30 min for visual LAMP and 62ºC for 40 min for real-time LAMP. Positive amplification was visualized as a change in color from orange to green on addition of SYBR® Green or detected as real-time amplification curves. Results: Specificity of LAMP assays was confirmed using a set of 10 samples. LOD for visual LAMP was up to 0.1%, detecting 40 target copies, and for real-time LAMP up to 0.05%, detecting 20 target copies. Conclusions: The developed methods could be utilized to confirm the integrity of seed powder prior to conducting a GMO test for specific GM events of cotton. Highlights: LAMP assays for the endogenous Sad1 gene of cotton have been developed to be used as an internal control for onsite GMO testing in cotton.

Isolation and Structural Characterization of Antioxidant Peptides from Degreased Apricot Seed Kernels

Zhang, Haisheng; Xue, Jing; Zhao, Huanxia; Zhao, Xinshuai; Xue, Huanhuan; Sun, Yuhan; Xue, Wanrui

ABSTRACT

Background: The composition and sequence of amino acids have a prominent influence on the antioxidant activities of peptides. Objective: A series of isolation and purification experiments was conducted to explore the amino acid sequence of antioxidant peptides, which led to its antioxidation causes. Methods: The degreased apricot seed kernels were hydrolyzed by compound proteases of alkaline protease and flavor protease (3:2, u/u) to prepare apricot seed kernel hydrolysates (ASKH). ASKH were separated into ASKH-A and ASKH-B by dialysis bag. ASKH-B (MW < 3.5 kDa) was further separated into fractions by Sephadex G-25 and G-15 gel-filtration chromatography. Reversed-phase HPLC (RP-HPLC) was performed to separate fraction B4b into two antioxidant peptides (peptide B4b-4 and B4b-6). Results: The amino acid sequences were Val-Leu-Tyr-Ile-Trp and Ser-Val-Pro-Tyr-Glu, respectively. Conclusions: The results suggested that ASKH antioxidant peptides may have potential utility as healthy ingredients and as food preservatives due to their antioxidant activity. Highlights: Materials with regional characteristics were selected to explore, and hydrolysates were identified by RP-HPLC and matrix-assisted laser desorption ionization-time-of-flight-MS to obtain amino acid sequences.

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Compositional and Structural Features of the Main Bioactive Polysaccharides Present in the Aloe vera Plant

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ABSTRACT

Aloe vera (A. barbadensis Miller) is probably one of the most popular plants, widely studied because of numerous properties associated with the polysaccharides present in its gel. In particular, two main types of bioactive polysaccharides can be distinguished in the A. vera gel: an acetylated mannose-rich polymer that functions as storage polysaccharide, and a galacturonic acid–rich polymer as the main component comprising the cell walls of the parenchymatous tissue. Interestingly, most of the beneficial properties related to the aloe plant have been associated with the acetylated mannose-rich polysaccharide, also known as acemannan. However, the composition and structural features of these polysaccharides, as well as the beneficial properties associated with them, may be altered by different factors, such as the climate, soil, postharvest treatments, and processing. Further, different analytical methods have been used not only to identify but also to characterize the main polysaccharides found in parenchyma of A. vera leaf. Within this context, the main aim of this review is to summarize the most relevant information about the structural and compositional features of the main polysaccharides found in the A. vera gel as well as the most relevant analytical techniques used for their identification and their influence on the technological, functional, and beneficial properties related to the A. vera plant.

Analyses of Aloe Polysaccharides Using Carbohydrate Microarray Profiling

Ahl, Louise I.1; Grace, Olwen M.2; Pedersen, Henriette L.3; Willats, William G.T.3; Jørgensen, Bodil3; Rønsted, Nina1

ABSTRACT

Background: As the popularity of Aloe vera extracts continues to rise, a desire to fully understand the individual polymer components of the leaf mesophyll, their relation to one another, and the effects they have on the human body are increasing. Polysaccharides present in the leaf mesophyll have been identified as the components responsible for the biological activities of A. vera, and they have been widely studied in the past decades. However, the commonly used methods do not provide the desired platform to conduct large comparative studies of polysaccharide compositions, as most of them require a complete or near-complete fractionation of the polymers. Objective: The objective for this study was to assess whether carbohydrate microarrays could be used for the high-throughput analysis of cell wall polysaccharides in aloe leaf mesophyll. Methods: The method we chose is known as comprehensive microarray polymer profiling (CoMPP) and combines the high-throughput capacity of microarray technology with the specificity of molecular probes. Results: Preliminary findings showed that CoMPP can successfully be used for high-throughput screening of aloe leaf mesophyll tissue. Seventeen species of Aloe and closely related genera were analyzed, and a clear difference in the polysaccharide compositions of the mesophyll tissues was seen. Conclusions: These preliminary data suggest that the polysaccharides vary between species and that true species of Aloe may differ from segregate genera.

Molecular Weight Determination of Aloe Polysaccharides Using Size Exclusion Chromatography Coupled with Multi-Angle Laser Light Scattering and Refractive Index Detectors

He, Kan1; Mergens, Bill1; Yatcilla, Mike1; Zheng, Qunyi1; Bao, Zhichao2; Zhang, Yuehong2; Li, Xu2; Xie, Zhaoyang2

ABSTRACT

Background: Size exclusion chromatography (SEC)/refractive index (RI) were used to determine molecular weight (MW) and molecular weight distributions (MWD) of polysaccharides. In aloe product research and quality control, commercially available pullulan and dextran are most commonly employed as calibration standards. Significant difference in the MW and MWD were found in literature when different methods were used. Objectives: This study was to investigate the traditional methods and more recent technologies used to determine the MW and MWD of Aloe vera polysaccharides. Methods: In this study, multi-angle laser light scattering (MALS) detection was studied on three polysaccharides, 1, 2, and 3, that were isolated and purified from A. vera leaf. The chemical structures of 1–3 were characterized as 1, 4-β-linked glucomannans by monosaccharide composition and glycosidic linkage analysis. Absolute MW and root-mean-square radius were determined by MALS on the isolated aloe polysaccharides. The conditions to obtain reliable results from MALS measurement were examined. Results: MALS analysis demonstrates that the 1, 4-β-linked glucomannan adopt the conformation of random coils or hard spheres in the analytical environment of a 0.1 M NaCl solution. Non-size exclusion effects and interactions between polysaccharide molecules were also observed in some aloe polysaccharides in the current analysis. The weight-average MW obtained by MALS measurement for 1, 2, and 3 are 55, 129, and 962 kDa, respectively. Comparing the results with SEC/RI calibrated by pullulan and dextran standards, marked differences in the MWD are found. Both overestimated the MW of 1 and 2 by factors of 4.4 and 4.2, and 2.4 and 1.6, when using dextran and pullulan calibration, respectively. Using pullulan calibration underestimated the MW of 3 by a factor of 3.1, but a similar result was obtained from dextran calibration compared to MALS measurement. The two isolated aloe polysaccharides were employed to be broad calibration standards or to be combined with narrow polydispersity pullulan calibration standards. Several aloe samples were tested using the different calibration curves, and the determined MWs were compared with the results obtained by MALS measurement. Conclusions: The results clearly indicated that until true polysaccharide standards become available MW and MWD's will be simply relative to the standards employed and the technologies used.

Chemical Investigation of Major Constituents in Aloe vera Leaves and Several Commercial Aloe Juice Powders

Zhang, Yuehong1; Bao, Zhichao1; Ye, Xiaoyan1; Xie, Zhaoyang1; He, Kan2; Mergens, Bill2; Li, Wenjie2; Yatcilla, Mike2; Zheng, Qunyi2

ABSTRACT

Background: There are a substantial number of papers in the scientific literature reporting on the chemical composition of the Aloe vera plant. None of these investigations are truly comprehensive nor address the differences in composition that occur through processing variations in fresh leaves and commercially available product forms. Objectives: This work was to analytically examine a range of these forms and compile the findings. Methods: Fresh A. vera leaves and a number of commercial aloe juice powders were investigated for their major chemical constituents. Samples included fresh leaves from China and Mexico, plus commercial powders from different manufacturers made from different plant parts and/or manufacturing processes. The test results include moisture, ash, fiber, protein, lipids, minerals, organic acids, free sugars, and polysaccharides. The analytical methods employed comprise inductively coupled plasma-optical emission spectroscopy for minerals, high-performance anion-exchange chromatography equipped with pulsed amperometric detection for free sugars, HPLC for organic acids, and size exclusion chromatography (SEC)–multi-angle laser light scattering (MALS)–differential refractive index (dRI) for polysaccharide analyses. The absolute MW and MW distribution were determined using MALS measurement. Results: The major constituents of A. vera fresh leaf are fibers, proteins, organic acids, minerals, monosaccharides, and polysaccharides, which accounted for 85–95% of the total composition determined. In the commercial powdered aloe juice samples, four major components—organic acids, minerals, monosaccharides, and polysaccharides—accounted for 78–84% of the total composition. Apart from the four major components, products manufactured by ethanol precipitation contained high amounts of fiber and protein, while the free sugars were removed. In ethanol-precipitated products, the polysaccharide MW was less affected by manufacturing conditions and the concentration of aloe polysaccharides was higher than in products made in the nonethanol manufacturing processes. The overall chemical profiles were found to be consistent, except for the MW and content of polysaccharides in the commercial aloe samples analyzed, which were largely dependent on the types of manufacturing processes employed. Conclusions: This present study provides a comprehensive investigation of the major chemical composition of A. vera leaf and commercially derived products. The use of the SEC combined with MALS and differential RI detectors has proved to be an improved tool for the accurate determination of polysaccharide MW and contents of the various commercially available A. vera products in this study.

Identification of Lysinibacillus fusiformis Isolated from Cosmetic Samples Using MALDI-TOF MS and 16S rRNA Sequencing Methods

Sulaiman, Irshad M.; Hsieh, Ying-Hsin; Jacobs, Emily; Miranda, Nancy; Simpson, Steven; Kerdahi, Khalil

ABSTRACT

Background: Lysinibacillus fusiformis is a Gram-positive, rod-shaped bacterium that can cause tropical ulcers, severe sepsis, and respiratory illnesses in humans. Objective: In this study, we analyzed cosmetic samples for the presence of human pathogenic microorganisms. Methods: Five unopened jars of exfoliating cream were examined initially by microbiological methods. Afterward, matrix-assisted laser desorption ionization-time-of-flight (MALDI-TOF) MS and 16S ribosomal RNA (rRNA) sequencing techniques were applied to characterize the recovered isolates. Results: Of the eight recovered Gram-positive bacterial subs, the VITEK® MS could provide genus-level identification to five subs and species-level identification to two subs (L. fusiformis with a 99.9% confidence value); one sub was unidentified. Subsequently, the deoxyriboneucleic acid sequencing of the 16S rRNA gene was done on an ABI 3500XL Genetic Analyzer for the confirmation of species identification. An analysis of sequencing data revealed a complete absence of genetic variation among the eight subs sequenced at this locus and confirmed the eight bacterial subs to be L. fusiformis, as their respective 16S rRNA sequences were identical to the available sequence in public domain (GenBank accession No. KU179364). Conclusions: Our results suggest that the VITEK MS and the 16S rRNA sequencing can be used for the identification of human pathogenic bacteria of public health importance. Highlights: We characterized eight isolates of Lysinibacillus spp. from cosmetics by MALDI-TOF MS and 16S rRNA sequence analyses.

Combination of Ultrasound-Assisted Cloud-Point Extraction with Spectrophotometry for Extraction, Preconcentration, and Determination of Low Levels of Free Formaldehyde from Cosmetic Products

Temel, Nuket Kartal; Gürkan, Ramazan

ABSTRACT

In the present study, a new preconcentration method was developed to quantify the free formaldehyde (FA) in hair cosmetics by combining ultrasound-assisted cloud-point extraction with spectrophotometry. The method is based on the ion association of FA with cationic phenothiazine group dye, toluidine blue in presence of sulfite at pH 5.0, and then extraction of the formed complex into the micellar phase of nonionic surfactant, Triton X-114. The analyte extracted into the micellar phase was diluted with ethanol, and then detected at 630 nm by spectrophotometer. Under optimal conditions, a good linear relationship was obtained in the range of 2–120 μg/L with a detection limit of 0.38 μg/L. By preconcentration of 15 mL sample, the calibration sensitivity increased by 74-fold. The intra- and inter-day precisions expressed as RSDs are lower than 4.7 and 5.2%, respectively. Compared with other techniques, the study shown here provides a simple, fast, accurate, and reliable method for the analysis of FA in cosmetic products. The contents of FA of the samples are in the range of 3.4–25.1 mg/kg with an RSD lower than 4.6%. The results were statistically in good agreement with those obtained by an independent comparison method. Highlights: A new method was developed for the preconcentration of free formaldehyde from hair cosmetics. The method is simple, fast, precise, selective, and sensitive to detect trace formaldehyde. The precision is lower than 5.2% with recovery higher than 95% after spiking. The method was validated by comparing the results with those of independent method. The formaldehyde levels of hair cosmetics were reliably monitored and determined.

Extraction Optimization for Phenolic- and Withanolide-Rich Fractions from Withania somnifera Roots: Identification and Quantification of Withaferin A, 12-Deoxywithastromonolide, and Withanolide A in Plant Materials and Marketed Formulations Using a Reversed-Phase HPLC–Photodiode Array Detection Method

Kumar, Satyanshu; Singh, Raghuraj; Gajbhiye, Narendra; Dhanani, Tushar

ABSTRACT

Background: Both the roots and leaves of Withania somnifera are products of commerce. They contain active compounds of therapeutic value and mostly different withanolides. Several pharmacological activities of W. somnifera have links to one or more withanolides. The presence of phenolic compounds in extracts could play a vital role in the reduction of blood glucose levels in diabetic subjects. Objective: The present study was carried out for the selection of a solvent to prepare extracts rich in phenolics, withaferin A (WA), 12-deoxywithastromonolide (12WD), and withanolide A (WDA). A simple, rapid HPLC method was also developed for the identification and quantification of WA, 12WD, and WDA. Methods: The extraction efficiency of aqueous alcoholic solvents including hexane, chloroform, ethyl acetate, and methanol were compared for three selected withanolides and total phenolic content. The contents of WA, 12WD, and WDA and total phenolics were determined in the extracts. The quality of nine formulations containing W. sominfera were also compared in terms of the content of WA, 12WD, and WDA and total phenolics. Results: The maximum extract yield and the total withanolide and phenolic content were obtained from aqueous alcoholic compositions at 50:50 (v/v), 70:30 (v/v), and 100:0 (v/v), respectively. In the case of organic solvents, chloroform and ethyl acetate yielded the highest concentrations of phenolics and three withanolides, respectively. The total phenolic content in formulations was in the range of 1.84–3.13%, and total withanolide content showed wide variability. Conclusions: The outcome of the present investigation could be utilized for the selection of extraction solvents to prepare W. somnifera–enriched extracts and their quality monitoring by using the developed and validated HPLC-Photodiode array detection method. Highlights: A process for preparation of phenolics and withanolides (withaferin A, 12-deoxywithastramonolide and withanolide A) enriched extracts of Withania somnifera. Simple and rapid HPLC method was also developed and validated as per the ICH guidelines for identification and quantification of three major withanolides. The developed HPLC method was applied to analyze the quality of extracts and marketed herbal products (mono, as well as poly constituents). Optimized extraction process could be utilized for upscaling process development in preparation of enriched extracts from Withania somnifera, crop improvement, bio-prospection studies and quality control.

Sustainable Eco-Friendly Ultra-High-Performance Liquid Chromatographic Method for Simultaneous Determination of Caffeine and Theobromine in Commercial Teas: Evaluation of Greenness Profile Using NEMI and Eco-Scale Assessment Tools

Shaaban, Heba; Mostafa, Ahmed

ABSTRACT

Background: Green analytical chemistry (GAC) aims to eliminate or minimize the amount of hazardous solvents consumed and generated daily worldwide. Considering the environmental impact of all analytical procedures and replacing the polluting methodologies with clean ones is of a paramount interest. Objective: This work aims to develop and validate a sustainable, fast, and economic ultrahigh-performance liquid chromatography method for simultaneous determination of methylxanthines in commercial tea samples as well as to evaluate the greenness profile of the proposed method using two greenness assessment tools: National Environmental Methods Index (NEMI) and analytical Eco-scale. Methods: The method was designed based on applying GAC principles in method development. The green chromatography approach was applied by using benign mobile phases. The chromatographic separation was optimized to minimize sample preparation, achieve short analysis time with low solvent consumption, and minimize waste generation. Results: All the studied analytes were separated in only 1.7 min. The detection limits of the studied analytes ranged from 0.015 to 0.03 mg/L, while LOQs were in the range of 0.05 to 0.1 μg/L with UV detection. The proposed method neither uses nor generates harmful chemicals, it passes the four quadrants of the NEMI greenness profile, and it has a high Eco-scale score. Conclusions: Compared with the reported methods, the proposed method is greener, more economical, and faster; therefore, it can be used as a green alternative to the existing conventional methods for routine analysis of the studied analytes without harming the environment.

Validation of the BetaStar® Advanced for Tetracyclines Test Kit for the Screening of Bulk Tank and Tanker Truck Milks for the Presence of Tetracycline Drug Residues

Ankrapp, David1; Schaus, Benjamin1; Clements, Lauren1; Klein, Frank1; Rice, Jennifer1; Rejman, John1; Boison, Joe2; Kijak, Philip3; Shelver, Weilin4

ABSTRACT

A validation study was conducted for an immunochromatographic method (BetaStar® Advanced for Tetracyclines) for detection of tetracycline antibiotic residues in raw, commingled bovine milk. The assay was demonstrated to detect tetracycline, chlortetracycline, and oxytetracycline at levels below the FDA tolerance levels but above the maximum sensitivity thresholds established by the National Conference on Interstate Milk Shipments. Results of internal and independent laboratory dose-response studies employing spiked samples were in agreement. All three drugs at the approximate 90/95% sensitivity levels were detected in milk collected from cows that had been treated with the specific drug. Selectivity of the assay was 100%, as no false-positive results were obtained in testing 881 control milk samples. Testing the estimated 90/95 sensitivity level for tetracycline (213 ppb), chlortetracycline (272 ppb), and oxytetracycline (180 ppb) and at 1000 ppb for each antibiotic resulted in 100% positive tests for each tetracycline. Results of ruggedness experiments established the operating parameter tolerances for the test. Results of cross-reactivity testing established that the assay detects certain other tetracycline drugs but does not cross-react with any of 32 drugs belonging to seven different drug classes. Abnormally high bacterial or somatic cell counts (SCC) in raw milk produced no assay interference.

Rapid Detection of Listeria in Ice Cream in 13 Hours Using the Roka Listeria Detection Assay

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ABSTRACT

Background: Listeria contamination is a major concern in the ice cream industry; therefore, early and accurate detection is vital. Current detection methods require about a 24 h enrichment period for detection. Objective: Enhance the early detection of Listeria in ice cream using the highly sensitive isothermal ribosomal RNA-based Roka/Atlas Listeria Detection Assay. Methods: The R2 Medium was developed for Listeria enrichment by Molecular Epidemiology, Inc. (Seattle, WA). Comparative growth curve studies were performed on the new R2 Medium for Listeria and the currently validated media for the Roka Listeria Detection Assay. Subsequently, a method comparison between the Roka Listeria Detection Assay and the U.S. Food and Drug Administration's (FDA) Bacteriological Analytical Manual (BAM) Chapter 10 reference method on ice cream was carried out. Results: The R2 Medium supports the growth of L. monocytogenes better than Buffered Listeria Enrichment Broth, Demi-Fraser broth, and Modified University of Vermont Broth, as indicated by the faster growth rate of the organism. When used as an enrichment medium in a method comparison study of ice cream, the results showed that R2 Medium–enriched samples tested with the Roka Listeria Detection Assay gave an equivalent performance compared with the 24 h FDA-BAM reference method at 10 and 18 h post-enrichment for Listeria. Conclusions: The results from this study indicate that the new R2 Medium and the highly sensitive Roka Listeria Detection Assay allowed for the rapid detection of Listeria species in ice cream in 13 h. Highlights: The Roka Listeria Detection Assay, in conjunction with a new media formulation (R2 Medium), allowed for the early detection of Listeria in ice cream and may be applied in other food matrixes and environmental samples.

Validation of the BetaStar® Advanced for Beta-lactams Test Kit for the Screening of Bulk Tank and Tanker Truck Milks for the Presence of Beta-lactam Drug Residues

Denhartigh, Andrew1; Reynolds, Lindsay1; Palmer, Katherine1; Klein, Frank1; Rice, Jennifer1; Rejman, John J.1; Boison, Joe2; Kijak, Philip3; Shelver, Weilin4

ABSTRACT

A validation study was conducted for an immunochromatographic method (BetaStar® Advanced for Beta-lactams) for the detection of beta-lactam residues in raw, commingled bovine milk. The assay detected amoxicillin, ampicillin, cloxacillin, penicillin, cephapirin, and ceftiofur below the U.S. Food and Drug Administration tolerance levels but above the maximum sensitivity thresholds established by the National Conference on Interstate Milk Shipments. The results of internal and independent laboratory dose-response studies employing spiked samples were in agreement. The test detected all six drugs at the approximate 90/95% sensitivity levels in milk from cows treated with each drug. Selectivity of the assay was 100%, as no false-positive results were obtained in testing 1148 control milk samples. Testing the estimated 90/95% sensitivity level for amoxicillin (8.5 ppb), ampicillin (6.9 ppb), cloxacillin (8.9 ppb), penicillin (4.2 ppb), and cephapirin (17.6 ppb), and at 100 ppb for each antibiotic, resulted in 94–100% positive tests for each of the beta-lactam drugs. The results of ruggedness experiments established the operating parameter tolerances for the assay. Cross-reactivity testing established that the assay detects other certain beta-lactam drugs, but it does not cross-react with any of 30 drugs belonging to seven different drug classes. Abnormally high bacterial or somatic cell counts in raw milk produced no assay interference.

Evaluation of a Multiplex PCR for Detection of the Top Seven Shiga Toxin-Producing Escherichia coli Serogroups in Ready-to-Eat Meats, Fruits, and Vegetables

Gao, Anli; Fishcher-Jenssen, Jennifer; Cooper, Colin; Li, Honghong; Li, Jiping; Chen, Shu; Martos, Perry

ABSTRACT

Background: Ready-to-eat (RTE) meats, fruits, and vegetables contaminated by Shiga toxin producing Escherichia coli (STEC) raise serious concerns because they are often consumed directly without further processing. Objective: To evaluate a multiplex PCR for the detection of STEC across food categories. Methods: Samples (25 g) from seven RTE meat and nine fruit and vegetable matrices were inoculated with each of seven STEC (O157:H7, O26, O121, O145, O45, O103, O111) strains targeting 10 CFU/25 g, enriched in 225 mL of modified tryptone soya broth (mTSB), and tested by a multiplex real-time PCR for stx and eae genes, following U.S. Department of Agriculture (USDA) Food Safety and Inspection Service (FSIS) Microbiology Laboratory Guidebook (MLG) 5B, which was originally validated for meat products and environmental sponge. Results: The mTSB was successful at enriching for STEC in RTE meat, fruit, and vegetable matrices, except for sprouts; however, mEHEC resulted in successful enrichment of target organisms in mung bean sprouts. Suppression of eae results by stx in PCR was observed in six fruit and vegetable matrices. Conversely, suppression of stx gene by eae was not observed. PCR solely targeting eae is recommended if a fruit or vegetable sample tested positive for stx and negative for eae. Despite the significant effect from food matrix, strain, and experimental batch, the cycle threshold of PCR was <30 in inoculated samples, and mostly 30–42 and up in uninoculated samples. Conclusions: The multiplex PCR can be adopted for detection of all seven regulated STEC in RTE meat, fruit and vegetable matrices after validation with cut off value selected and justified based on real samples.

Simultaneous Detection of Yersinia Enterocolitica and Listeria Monocytogenes in Foodstuffs by Capillary Electrophoresis and Microchip Capillary Electrophoresis Laser-Induced Fluorescence Detector

Li, Yongru1; Su, Hongwei2; Lan, Yajia1

ABSTRACT

Background: Food safety is one of the most important public health problems in the world, and pathogenic bacterium is a major factor causing serious foodborne diseases. Objective: Two methods of duplex PCR combined with capillary electrophoresis laser-induced fluorescence detector (CE-LIF) and microchip capillary electrophoresis laser-induced fluorescence detector (MCE-LIF) have been developed for the simultaneous detection of Yersinia Enterocolitica and Listeria Monocytogenes in various foods. The specific conservative sequences of these two bacteria were amplified. Methods: After labelled with nucleic acid dye SYBR Gold and SYBR Orange, the PCR products were analyzed by CE-LIF and MCE-LIF, respectively. Under the optimal conditions, the detection of PCR products of the target bacteria was achieved in less than 15 min by CE-LIF and within 6 min by MCE-LIF. Results: The alignment analysis demonstrated that the PCR products had good agreement with the sequences published in GenBank. The CE-LIF method could detect 10 CFU/mL Y. enterocolitica and L. monocytogenes, and the MCE-LIF method could detect 100 CFU/mL Y. enterocolitica and L. monocytogenes. The intraday precisions of migration time and peak area of DNA markers and PCR products were in the range of 1.13 to 1.18% and 1.60 to 6.29%, respectively, for CE-LIF and 1.18 to 1.48% and 2.85 to 4.06%, respectively, for MCE-LIF. Conclusions: The proposed methods could be applied to target bacterial detection infood samples rapidly, sensitively, and specifically. Highlights: Two new methods based on CE and MCE have been developed for the simultaneous detection of Y. enterocolitica and L. monocytogenes in foodstuffs, and they can detect the bacteria directly without any enrichment because of their high sensitivity.

Simultaneous Preconcentration and Determination of Brilliant Blue and Sunset Yellow in Foodstuffs by Solid-Phase Extraction Combined UV-Vis Spectrophotometry

Bişgin, Abdullah Taner

ABSTRACT

Background: Brilliant Blue and Sunset Yellow, two highly water-soluble synthetic food dyes, are the most popular food dyes used and consumed. Although they are not highly toxic, some health problems can be observed when excessive amounts of food products containing these dyes are consumed. Objectives: The aim of the study was to develop a simultaneous UV-Vis combined solid-phase extraction method, based on the adsorption onto Amberlite XAD-8 resin, for determination of Brilliant Blue and Sunset Yellow dyes. Methods: Sample solution was poured into the reservoir of the column and permitted to gravitationally pass through the column at 2 mL/min flow rate. Adsorbed dyes were eluted to 5 mL of final volume with 1 mol/L HNO3 in ethanol solution by applying a 2 mL/min flow rate. Dye concentrations of the solution were determined at 483 and 630 nm for Sunset Yellow and Brilliant Blue, respectively. Results: The detection limits of the method for Brilliant Blue and Sunset Yellow were determined as 0.13 and 0.66 ng/mL, respectively. Preconcentration factor was 80. Brilliant Blue contents of real food samples were found to be between 11 and 240 µg/g. Sunset Yellow concentrations of foodstuffs were determined to be between 19 and 331 µg/g. Conclusions: Economical, effective, and simple simultaneous determination of Brilliant Blue and Sunset Yellow was achieved by using a solid-phase extraction combined UV-Vis spectrometry method. Highlights: The method is applicable and suitable for routine analysis in quality control laboratories without the need for expert personnel and high operational costs because the instrumentation is simple and inexpensive.

Direct Comparison of Cavity Ring Down Spectrometry and Isotope Ratio Mass Spectrometry for Detection of Sugar Adulteration in Honey Samples

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ABSTRACT

In the last several years, economically motivated adulteration (EMA) of foods including honey has received increased attention. The addition of inexpensive sweeteners such as high fructose corn syrup or cane sugar to honey is still encountered despite scientific methods that can routinely detect this type of adulteration. The standard method for detection of these adulterants utilizes isotope ratio mass spectrometry (IRMS); however, this technique requires an elevated degree of technical knowledge for operation as well as a high cost for purchase and maintenance. Cavity ring down spectroscopy (CRDS) has demonstrated potential for this type of analysis and is less expensive with simpler operation. This study evaluates CRDS for the detection of low-cost sweeteners added to honey and compares the performance of CRDS to IRMS. Several honey samples were analyzed, and the advantages and limitations specific to CRDS were evaluated. Overall, the results indicate that CRDS provides a performance comparable to the benchmark technique IRMS for EMA honey analysis.

Comparison of Real-Time Polymerase Chain Reaction and Enzyme-Linked Immunosorbent Assay for Sensitive and Quantitative Detection of Hazelnuts in Nut Pastes

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ABSTRACT

Background: Hazelnuts, being a frequent agent of allergenic reactions, need to be detected in food products. Thus, it is necessary to develop and further investigate appropriate methods for detection. Objective: The aim of the study was to compare the analysis of nut pastes (peanut paste spiked with different amounts of hazelnut paste) as a model of contamination of confectionery. Methods: Real-time PCR and sandwich ELISA (RidaScreen Hazelnut Fast Kit) were used. Results: For real-time PCR, LOQ of 2 mg/kg and a quantification range from 2 to 10 000 mg/kg were determined. For ELISA, LOQ of 1 mg/kg and a quantification range from 1 to 100 mg/kg were determined. Conclusions: The comparison shows that the methods had comparable sensitivity with LOQs in the same order of magnitude. Although ELISA was slightly more sensitive, it required dilution of samples at higher concentrations of the analyte because of its narrow quantification range. Results of this study suggest that real-time PCR and ELISA are both suitable methods for the analysis of nut pastes over a wide range of concentrations. Achieved results could be useful for control as well as for technological purposes. Highlights: Real-time PCR analysis of peanut paste spiked with different amounts of hazelnut paste as a model is proposed. Sandwich ELISA analysis of peanut paste spiked with different amounts of hazelnut paste as a model is proposed. The analytical parameters of real-time PCR and ELISA methods are compared.

Total Folate in Infant Formula and Adult Nutritionals by Trienzyme Extraction and LC-MS/MS Quantitation: A Multilaboratory Testing Study, Final Action 2011.06

Bhandari, Sneh D.; Gao, Ming; Szpylka, John

ABSTRACT

Background: The need for an updated reference method for folate was identified as a priority by the AOAC's Stakeholder Panel on Infant Formula and Adult Nutritionals (SPIFAN) in 2011. An Expert Review Panel (ERP) found AOAC Official Method SM 2011.06 suitable for the purpose and approved it as a First Action Official Method. Objective: To determine the repeatability and reproducibility of Method 2011.06: Total Folate in Infant Formula and Adult Nutritionals by Trienzyme Extraction and LC-MS/MS Quantitation. Methods: A multilaboratory collaborative study was conducted. Eleven laboratories located in five countries participated and completed analysis of all multilaboratory testing (MLT) samples. The study was divided into two parts. In the first part, the laboratories analyzed two practice samples (blindly coded) using the updated folate Method 2011.06. The laboratories providing results within the expected range qualified for part two, in which they analyzed 11 MLT samples in blind duplicates. Results: The results were compared with the Standard Method Performance Requirements (SMPR 2011.006) established for folate. The precision results met the requirements stated in the SMPR for all of the samples. Repeatability and reproducibility relative standard deviations ranged from 3.5 to 6.6 and from 9.0 to 15.7%, respectively. Horwitz ratio values for all of the samples were well below 2 (0.61–1.06). Conclusions: The ERP determined that the method performance met the SMPR requirements in September 2017 after reviewing the presented MLT data. Highlights: The ERP recommended the method for Final Action status.

Biogenic Synthesis and Characterization of Silver Nanoparticles Using Some Medical Plants and Evaluation of Their Antibacterial and Toxicity Potential

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ABSTRACT

Background: Silver nanoparticles (AgNPs) are employed in various applications in the areas of catalysis, optoelectronics, detection and diagnostics, antimicrobials, and therapeutics. Objective: The aim of this work was to study the antimicrobial activity of aqueous and methanolic leaf extracts of Thymus vulgaris and Urtica dioica and biologically prepared silver nanoparticles, as single or in combination treatments, against Escherichia coli and methicillin-resistant Staphylococcus aureus isolates. Methods: The minimum inhibitory concentration (MIC) was quantified by using a microdilution method in sterile 96-well microtiter plates. The assessment of the toxicity of AgNP solutions was evaluated on human blood lymphocyte cells. Results: The results of this study revealed that all AgNP solutions have the lowest MIC values against the bacterial isolates in relation with the methanolic and aqueous extract solutions. However, the results showed that the increasing AgNP concentration was a critical factor influencing the interaction between AgNPs and the human lymphocytes. Conclusions: The cytotoxicity of nanoparticles increased significantly ($P < 0.05$) at high concentrations. In addition, the biosynthesized AgNPs have an increased antimicrobial activity against all tested bacterial isolates. Highlights: AgNPs have been recognized as an effective antimicrobial agent that exhibits low toxicity in humans and has diverse in vitro and in vivo applications.

Developing Inclusivity and Exclusivity Panels for Testing Diagnostic and Detection Tools Targeting Burkholderia pseudomallei, the Causative Agent of Melioidosis

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ABSTRACT

Background: Diagnostic tools designed to target Burkholderia pseudomallei, the causative agent of melioidosis that was classified as a Tier 1 Select Agent by the U.S. Centers for Disease Control and Prevention, have typically suffered from false-positive and false-negative results because of a lack of understanding of the genomic diversity of B. pseudomallei and its genetic near neighbors. Objective: In this review, we discuss a strategy for using comparative genomics to guide the design of inclusivity and exclusivity panels for the validation of assays as defined by the Standard Method Performance Requirement (SMPR). Methods: Based upon a literature review, comparative genomic analyses, and hands-on experience with diagnostic development and testing, we describe important factors to consider when developing inclusivity and exclusivity panels for testing diagnostic and/or detection tools. Results: The genomic diversity of B. pseudomallei is substantial, with the genome characterized by horizontal gene transfer, including the acquisition of genomic islands from near-neighbor species. This genomic diversity, core genome reduction, and signal erosion can complicate molecular diagnostic tool development and validation. Conclusions: Accurate diagnostic and/or detection tools targeting B. pseudomallei, an important pathogen from a public health and biodefense perspective, are needed for many applications. Utilizing whole genome sequencing data and comparative genomic techniques can guide the development and validation of such tools. Amplicon sequencing assays and assay redundancy can provide improved assay performance. Highlights:

Dye Residue Analysis in Raw and Processed Aquaculture Products: Matrix Extension of AOAC INTERNATIONAL Official Method 2012.25

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ABSTRACT

Background: Triphenylmethane dyes and metabolites are known or suspected mutagens and are prohibited in animals intended for human consumption. Despite toxicity, triphenylmethane dyes are used illegally as inexpensive treatments for fungal and parasite infections in aquatic animals. Objective: AOAC INTERNTIONAL Official Method 2012.25 for the LC-MS/MS determination of malachite green, crystal violet, brilliant green, and metabolites leucomalachite green and leucocrystal violet in seafood products was previously validated for finfish (trout, salmon, catfish, and tilapia) and shrimp, but had not been fully validated for other types of aquacultured products such as eel, molluscan shellfish, or frog or for processed seafoods. Methods: Method 2012.25 was applied to a wide scope of raw and processed aquaculture products including Arctic char, barramundi, eel, frog legs, hybrid striped bass, pompano, scallops, seabream, smoked trout, dried shrimp, and highly processed canned eel and dace products. The canned products contained oil, salt, sugar, flavorings, spices, sauces, and/or preservatives. Results: Dyes and metabolites were recovered with >85% accuracy and precision generally <20% relative standard deviation. The method detection limit was ≤0.60 μg/kg and LOQ was <1.0 μg/kg. Compounds were identified in 99% of 330 fortified and incurred samples. Conclusions: This study supports the use of Method 2012.25 for triphenylmethane dye residue analysis in a wide variety of aquacultured and seafood products. Highlights: Method 2012.25 performed well with results consistent with previous validation studies, regardless of presence of additional food ingredients or the type of processing.

Multiresidue Method of Analysis of Pesticides in Medical Cannabis

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ABSTRACT

Three related analytical methods were developed and validated for the determination of pesticides in cannabis leaves, dried cannabis flowers, and cannabis oil. The methods follow the generic sequence of an acetonitrile extraction, followed by solid-phase extraction cleanup and analysis by HPLC-tandem mass spectrometry (HPLC-MS/MS), GC-MS/MS, and GC-MS. These methods were developed to accommodate sample quantity and lipid content of the different matrices. Validation at a spiking level of 0.01 μg/g was successful for 39 pesticides in cannabis leaves and 40 pesticides in cannabis oil, and at 0.02 μg/g for 32 pesticides in cannabis flowers, with the majority of analytes showing recoveries within the acceptable range of 70–130%. With these methods established, unannounced inspections of Canadian licensed producers of cannabis revealed that out of 144 samples collected, 26 showed the presence of unauthorized pest control products.

Performance Comparison Between Monolithic, Core-Shell, and Totally Porous Particulate Columns for Application in Greener and Faster Chromatography

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ABSTRACT

Background: The introduction of monolithic rods and core-shell particles as new morphologies of packing materials different from the conventional totally porous particles resulted in a leap forward for performance in LC. Meanwhile, environmental safety has become increasingly important in many areas, especially in industry and research laboratories. Objective: This study compared the efficiencies of commercially available columns of different lengths and diameters when greener chromatographic conditions were utilized. The main purpose of this study is to help practitioners select the most appropriate stationary phase for faster and greener analysis. Methods: The three types of stationary phases were compared in terms of separation efficiency, number of theoretical plates, peak shape, selectivity, resolution, analysis time, mobile phase consideration, and permeability using six drug molecules. Results: Results indicated that core-shell and monolithic stationary phases had superiority over the conventional totally porous particles in terms of efficiency and speed of analysis. Monolithic rods had lower column backpressure and higher permeability, so they are more suitable for higher mobile phase flow rates and viscosities. However, core-shell particles provided enhanced peak shapes and number of theoretical plates. Conclusions: The choice will depend on the main purpose of analysis and the composition of the mobile phase. Compromise must be made to obtain the best trade-off between separation efficiency and analysis speed. Highlights: This study is the first to consider green chromatography concepts for the selection of the best stationary phase of new morphologies.

High-Performance Thin-Layer Chromatography Coupled with Electrospray Ionization Tandem Mass Spectrometry for Identifying Neutral Lipids and Sphingolipids in Complex Samples

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ABSTRACT

High-performance thin-layer chromatography was directly combined with electrospray mass spectrometry (ESI-MS) for structural identification issues below the level of lipid classes in complex samples through a portable, automated, elution-based interface. For samples as diverse as biodiesel and human plasma, separation conditions using Automated Multiple Development were selected in each case to provide lipid classes as zones narrow enough to ensure a direct transfer of them to ESI-MS. The respective zone of interest can be selected at will. ESI+ spectra of neutral lipids and sphingolipids showed sodium adducts when recorded from the plate. By using the described technique and ion-trap technology, the respective sodium adducts were fragmented. Sodium remained as the charge of the fragment ions and, thus, was useful for their structural identification through MSn. In this way, composition profiles of each class by ESI+-MS, and further identification of individual lipids and the molecular species belonging to each of them, were obtained by MS/MS and/or high-resolution MS. Thus, mono and diacylglycerides in ESI+ and fatty acids (in ESI−) were identified as low-concentration impurities in a fatty acid methyl ester–based biodiesel sample. Likewise, molecular species of sphingomyelins and globotriaosylceramides were unequivocally identified in human plasma samples.

End

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