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HUMAN HEALTH

ENVIRONMENTAL HEALTH

FOOD TESTING APPLICATION COMPENDIUM

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INTRODUCTION

Food Testing Compendium

With an increasing number of food safety scares, the ability to quickly and accurately determine any contamination, confirm product safety or simply complete routine testing for regulatory compliance, has become central to any companies' strategy. This compendium of applications provides insight into recent applications in food analysis: spanning across the value chain, from raw material testing to end product and packaging evaluation.

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APPLICATION NOTE

UHPLC

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Simultaneous Analysis of Nine Food Additives with the PerkinElmer Flexar FX-15 System Equipped with a PDA Detector

Introduction

Food additives are natural or synthetic substances that are added in food, beverage and pharmaceutical products for their microbicidal, preservative and flavoring properties. Among the commonly used additives, benzoic acid and its salts are widely used in beverage and food for preservation. Artificial sweeteners are widely used as sugar substitute in calorie-conscious societies, where their intake provides practically no calories and also helps fight obesity and its related ailments.

In most countries, the use of additives is regulated. In the U.S., most additives are part of the Generally Recognized As Safe (GRAS) ingredients although the FDA has established Acceptable Daily Intake (ADI) for each of them. There is a need for analytical techniques to identify and quantify additives because the food industry is required to list the type and amount of each ingredient on product labels to help consumers make dietary choices and manage food allergies.

This application note presents a fast and robust liquid chromatography method to simultaneously test nine widely used additives. Among the additives tested are: preservatives (benzoic acid, sorbic acid, dehydroacetic acid and methylparaben); artificial sweeteners (acesulfame potassium, saccharin and aspartame); flavoring agent (quinine); and a stimulant (caffeine). Method conditions and performance data including precision, accuracy and linearity are presented. The method is applied to a mouthwash and a tonic soda and the type and amount of additives are confirmed.

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APPLICATION NOTE

UHPLC

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Analysis of Color Additives in Beverages with the PerkinElmer Flexar FX-15 System Equipped with a PDA Detector

Introduction

Food colors have a great impact on consumers' perception of food quality. That explains why the use of color additives or dyes in food has become pervasive, not only in highly processed food such as cereals and frozen dessert, but also in seemingly natural food such as dairy products. Dyes are used to intensify the color of food products and make them look tempting. They are also used to minimize color variation, and to prolong color stability on shelf. There are instances however, where dyes are used unscrupulously to mask the poor quality of food products. In the U.S., the Food and Drug Administration's (FDA) data shows an alarming five-fold increase in consumption of dyes since 1955.

Color additives in food products have practically no nutritional value. Some color additives are of natural origin and are generally safe but most are synthesized from petroleum and have the potential of tainting food supplies. A commonly used dye in the U.S. called sunset yellow has carcinogenic impurities such as sudan I. In vitro studies suggest that brilliant blue, another widely used dye has the potential for neurotoxicity. A study by Schab (2004) and another study by McCann (2007) suggest that mixtures of dyes cause hyperactivity and other behavioral problems in some children. In 2010, acting on these concerns, European countries mandated a warning label stating that food containing dyes may have adverse effect on activity and attention in children.

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APPLICATION NOTE

Liquid Chromatography

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Analysis of Common Sweeteners and Additives in Beverages with the PerkinElmer Flexar FX-15 System Equipped with a PDA Detector

Introduction

Sweeteners are low or zero-calorie sugar substitutes that are added in drinks, processed foods and pharmaceutical products to provide the sweet taste of table sugar, which is also called sucrose. Sweeteners, especially artificial sweeteners, contain practically no calories because their metabolism follows a different pathway than that of sucrose. On the other hand, the intake of sucrose and the calories that derive from its metabolism is one of the leading causes of obesity and its related health problems including heart disease and diabetes. People with diabetes are unable to properly metabolize sucrose causing an abnormally high concentration of it in the blood stream with damaging effects on blood vessels and other vital body organs. In 2007 there were 23.6 million people in the U.S. living with diabetes with an alarming 1.6 million new cases each year at an annual cost of $174 billion. Worldwide, about 246 million people live with diabetes, with another seven million more cases each year making it a global epidemic. Substituting sucrose with artificial sweeteners, in addition to getting regular physical exercise and having healthy eating habits, is effective in fighting obesity and preventing or managing diabetes.

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APPLICATION NOTE

Dynamic Mechanical Analysis

Dissolution of Gelatin Monitored by DMA

Summary

This application note describes the dissolution process of gelatin obtained from an empty pharmaceutical gelcap using a PerkinElmer® DMA 8000. The experiment was performed by cutting a piece of gelatin from the gelcap and mounting it in the DMA 8000. The sample was immersed in water and the mechanical properties monitored as a function of time.

It will be shown how the temperature greatly influenced dissolution rate. The modulus gives a good indication of the softening of the material over time and the tan δ gives an indication of the material becoming more viscous over time.

Introduction

Gelatin is commonly used in both foods and pharmaceuticals. Pharmaceutically, a gelcap is used to encapsulate an active ingredient or therapeutic formulation. The composition of the gelatin can be formulated to give the best dissolution profile with respect to pH, temperature etc. so that the contents are released at the appropriate time after swallowing. The thickness, overall size, shape and composition of the gelcap can all influence the dissolution properties.

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APPLICATION NOTE

Liquid Chromatography

Increased Throughput and Reduced Solvent Consumption for the Determination of Isoflavones by UHPLC

Authors:

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Wilhad Reuter, PerkinElmer

Studies suggest that the isoflavones found in soy can exert positive physiological effects.

Introduction

All plant foods are complex mixtures of chemicals including both nutrients and biologically active non-nutrients, referred to as phytochemicals. Soy is known for having high concentrations of several physiologically-active phytochemicals, including isoflavones, phytate (inositol hexaphosphate), saponins, phytosterols and protease inhibitors. The isoflavones are what makes soy unique. Soy isoflavones are non-steroidal molecules structurally and functionally related to 17β-estradiol. Soybeans and soy foods are the only natural dietary sources that provide nutritionally relevant amounts of isoflavones.

Clinical studies suggest that consumption of isoflavones can exert positive physiological effects[1]. Recent data has demonstrated that isoflavones have potent antioxidant properties, comparable to that of the well known antioxidant vitamin E[2]. Research in several areas of healthcare has linked isoflavones to lowering risks for disease, easing menopause symptoms, reducing heart disease and cancer risk, and improving prostate and bone health. As a result of the potential health benefits of isoflavones, many soy products and isoflavone supplements are available to consumers. These fall into a category of products known as nutraceuticals or functional foods, which provide a potential health benefit from a naturally occurring substance. This has created the need for an analytical technique which can qualify and quantify the type and amount of isoflavones in a nutraceutical product.

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APPLICATION NOTE

Gas Chromatography/
Mass Spectrometry

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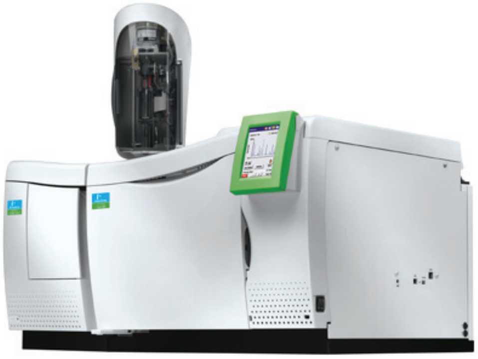
Using the D-Swafer to Investigate Solvent Impurities by Heartcutting GC/MS



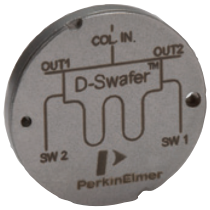
Introduction

Solvents are widely used in the pharmaceutical and food industries for a variety of purposes. It is important that such solvents are carefully quality-control (QC) tested prior to use to ensure that no unsafe levels of impurities are present.

Gas chromatography (GC) is normally the preferred technique for the determination of impurities in solvents. The inclusion of a mass spectrometric (MS) detector enables the identities of the impurities to be established.

Because many solvents are produced by fractional distillation, their impurities will have similar boiling points to that of the solvent. Thus in GC, the retention times will be similar to that of the solvent and the risk of co-elution can be high.

Furthermore, if the MS is kept active during solvent elution, contamination of the ion source or analyzer may result, and the risk of filament damage is greatly increased. This application note describes a heartcutting technique that allows the entire injected sample to reach the detector and yet resolve the issues with solvent-peak resolution and potential detector damage.



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APPLICATION NOTE

Thermal Analysis

Investigation of Amorphous Sucrose Using Material Pockets and Humidity Generator

Summary

Sucrose is a well known material used for a variety of applications. In its simplest form it is used as sugar in cooking or for coffee. It is also used as an excipient in some pharmaceutical preparations. This study shows how the amount of water present in a sugar sample will greatly affect its mechanical properties. Using the Triton Technology Humidity Controller linked to the PerkinElmer® DMA 8000, it is shown how the Tg of amorphous sucrose changes when exposed to relative humidity. In addition, a comparison of a very dry sample of sucrose with one exposed to lab atmospheric moisture is shown.

Introduction

Dynamic Mechanical Analysis (DMA) is one of the most appropriate methods to investigate relaxation events. This fact, until now, has not been exploited for powdered materials due to the difficulty in handling powders. Some work has been done with dilatometry, but with the development of the Material Pocket, it has become easier for powdered materials to be investigated in a DMA 8000.

DMA works by applying an oscillating force to the material and the resultant displacement of the sample is measured. From this, the stiffness can be determined and the modulus and tan δ can be calculated. Tan δ is the ratio of the loss modulus to the storage modulus. By measuring the phase lag in the displacement compared to the applied force it is possible to determine the damping properties of the material. Tan δ is plotted against temperature and glass transition is normally observed as a peak since the material will absorb energy as it passes through the glass transition.

The following example shows sucrose to be very hygroscopic, at least in the initial adsorption of water. It also shows the effect of moisture on the glass transition temperature.

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APPLICATION NOTE

FT-NIR Spectrometry

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Shelton, CT

Detection of Honey Adulteration Using FT-NIR Spectroscopy

Introduction

It is not uncommon to find high-value commodities such as foods to have compromised quality. These commodities can be adulterated by unscrupulous suppliers to increase their profit margins. Unfortunately, it is often difficult to determine if products have been adulterated.

A high-value product commonly associated with adulteration is honey. Adding corn syrup allows dishonest suppliers to maintain the sweet taste without a noticeable difference in the product. Without testing, it is hard to tell which honeys are adulterated and which are not. Traditional testing methods for adulterated honey can be lengthy and expensive. Fraudulent mislabeling of honey is also a major problem. FDA guidelines for labeling of honey state:

- If a food contains only honey, the food must be named "honey".
- If a food contains honey and any other ingredients such as sweeteners it must be labelled accordingly, for example, "blend of honey and sugar".
- The floral source can be stated, such as Clover Honey.
- Any product that is not pure honey cannot be labeled as "honey."

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APPLICATION NOTE

Liquid Chromatography

Authors:

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Analysis of Sugars in Honey Using the PerkinElmer Altus HPLC System with RI Detection

Introduction

Honey consumption has grown significantly during the last few decades due to its high nutritional value and unique flavor. The price of natural bee honey is much higher than other sweeteners making it susceptible to adulteration with cheaper sweeteners, primarily sucrose. Besides lower levels of non-sugar ingredients, natural honey primarily consists of glucose and fructose and may contain low levels of sucrose and/or maltose.[1,2] However, according to the international regulations, any commercially available "pure"-labeled honey products that are found to have in excess of 5% by weight of sucrose or maltose are considered to be adulterated.[3]

With the focus on possible honey adulteration, this application highlights the LC separation of various sugars found in honey and the analysis of these components in four store-bought honey samples. Method conditions and performance data, including linearity and repeatability, are presented.

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APPLICATION NOTE

Dynamic Mechanical Analysis

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Characterizing the Hydrothermal Behavior of Starch with Dynamic Mechanical Analysis

Introduction

Starch is one of the primary sources of energy in the human diet, and is also used in a wide range of industrial processes, including brewing, bioethanol production, paper manufacture and in the production of biodegradable plastics.[1]

Starch exists in plants in a granular form, the granules being between 1 and 100 µm in diameter, and has a complex semi-crystalline structure. Starch consists of two polymeric components: amylose, which is an essentially linear α (1→4) linked glucose chain, and amylopectin, which is a branched polymer of α (1→4) linked glucose chains interspersed with α (1→6) branch points. The relative proportions of amorphous and crystalline material in the starch granule, and the arrangement of structure in the granule, have a significant bearing on the behavior of the starch and its response to hydrothermal treatments.[2]

One of the most important modifications of starch structure that occurs during processing of starch, for both food usage and industrial applications, is gelatinization. When heated in excess water, starch goes through a thermal transition, termed gelatinization, at temperatures between 50 and 70 °C. Starch gelatinization is an endothermic transition associated with rapid swelling of the granule and melting of crystalline regions. In the absence of water, starch crystallites go through a melting transition at much higher temperatures

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APPLICATION NOTE

Gas Chromatography/
Mass Spectrometry

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Qualifying Mustard Flavor by Headspace Trap GC/MS using the Clarus SQ 8

Mustard is a common condiment used across many cultures and culinary styles to enhance the dining experience. It is derived from the mustard seed and is used either as a dried spice, spread or paste when the dried spice is mixed with water, vinegar or other liquid. The characteristic sharp taste of mustard arises from the isothiocyanates (ITCs) present as result of enzymatic activity made possible when the ground seed is mixed with liquids. The focus of this application brief is the characterization of these ITCs by headspace trap gas chromatography/mass spectrometry (GC/MS) and a qualitative description of their relationship to sharpness in taste across various mustard products.

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APPLICATION NOTE

FT-IR Spectroscopy

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Shelton, CT

The Use of FT-IR Spectroscopy as a Technique for Verifying Maple Syrup Authenticity

Introduction

Although usually not thought of until pancakes or waffles are on the table, maple syrup is a serious business. It is one of the key crops where demand is greater than supply. Surprisingly, it takes 10 gallons of sugar maple tree sap to produce one quart of maple syrup. Because the syrup produced is only 1/40th of the actual sap yield, unscrupulous syrup suppliers are tempted to fraudulently adulterate their products with lower value commodities, in order to maximize their profit. Adulterants include cane syrup, high fructose corn syrup, beet syrup, and rice syrup. Infrared spectroscopy is shown here to be a fast and easy technique for detection and identification of these adulterants.

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APPLICATION NOTE

Liquid Chromatography

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Shelton, CT

The Qualitative and Quantitative Analysis of Steviol Glycosides by HPLC-PDA in Energy/Vitamin Drinks

Introduction

With an emphasis on decreasing calorie intake, more and more individuals are focusing on lowering their sugar consumption from foods and beverages. In response to this, food/beverage manufacturers are now introducing the highly touted sugar substitute rebaudioside A (Reb A), in place of all or most of the sugar in certain foods and beverages. The primary interest in Reb A is the fact that it is a naturally derived sweetener considered to be at least 400 times sweeter than sugar and, therefore, can be added to products in considerably lower concentrations. The use of Reb A has been especially accelerated now that it is considered as *Generally Recognized as Safe* (GRAS) by the U.S. FDA[1]. Reb A and stevioside, both steviol glycosides, are the primary extracts from the *Stevia rebaudiana* plant from South America, particularly from Paraguay. Along with Reb A and stevioside, two secondary steviol glycosides, rebaudioside B (Reb B) and rebaudioside C (Reb C), may also be introduced into food/beverage products as part of the stevia extracts, though both of these are not as sweet.

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APPLICATION NOTE

Food Safety

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The Analysis of Baby Foods and Juices for Metals to Protect a Sensitive Population

Introduction

Babies and small children are an especially sensitive population to exposure to environmental contaminants. Their small mass and developing systems, including brain development may show adverse health effects from even low levels of contamination on a chronic or single dose case. Foods, infant formula, milk, and water provide significant exposure routes for metal contaminants. The effect of lead exposure at low levels has been well established and levels below toxic have been shown to contribute to behavioral and learning issues[1]. Other elements, such as arsenic and cadmium can contribute to cancer, neurological effects and diseases.

Although baby food and juice may contain metals from fruit grown in contaminated soil or introduced during the manufacturing process, there is very little guidance on levels considered to be safe. In the US, the Food and Drug Administration (FDA) has issued a guidance memo on lead in children's candy lollipops for import, suggesting that lead content above 0.1 ppm (mg/kg) would provide an unacceptable risk to children who regularly consume sweets.[2] This is extrapolated to guidance for many food items. Canada has a specific tolerance level of 0.1 mg/kg (ppm) for arsenic and of 0.2 ppm for lead in ready-to-serve fruit juices, nectars, and beverages[3]. Europe has examined the problem more carefully and several elements are regulated in a variety of foodstuffs through Commission Regulation (EC) No 1881/2006.[4] Lead and cadmium are regulated at low levels in materials such as cereals, fruit, and vegetables, although not specifically baby food. Tin is regulated at 50 ppm specifically in canned baby food and infant formula.

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APPLICATION NOTE

Atomic Absorption

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Determination of Arsenic in Baby Foods and Fruit Juices by GFAAS

Introduction

The United States does not have specific regulations specifying the allowable levels of toxic elements in foods, but many other countries do. For example, Canada has a specific tolerance level for arsenic of 0.1 ppm in ready to serve fruit juices, nectars, and beverages[1]. The toxic nature of arsenic is such that chronic exposure to the element can lead to internal cancers of the bladder and kidney, skin cancer, neurological effects, and cardiovascular disease.

Arsenic can find its way into food through a variety of paths. In the recent past, various organic arsenicals were used as herbicides and antimicrobial agents in growth fields as well as applied directly on fruits and fruit trees. Prior to 2003, arsenic was commonly used as a wood preservative. Sawing and/or sanding of this wood would yield arsenic contaminated sawdust. In some areas, arsenic is naturally found in rock formations and can enter soil and water which is used in the growth of food products. Foods can also be contaminated during manufacturing, processing, packaging and transport processes.

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APPLICATION BRIEF

ICP - Mass Spectrometry Analytics

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Geographical Differences of Trace Elements in Wines – Analysis with NexION 300X/350X ICP-MS and Visualization with TIBCO Spotfire Software

Introduction

Traceability of the wine origin is important for brand protection. Elemental profiles of wines have been shown to be specific for their geographic origin[1, 2], since the levels of trace metals in wines are related to the soil in the grapevine cultivation area.

In this study, a total of 75 Italian red wines from different regions and grape types were analyzed by ICP-MS to determine whether elemental profiles correlate to the region of origin. Results were imported into TIBCO Spotfire® software for statistical calculations and to display geospatial distribution.

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APPLICATION NOTE

UV/Visible Spectroscopy

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Wine Analysis Using the LAMBDA Series Spectrophotometers

Introduction

Wine has been made for the past 8,000 years using traditional methods. In recent years, there has been an worldwide increase in production – much of it coming from "New-World" wine producers such as the USA (California, in particular), Australia, New Zealand, South Africa, and some South American countries – notably Argentina and Chile. Even unlikely candidates as wine producers, such as the UK and Canada, are producing quality wines in relatively small quantities. This broadening of geographic locations has also led to an increase in the technology used in both the manufacturing and testing of the product to ensure consistency of flavor and product safety. Modern analytical techniques, such as Gas and Liquid Chromatography (GC and HPLC), are used by big producers. Another technique is UV/Visible spectroscopy.

One indicator of wine quality is its color. In previous times, this would have been done by eye but this is semi-quantitative at best. It is useful to be able to assign numbers to wine color using instrumentation. Although color is mainly a quality consideration, it does also help with safety issues as a change in color may indicate a fault in the brewing process or indicate bacterial or other contamination.

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APPLICATION NOTE

Atomic Absorption

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The Analysis of Copper, Iron, and Manganese in Wine with FAST Flame Atomic Absorption

Introduction

With the growing popularity of wine consumption in China, regulations on the safety and quality of wine are being implemented. Recent wine imports into China are required to meet mandated elemental limits and are subject to local testing upon arrival. If a wine does not meet the specifications listed in Table 1, it can be subject to destruction or return to its point of origin.

These elements are naturally occurring in wine grapes and, as such, are normally present in the wine produced from them. Concentrations of these elements can vary from region to region and from variety to variety due to the presence of nutrients in the soil the grapes are grown in, the uptake of these nutrients by the vine itself, and the process by which the wine is made. Because of this great variability, there is no way to ensure that a given wine meets the import specifications without undertaking analytical testing. Due to the possibility of the wine being rejected upon arrival into China and the financial impact this represents, wine producers and exporters are interested in a simple and accurate method for determining the concentrations of elements of interest in their wine.

Table 1. Elemental limits on wines imported into China.

Element	Limit (mg/L)
Copper (Cu)	1
Iron (Fe)	8
Manganese (Mn)	2

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PRODUCT NOTE

Gas Chromatography/
Mass Spectrometry

Clarus® SQ 8 GC/MS with TurboMatrix Headspace Trap System Application Pack for Monitoring Volatile Organic Compounds in Beer Production

Beer is a popular beverage produced by the fermentation of hopped malt extracted from barley and other grains. Although simple in concept, beer is a highly complex mixture of many compounds including sugars, proteins, alcohols, esters, acids, ketones and terpenes. Flavor is an important quality of any beer and the chemical content of the beer is responsible for that flavor. Aroma is also an extremely important part of the beer's trademark, so there is a strong interest by brewers in the volatile organic compounds (VOCs) in beer.

Some VOCs have a positive effect on aroma (attributes) and some have a negative effect (defects). The ability to characterize these in beer products before, during and after fermentation is an important tool in process control, quality assurance and product development. This application pack contains all the consumables needed to perform your analysis.

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APPLICATION NOTE

Gas Chromatography – Mass Spectrometry

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The Determination of Low Levels of Nitrosamines in Beer Using the Clarus 680 GC/MS and a D-Swafer System

Introduction

Nitrosamines are a class of compounds that are often found in food and other organic products. They are highly carcinogenic and many countries apply controls on the acceptable levels of these compounds in food. Nitrosamines are formed as food is heated through the reaction of amines with nitrites, which are sometimes added as a preservative.

Malt and its derivative products are of particular concern and beer represents (along with fried bacon and tobacco) the major source of ingested nitrosamines in humans. Historically, malt was roasted over open fires and the nitrogen oxide gases in the smoke would react with amines in the malt to form nitrosamines. Modern malt production uses indirect fire roasting and the levels of nitrosamines have consequently dropped significantly – by a factor of over 50x from malt produced 20 years ago.

Nitrosamines generated during malt production will pass into beer. Examples of maximum acceptable levels of nitrosamines in beer are 5 µg/kg in the United States, 0.5 µg/kg in Italy, Switzerland and Germany and 2-15 µg/kg in Russia.

The main compound that is monitored in malt and beer is nitrosodimethylamine (NDMA). This compound and its homolog, nitrosodiethylamine (NDEA) are the compounds targeted in this application note.

To determine NDMA and NDEA at low concentrations in beer typically involves a liquid-liquid extraction followed by a multi-step extensive sample clean-up regime and determination by gas chromatography, including a highly specific and selective detector, such as a thermal energy analyzer (TEA) detector.

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APPLICATION NOTE

Atomic Absorption

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Elemental Analysis of Beer by Flame Atomic Absorption Spectrometry with the PinAAcle 900 AAS

Introduction

Beer is a widely consumed beverage with both organic and inorganic components. The concentrations of the inorganic components may vary depending on raw materials and brewing processes. Knowledge of the type and concentration of inorganic components in beer is of considerable interest from various perspectives, as they may affect taste, appearance, product stability, and health of the consumer[1]. The determination of elements in beer by flame atomic absorption spectrometry (FAAS) is a well-known procedure[2]. For example, the American Society of Brewing Chemists (ASBC) in St. Paul, Minnesota, USA, is proposing the regular determination of calcium (Ca), copper (Cu), iron (Fe), and sodium (Na) in beer by FAAS[3].

FAAS has the benefit of providing precise and accurate measurements at a lower cost per element than more advanced elemental techniques, and also requires less operator training than many other trace elemental techniques. The PinAAcle™ 900 FAAS provides an intuitive, highly efficient system capable of simplifying analyses while maintaining peak performance and unmatched productivity.

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FIELD
APPLICATION
REPORT

ICP Optical Emmission

Beer Analysis Using the Optima ICP

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Introduction

Beer is one of the oldest beverages with references dating all the way back to 6000 B.C. The analysis of elements is an important parameter for determining the quality of beer. The analysis of beer is complicated by the presence of alcohol, dissolved solids and carbonation. Some elements affect the taste of beer, including Fe and Cu. These are usually found in very low concentrations, so the instrument's detection limits are important. Because of the low levels found, it is desirable to avoid dilution of the samples. Some elements are found at much higher concentrations, such as K, which can be several hundred mg/L. Inductively coupled plasma optical emission spectroscopy's (ICP-OES) multi-element capabilities, large dynamic range, and low detection limits (using axial viewing), make it ideal for the determination of metals in beer. The extensive linear range of ICP allows the analysis of both the low level elements as well as the major elements, without further dilution.

The direct analysis of beer by ICP can be challenging. The alcohol content requires matrix matching the standards to the samples containing ethanol. Also, the sample introduction system must be optimized for the volatile, organic ethanol component of the matrix. Due to high levels of dissolved solids, the nebulizer and injector must be capable of handling the samples without clogging. The carbonation in the beer samples must be removed to prevent out-gassing during thenebulization process and to eliminate poor reproducibility.

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APPLICATION NOTE

UHPLC

Authors

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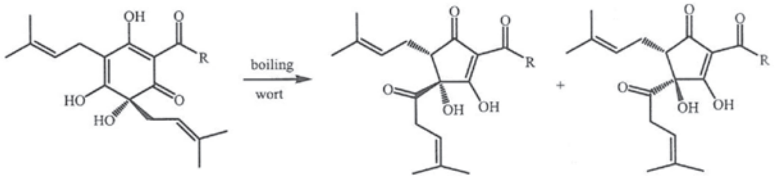
Determination of α-acids in Hops Using Third Party Software

Introduction

Hops are a major ingredient used in beer brewing. They preserve beer and provide it with its recognizable bitter taste and aroma. Hops come from a cone like plant called Humulus lupulus, which houses a lupulin gland containing resins and oils. The resins contain a number of α-acids that impart the bitter taste to most beers; the oils in large part give beers their aroma.

In beer breweries around the world, one essential part of quality control is ensuring that the type and amount of α-acids are the same from batch to batch, and that their transformation into the bitter iso-α-acids during the brewing process consistently gives individual brands their recognizable taste (Fig. 1).

This application note presents a straightforward method to determine the type and amount of α-acids in pellets from three hops varieties.



ICE 3 (**α-acids**)
CH(CH3)2 cohumulone
CH2CH(CH3)2 humulone
CH2 (CH3) C2H5 adhumulone

cis-iso-α-acids
cis-cohumulone
cis-humulone
cis-adhumulone

ICS- I3 (*trans*-iso-α-acids)
trans-cohumulone
trans-humulone
trans-adhumulone

Figure 1. Isomerization of hop α-acids to iso-α-acids during brewing.

APPLICATION NOTE

Gas Chromatography/
Mass Spectrometry

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Characterization of Hop Aroma Using GC/MS, Headspace Trap and Olfactory Port

Introduction

Hops are a critical ingredient in beer. They provide an important balance to the malt in the taste of many beers. They also aid the brewing process in precipitating out proteins, etc. during the boil. Hops also have preservative properties that help keep beer fresh and free from bacteriological attack.

Hops contribute to the taste of beer in three ways:

- Bittering – hops contain compounds such a humulones that are very insoluble in water but isomerize on boiling to form isohumulones, which are partially soluble and impart the bitter flavor to beer.
- Flavoring – compounds such as terpenes and esters provide the fruity, citrus, earthy, resiny flavors to many beers.
- Aroma compounds – these are the volatile organic compounds that migrate into the vapor above the head of beer and gives the beer its characteristic smell. This can be flowery, citrusy, fruity, etc. They form a very important part of the overall flavor of beer.

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APPLICATION NOTE

Gas Chromatography

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Aroma Study of Potable Spirits

Introduction

The production of whisky requires maturation in wooden casks for the full development of the finished product's character. Subtle differences in the casks' conditioning can produce quite different flavors and aromas that require skillful blending to achieve a consistent product. The PerkinElmer TurboMatrix™ headspace trap system coupled with a Clarus® SQ 8 GC/MS and SNFR™ olfactory port is an effective means of identifying low concentration volatile organic compounds (VOCs) in potable spirits.

Manufacturing whisky is a lengthy process due to Scottish law, which mandates that the distilled spirit be matured in oak casks for a minimum of three years and one day before it is bottled. During the maturation process the spirit takes on a distinct character in each cask, which must then be blended to give the recognized finished product. Much of the blending is performed by the master blender, a craft that can take 12 years in apprenticeship. One of the key characteristics of whisky enjoyment is the aroma from the spirit with a recently designed copita (a tulip-shaped sherry glass), having been developed to maximize this experience. Functional groups that give character to whisky include alcohols, esters, acids and carbonyls, and the odor thresholds of these analytes of interest vary greatly by each group.

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APPLICATION NOTE

UHPLC

Author

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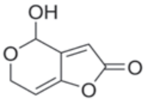
Analysis of the Mycotoxin Patulin in Apple Juice Using the Flexar FX-15 UHPLC-UV

Introduction

Patulin is produced by various molds, which primarily infect the moldy part of apples. Removing the moldy and damaged parts of the fruit may not eliminate all the patulin because some of it may migrate into sound parts of the flesh. Also, patulin can be produced within the fruit, even though it may not be visibly moldy. If moldy apples are used to produce apple juice, the patulin goes into the juice. It is not destroyed by heat treatments such as the pasteurization process. Patulin is a natural human toxin and therefore can have genetic affects within cells, including a developing fetus, the immune system and the nervous system. The recommended advisory level is 50 µg of patulin/kg in apple juice [50 parts per billion (ppb)].[1] Hydroxymethylfurfural (HMF), also 5-(Hydroxymethyl)furfural, is an organic compound derived from dehydration of sugars. HMF has been identified in a wide variety of heat-processed foods including milk, fruit juices, spirits, honey, etc.[2]



Synonyms: 2-Hydroxy-3,7-dioxabicyclo[4.3.0]nona-5,9-dien-8-one
Formula: $C_7H_6O_4$
MW: 154.12
MP: 110 °C

Figure 1. Structure and properties of patulin.

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APPLICATION NOTE

HPLC/ICP-MS

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Determination of Arsenic Speciation in Apple Juice by HPLC/ICP-MS Using the NexION 300/350

Introduction

Recent media coverage has brought the issue of arsenic (As) in apple juice into public awareness. Because arsenic can exist in several forms (some toxic, some non-toxic), it is important to identify the individual forms present in the juice. This is most easily accomplished through the use of liquid chromatography (HPLC), to separate the species, and inductively coupled plasma mass spectrometry (ICP-MS), to detect them. A challenge with juice analysis is the high level of sugars which can affect both the chromatography and the ICP-MS.

This work demonstrates an HPLC/ICP-MS method for the separation and determination of arsenic species in a variety of apple juices.

Experimental

Samples and Sample Preparation

The apple juice samples were purchased from local grocery stores. Sample preparation involved a 2-fold dilution in the aqueous component of the mobile phase (i.e. without the methanol) with pH adjusted to 7.0 prior to analysis. All quantitative measurements were made against external calibration curves, with calibration standards prepared the same way as the samples.

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APPLICATION NOTE

Mass Spectrometry

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Rapid Screening of Adulteration in Pomegranate Juice with Apple Juice Using DSA/TOF with Minimal Sample Preparation

Introduction

Pomegranate juice is in high demand due to its well documented health benefits. Studies based on a significant body of scientific research conducted on authentic pomegranate juice have established that pomegranate juice has superior antioxidant activity compared to other popular fruit juices such as grape, orange and apple[1,2,3]. These superior health benefits have led to a meteoric rise in the popularity of pomegranate juice over the last 10 years. This has resulted in pomegranate juice having a significantly higher cost than other juices, especially in comparison to apple, orange and grape juice. This high cost has provided an economic incentive for the adulteration of pomegranate juice[4]. There are a number of of other reasons for adulteration including: customers demanding their suppliers reduce costs; there comes a point when a supplier may adulterate the product to lower the cost and maintain a workable margin. Another reason for pomegranate juice adulteration is that it is in high demand and short supply, leading to incentives to extend limited supplies by the addition of other cheaper fruit juices.

The adulteration of pomegranate juice is accomplished by using cheap ingredients. One of the common adulterants of pomegranate juice is a cheaper so called filler fruit juice such as apple juice. The advantage of adulterating pomegranate juice with apple juice is that it is clear and provides no noticeable change to the sugar content. Organic acid analysis plays a fundamental role in testing the authenticity of pomegranate juice. One organic acid of interest is malic acid. Although it is present in both pomegranate and apple juice, the amount in apple juice is significantly higher than in pomegranate juice, 4.5 g/L and 0.57 g/L respectively[5,6]. Therefore, the adulteration of pomegranate juice with apple juice can be detected by the elevated levels of malic acid present in the juice. Analytical methods used routinely for

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APPLICATION NOTE

Liquid Chromatography/ Mass Spectrometry

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Shelton, CT

Rapid Analysis of Apple Juice Adulteration with Pear Juice Using LC/TOF

Introduction

Adulteration of fruit juices is quite common in the market place and is often done through juice substitution. Pear juice has a slightly lower market value than apple juice yet both have similarities in major chemical composition, making pear juice an ideal adulterant for apple juice. Both juices have similar carbohydrate, organic acid and amino acid levels, therefore the detection of apple juice adulteration with pear juice, based on these compounds, has limited applicability. According to various references[1-4], the phenolic profiles in pear and apple juice are slightly different. One of the phenolic compounds, arbutin, can be used as a marker compound to detect adulteration. The structure of arbutin is shown in Figure 1. In this application note, we present a rapid method for detecting apple juice adulteration with pear juice using LC/TOF, which requires minimal sample preparation.

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APPLICATION NOTE

Mass Spectrometry

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Rapid Screening of Adulteration in Pomegranate Juice with Grape Juice Using DSA/TOF with No Sample Preparation

Introduction

Pomegranate juice is in high demand due to its well documented health benefits. Studies based on a significant body of scientific research conducted on authentic pomegranate juice have established that pomegranate juice has superior antioxidant activity compared to other popular fruit juices such as grape, orange and apple[1,2]. These superior health benefits have led to a meteoric rise in the popularity of pomegranate juice over the last 10 years. This has resulted in pomegranate juice having a significantly higher cost than other juices, especially in comparison to apple, orange and grape juice. This high cost has provided an economic incentive for the adulteration of pomegranate juice[3]. There are a number of other reasons for adulteration including: customers demanding their suppliers reduce costs; there comes a point when a supplier may adulterate the product to lower the cost and maintain a workable margin. Another reason for pomegranate juice adulteration is that it is in high demand and short supply, leading to incentives to extend limited supplies by the addition of other cheaper fruit juices.

One of the common adulterants of pomegranate juice is grape juice. Grape juice is added as a sweetener and coloring agent substitute for natural pomegranate color. Organic acid analysis plays a fundamental role in testing authenticity of pomegranate juice. One of the organic acids, tartaric acid, is present in large amounts in grape juice (1 g/L) but it is absent in pomegranate juice[4,5]. Therefore, the presence of tartaric acid in pomegranate juice can be used as an indicator of grape juice adulteration. Analytical methods used routinely for organic acid analysis in fruit juices are based on liquid chromatography (reverse phase or ion exchange) coupled to UV detection[6] or mass spectrometry[7,8]. These measurement techniques are either expensive or time consuming, or both and require extensive method development and sample

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CASE STUDY

Food Fraud

Pomegranate Juice Adulteration

Introduction

Pomegranate juice's popularity has skyrocketed in the last 10 years. This has been due to a combination of the perceived health benefits of consuming the juice's various antioxidant compounds (punicalagin, anthocyanins and ellagic acid) and its increased mainstream availability through Western pomegranate producers. This increase is highlighted by the rise in the consumption of 8-ounce servings of pomegranate juice in the U.S., which went from 75M servings in 2004 to 450M servings by 2008.[1] Interestingly, this data indicates that in 2004, there was 50:50 pure-to-blended pomegranate juice consumption, whereas in 2008, 100% pomegranate juice made up 75% of that consumed.[1] Popular juice blends, such as apple and grape, are less bitter and can make the overall juice taste more pleasant to those new to pomegranate. These blends have an additional advantage of being cheaper than pure pomegranate juice. Whereas a gallon of pomegranate juice concentrate costs \$30-60, a gallon of apple or grape juice is between \$5-7. This means if a pomegranate juice product is labeled as a blend with apple and grape juice, the consumer can expect to pay less than the cost of pure pomegranate juice.

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APPLICATION NOTE

Gas Chromatography/
Mass Spectrometry

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The Qualitative Characterization of Fruit Juice Flavor using a TurboMatrix HS Trap and a Clarus SQ 8 GC/MS

Introduction

The PerkinElmer® TurboMatrix™ Headspace Trap system coupled with a Clarus® SQ 8 GC/MS is a very convenient means of identifying low concentration volatile organic compounds (VOCs) in foodstuffs. In this application note, the VOCs in various fruit juices were investigated. Sample preparation simply involved dispensing a fixed volume of fruit juice into a sample vial and sealing it. The analysis was fully automated.

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APPLICATION NOTE

Gas Chromatography

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A Method for the Quantification of Ethanol Content in Consumable Fruit Juices by Headspace Injection

Introduction

The definition of an alcoholic beverage in the United States of America is a beverage that contains in excess of 0.5% ethanol by volume that is intended for consumption alone or when diluted. Production of alcohol has been long established in society with many styles that take advantage of the metabolism of sugars into ethanol. While the production of ethanol is desirable for alcoholic beverages, it is undesirable for other beverages which contain sugars that do not wish to be sold as an alcoholic beverage. Such sugar metabolism is naturally occurring and is well understood to happen in raw fruit as well as processed juice and can vary by type, variety and maturation in the growing season.

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APPLICATION NOTE

Liquid Chromatography/
Mass Spectrometry

Authors

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Rapid Quantitative Analysis of Carbendazim in Orange Juice using UHPLC Coupled to the AxION 2 TOF Mass Spectrometer

Introduction

Fungicides in imported orange juice have been on the national stage in recent months, creating a need for simple and rapid detection methods.

When a private company alerted the FDA to the presence of the fungicide, carbendazim, in orange juice imported from Brazil, the agency expanded its testing of imports for this residue, which is illegal in the United States. In response to the increased concern, we have developed a quick and straightforward method of analysis and quantitation of carbendazim at concentrations below 10 parts per billion.

We present a method for detecting both targeted analytes, such as carbendazim, as well as non-targeted components, by taking advantage of the full-spectrum, high mass accuracy data provided by the AxION® 2 time-of-flight (TOF) mass spectrometer (MS) using an Ultraspray™ 2 ionization source with lock mass for on-the-fly calibration. Unlike a triple quadrupole instrument, which requires predefined targets, a TOF instrument collects accurate mass and high resolution data across a full spectrum without a loss in sensitivty allowing analysis and identification of an unlimited number of compounds. Any emerging analytes of concern can be detected in pre-existing data simply by searching for the exact mass of the analyte of interest, and can quickly be confirmed by retention-time matching.

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APPLICATION BRIEF

Near-Infrared Spectroscopy

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Lauren McNitt

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Verification of Coffee Roast Using Fourier Transform Near-Infrared Spectroscopy

Introduction

With approximately 100 million people drinking coffee daily, it is no surprise that $18 billion are spent on coffee, frappes, and cappuccino annually in the United States. Grown around the world, coffees from different nations each have a distinct flavor. In most regions, beans are grown on small local farms then brought to market to sell. At the market, a buyer carefully inspects the beans before purchase and sends them off to be processed.

Processing coffee includes roasting the beans, as specific beans are roasted at different levels. The roast can be a dark, medium, or light with different levels within each. To determine what level of roast the coffee is while roasting, the master roaster has to listen for cracking noises or remove sample beans to distinguish the roast by color. When master roasters use the cracking method, there are two distinct noises they listen for. The first crack is caused by water and carbon dioxide bursting out of the bean; it also signifies the beginning of the bean becoming a light roast. The second crack is created by the cellulose matrices in the bean beginning to fracture. This also represents the beginning of the dark roast level. Near-Infrared (NIR) spectroscopy is shown here to be a fast and simple technique for measuring the roast levels of coffee beans and ground coffee.

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APPLICATION NOTE

Thermal Analysis

Dynamic Mechanical Analysis of Coffee

Summary

This application note demonstrates the ability of DMA to investigate a complex powder formulation. Both granulated instant coffee and granulated filter coffee were analyzed by DMA. A multi-frequency experiment clearly shows complex relaxation processes and other phase transitions. When run over a large temperature range, relaxations were observed as well as reproducible events below -150 °C. As the coffee is in powdered form, the Material Pockets were used to hold the sample in the PerkinElmer® DMA 8000.

Introduction

Dynamic Mechanical Analysis (DMA) is one of the most appropriate methods to study amorphous materials. The glass transition (Tg) is a key process in any material, and can be observed with ease by DMA. This technique provides very revealing information about these relaxations through the tan δ vs. temperature data. Coffee, when in the final marketed state, is often in a powdered or granular form. This applies to both instant and filter coffee. Powders can be easily investigated in the DMA 8000 by using Material Pockets which sandwich the powder in a stainless steel prior to measurement.

DMA works by applying an oscillating force to the material and the resultant displacement of the sample is measured. From this, the stiffness can be determined and the modulus and tan δ can be calculated. Tan δ is the ratio of the loss modulus to the storage modulus. By measuring the phase lag in the displacement compared to the applied force it is possible to determine the damping properties of the material. Tan δ is plotted against temperature and glass transitions are normally observed as a peak.

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APPLICATION NOTE

Gas Chromatography/
Mass Spectrometry

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Coffee Characterization Using Clarus SQ 8 GC/MS, TurboMatrix HS Trap and GC SNFR Olfactory Port

Introduction

Coffee is a very popular drink in most parts of the world and is one of the most traded agricultural commodities on the planet. The drinking of coffee, however, is a fairly recent activity. Although its origin may be attributed to Ethiopia a thousand years ago, its popularity as a beverage really started in the Middle East around the start of the 17th century.

Part of its popularity is due to the stimulating effect of its caffeine content (a cup of coffee may contain as much as 150 mg) and part is due to its rich complex taste. The taste of a cup of coffee depends on many factors – the coffee bean variety and horticulture and the way the beans are stored, roasted, ground and brewed. Even the water used to make the coffee can have an effect on its flavor.

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APPLICATION BRIEF

Thermogravimetric Analysis – GC Mass Spectrometry

TG-GC/MS Technology – Enabling the Analysis of Complex Matrices in Coffee Beans

Introduction

The combination of a thermogravimetric analyzer (TGA) with a mass spectrometer (MS) to analyze the gases evolved during a TGA analysis is a fairly well-known technique. In cases of complex samples, TG-MS often results in data in which it is nearly impossible to differentiate gases that evolve simultaneously.

Combining TGA with gas chromatography mass spectrometry (GC/MS) allows for a more complete characterization of the material under analysis and precisely determines the products from the TGA.

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APPLICATION NOTE

Atomic Absorption

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Analysis of Pb, Cd and As in Tea Leaves Using Graphite Furnace Atomic Absorption Spectrophotometry

Introduction

Tea is drunk by about half of the world's population. It is widely cultivated and consumed in Southeast Asia. Tea is rich in many trace inorganic elements.[1,2] In addition to many essential elements required for human health, some toxic elements may also be present in tea leaves. This could be due to polluted soil, application of pesticides, fertilizers or industrial activities. There is often little information available about the safety of tea leaves and finished tea products with respect to heavy metal contamination. Due to the significant amount of tea consumed, it is important to know the toxic metal contents.

The toxicity and effect of trace heavy metals on human health and the environment has attracted considerable attention and concern in recent years. Among the heavy metals, lead (Pb), cadmium (Cd) and arsenic (As) are especially toxic and are harmful to humans even at low concentrations. They have an inherent toxicity with a tendency to accumulate in the food chain and a particularly low removal rate through excretion.[3] Exposure to heavy metals above the permissible level can cause high blood pressure, fatigue, as well as kidney and neurological disorders. Heavy metals are also known to cause harmful reproductive effects.[4]

A major challenge in the analysis of tea leaves is the extremely low analyte levels and the very high matrix levels. For many years, graphite furnace atomic absorption spectrophotometry (GFAAS) has been a reliable technique and the preferred method for this analysis. The use of longitudinal Zeeman background correction and matrix modifiers help to achieve extremely low detection limits in high matrix samples such as tea leaves, making GFAAS an indispensible tool for carrying out such analyses.

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APPLICATION NOTE

ICP-Mass Spectrometry

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Shelton, CT

TotalQuant Analysis of Teas and Wines by ICP-MS

Introduction

TotalQuant is a software feature unique to the ELAN® ICP-MS systems for quantifying 81 elements in a sample by interpretation of the complete mass spectrum. Measuring the full mass range takes only a minute, and the spectral interpretation itself takes a few seconds. During the TotalQuant analysis, each element is assigned a response value (cps/ppm) which is updated when a calibration is performed. Even though TotalQuant is an ideal tool for semiquantitative analysis during method development, it can also be used for a final material characterization.

When using TotalQuant, spectral interpretation is performed automatically by the software and intensities are assigned to elements after correction for interferences on individual isotopes. Intensities are compared with a stored response table to convert them into concentrations. TotalQuant, being a semiquantitative program, gives quantitative results typically within +/-25% of the real value in simple matrices.

APPLICATION BRIEF

FT-NIR Spectrometry

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Rapid Testing for Adulteration of Yogurt Candy using Near-infrared Spectroscopy and Adulterant Screen

Introduction

Melamine is an adulterant commonly found in milk, as it can increase the nitrogen content. Therefore, its apparent protein content, resulting in a better market price. Melamine adulteration can be fatal, as was highlighted in 2008 when six infants died due to melamine adulteration in milk powder and thousands were sickened in China. Consequently, there have been stricter regulations globally and improved testing methods including the use of the PerkinElmer DairyGuard™ instrument for powdered milk testing.

However, cases of melamine adulteration are still appearing in other products. This year in Guangdong Province, China, 25 tons of yogurt candy tablets were seized as they were found to contain melamine. What follows is a description of a near-infrared (NIR) testing method of yogurt candy for melamine adulteration.

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APPLICATION NOTE

Thermal Analysis

PerkinElmer Pyris 1 TGA

Better Quantitative Analysis of Chewing Gum with AutoStepwise TGA

Introduction

The gum base of chewing gums is a complex mixture of a number of components, including: PVAC (poly vinyl acetate), natural elastomers, glycerin, softening agents, and carbonates. In addition, chewing gum contains flavoring agents, sucrose or other sweeteners and colorants. These various components provide the desired textural and viscoelastic properties to the chewing gum. The correct combination of the gum formulation provides the end characteristics to the chewing gum, including: stickiness, softness and chewability.

It is desired to have a means of characterizing the composition of chewing gum for quality assurance purposes. One of the best means of detecting components in a complex formulation, such as chewing gum and other foods, is with thermogravimetric analysis (TGA).

TGA is a well-established method for the characterization of materials. The technique measures sample mass as a function of temperature or time and provides useful decomposition temperature information.

However, for complex formulations or multi-component materials, standard (constant heating) TGA may not possess the necessary resolution for separating out the weight losses associated with the various components. What is required is a TGA, which can provide enhanced resolution or separation using AutoStepwise isothermal methods.

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APPLICATION NOTE

Thermal Analysis

Characterization of Fats in Cookies Using Power Compensation DSC

Introduction

Differential scanning calorimetry (DSC) is a useful technique for the characterization of food products, including:

- The gelatinization and staling (retrogradation) behavior of starches
- Polymorphism of fats such as cocoa butters and chocolate
- Effects of moisture content or absorbed moisture
- Aging effects
- Protein denaturation
- Determination of fat content or solid fat index (SFI)

The processing and handling behavior of food fats has been found to depend upon the solid-to-liquid fat ratio in the food sample. Many rheological or flow properties, and their resultant effect on the texture of the final product, stem from this fat ratio index.

The study of the fat content and the nature of the fats of foods is becoming increasingly more important due to health considerations, especially with regards to the level of solid fats, saturated fats and trans fats in food products. There is a variety of fats with different levels of solid fats available in food products. An example of this is the Oreo® Cookie where there is the regular Oreo® and the reduced fat version. There are also Oreo®-like cookies with no solid, hydrogenated fats present.

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APPLICATION NOTE

FT-IR Spectroscopy

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Investigating Phase Transitions with Variable Temperature ATR

Introduction

Heating and cooling cause phase changes in many materials with consequent effects on their physical properties. IR spectroscopy is a powerful tool for studying these changes since the spectra are sensitive to variations in both intermolecular and intramolecular interactions. Changes in the IR spectra due to temperature variations can be correlated with structural changes and the spectra can be associated with specific crystalline forms. For example, any transition from a crystalline to an amorphous phase results in a broadening of bands as the molecules are in more varied environments. In crystalline materials, a change from one polymorph to another generally causes band shifts and splitting. For many materials transmission and external reflection measurements are not practicable without some sample preparation but ATR can often be used directly. In this note we illustrate the use of heated ATR to follow thermal changes in chocolate, which is largely a suspension of sucrose and cocoa solids in a matrix of cocoa butter. Chocolate and cocoa butter both have several polymorphs with melting points between 17 and 37 °C.[1] The polymorphic form has to be carefully controlled in manufacture because of its importance for storage and its major contribution to the sensory experience when eating chocolate.

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APPLICATION BRIEF

FT-IR NIR Spectrometry

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Single NIR Measurement for the Detection of Adulteration and Measurement of Important Parameters in Cocoa Powders

Introduction

Cocoa powder is a product regularly used within the personal care, food, and beverages sectors. There have been reports indicating several health benefits of cocoa, from lowering blood pressure antioxidants to containing essential fatty acids, fiber, and minerals.

There have been cases of cocoa adulteration reported in Eastern Europe and across the world. In a recent case, samples contained 20% less cocoa compared to values listed on the labels, which was found to be fraudulent activity by the supplier.[1]

The initial method used to assess the quality of cocoa beans is the cut-and-taste test, although this is a subjective technique. Liquid chromatography methods are also often used for testing cocoa powder, but they are time consuming and can be complicated. A much faster and easier technique to verify the authenticity of cocoa is Near-Infrared (NIR) spectroscopy.

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APPLICATION NOTE

Gas Chromatography/
Mass Spectrometry

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Solid Phase Extraction and GC/MS Analysis of Melamine Adulteration in Dairy Products

Introduction

In September 2008, melamine again made global headlines with contamination and adulteration of dairy products in China. This incident occurred about 18 months after melamine contamination of pet foods. During the initial melamine scare, gas chromatography/mass spectrometry (GC/MS) analysis was used successfully in testing finished food products as well as raw materials. This analysis is presented in the PerkinElmer application note "Screening for Melamine Adulteration in Protein-Based Foods by GC/MS"[1].

When the melamine in milk crisis began, similar test methods were used to test baby formula and other dairy products. The sample matrix of milk and dairy products is, however, much different than that of pet foods, with a much higher content of fat and sugar. This difference in matrix required that sample preparation methods be modified from those used in GC/MS analysis of pet foods. The major modification necessary is solid phase extraction (SPE) of the sample extract to remove the matrix of the milk. This paper will present the modifications necessary to successfully analyze dairy products for melamine with GC/MS. Additionally, GC/MS analysis of the data will support the method modifications.

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APPLICATION NOTE

Atomic Absorption

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Accurate Determination of Lead in Different Dairy Products by Graphite Furnace Atomic Absorption Spectrometry

Introduction

Milk is one of the basic food groups in the human diet, both in its original form and as various dairy products. The Chinese contaminated baby formula scandal in 2008 has increased public awareness of contamination possibilities, and has lead to tighter supervision of dairy products as China is faced with demands – both from home and abroad – to improve its food safety record. It is well-known that lead (Pb) is toxic and causes damage to the nervous system; it has a particularly detrimental effect on young children[1] and it has become a cause of major concern since the 1970s. As per World Health Organization (WHO) standards, the permissible limit of lead in drinking water is 10 µg/kg (parts per billion, ppb). Following an in-depth review of the toxicological literature, the Chinese guideline for maximum levels of lead content is set at 20 µg/kg (ppb wet weight) in infant formula (use of milk as a raw material measured by fluid milk diluted from powder, referring to the product ready-to-use) and at 50 µg/kg (ppb) in fresh milk, respectively.[2]

Lead analysis has traditionally been one of the major applications of graphite furnace atomic absorption spectrometry (GFAAS) worldwide. Currently, the Chinese regulatory framework approved standard methods for lead analysis has set GFAAS as the technique for the compulsory arbitration in food testing.[3] In order to ensure protection of consumers, analysis should be sensitive, efficient, and cost-effective so that more effective monitoring can be accomplished. Because GFAAS is a mature technique, it is well-understood and routinely used by technicians and suitable for this determination. Sample preparation is an important part of an analysis and yet can be time consuming.

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Milk Authenticity – Organic vs Non-organic

With increasing concerns over contaminants in milk, both intentionally and unintentionally added, a growing number of people are switching to organic milk (sales of whole organic milk were up 17% between January and October of 2011 in the U.S. with reduced fat organic milk up 15%).[1] This surge in popularity, coupled with high food and fuel prices, has caused shortages in the supply of organic milk.[2] With demand therefore outstripping supply, and a gallon of organic milk costing anywhere from 25% to 100% more than conventional milk, the selling of conventional milk as organic is an attractive proposition to fraudsters. In the U.S. and E.U., the labelling of organic products has meant stricter policing of farming practices but this is not the case with all countries. Furthermore, with the growing export of organic milk powders, these fake organic milk powders can find their way into the West through distributors or through processed foods, such as chocolates, which will also command a higher price if claiming to be organic. While these substitutions invariably do not cause health problems it is still fraud, with consumers not getting what they paid for and hardworking organic farmers losing business and having profit margins eroded.

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APPLICATION NOTE

Near Infrared Spectroscopy

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DairyGuard: Augmenting Nutritional Testing of Milk Powder with Adulterant Screening

Introduction

Milk powder is one of the most widely traded food commodities, with over 2.5 million metric tons exported annually[1], and is used in a huge array of food products, from infant formula to baked goods and confectionary. Unfortunately, dairy products are also a frequent target of food fraud, with 137 cases of economically motivated adulteration worldwide recorded by the United States Pharmacopoeia in 2011-2012[2]. The value of milk powder is linked to its protein content, and standard methods for protein analysis rely on a simple nitrogen assay, with the protein concentration inferred from the nitrogen content. Consequently, the addition of chemicals rich in nitrogen can artificially increase the apparent protein and thus the price demanded.

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APPLICATION BRIEF

ICP - Mass Spectrometry

Authors:

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Shelton, CT

The Elemental Analysis of Milk Powder with NexION 300/350 ICP-MS

Introduction

The elemental capabilities and dynamic range of inductively coupled plasma mass spectrometry (ICP-MS) makes it ideally suited for the analysis of food materials. The ultratrace detection limits of ICP-MS permit the determination of low-level contaminants, such as Pb, As, Se, and Hg, while the macro-level nutritional elements, such as Ca, Mg, K, and Na, can be quantified using the extended dynamic range capability of ICP-MS which provides the ability to measure concentrations over nine orders of magnitude. However, there are still a number of challenges to overcome, including complex sample matrices, high levels of dissolved solids, and interferences. With the proper ICP-MS instrumental conditions and design, all of these issues can be overcome, allowing for the successful analysis of food samples, as described elsewhere[1]. This work will focus on the analysis of non-fat milk powder, an important food staple, especially in the developing world.

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APPLICATION NOTE

LC/Mass Spectrometry

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LC/MS Study of Casein Proteins in Milk

Introduction

We have developed a method for extracting and measuring the masses of proteins in milk by using liquid chromatography coupled to electrospray time-of-flight mass spectrometry (LC-TOF MS). This method was used to look at the patterns and masses of the casein proteins in cow milk.

Methods

Proteins were extracted from milk by the addition of a two-fold volume of an aqueous denaturing solution containing 8 M urea, 165 mM TRIS, 44 mM sodium citrate and 0.3% (v/v) mercaptoethanol, followed by filtration[1]. This method extracted fat soluble casein proteins in addition to whey proteins.

APPLICATION NOTE

LC/Mass Spectrometry

Authors:

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Waltham, MA

Milk Adulteration: Detecting Species-Specific Proteins by LC/MS

Introduction

The adulteration of milk via the addition of low molecular weight compounds, such as melamine for economically motivated fraud, is a well-known concern[1]. Methods for adulteration detection have been examined extensively using, amongst other tools, LC/MS techniques. Current methods tend to focus on small molecule adulteration; however, large molecule adulteration with other animal or plant proteins is becoming increasingly common and is not yet regulated within the dairy industry.

One of the simplest forms of milk adulteration is to dilute more expensive milks from species, such as sheep and buffalo, with less expensive cow milk. We have developed a method for measuring the addition of bovine milk to more expensive milks (goat, sheep, buffalo and camel) by using liquid chromatography coupled to electrospray time-of-flight mass spectrometry (LC-TOF MS) to detect species-specific marker proteins in the milk.

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APPLICATION NOTE

Liquid Chromatography/
Mass Spectrometry

Author:

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Shelton, CT

Analysis of Quaternary Ammonium Compounds (QACs) as Possible Disinfectant Residues in Milk by LC-TOF

Introduction

Quaternary ammonium compounds (QACs) have the basic structure NR_4^+. Those possessing R groups with long alkyl chains are known to be especially effective as antimicrobial agents and particularly useful for the disinfection of containers and surfaces. This is particularly relevant in the milk industry, as QACs are typically used to disinfect the insides of tanks used for transporting milk from farms to processing plants. If significant QAC residues are left behind after tank disinfection, these compounds may leach into the milk and, ultimately, may get into the store-bought milk supplies at levels compromising personal health. Recent data points to nearly 12% of all monitored milk to be tainted with QACs.[1]

The primary QACs that may be found in milk are benzyldimethyldodecylammonium chloride (BAC 12), benzyldimethyltetradecylammonium chloride (BAC 14), benzyldimethylhexadecyl ammonium chloride (BAC 16) and didecyldimethylammonium chloride (DDAC). Their chemical structures and expected parent masses in solution are shown in Figure 1.

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APPLICATION BRIEF

Near-Infrared Spectroscopy

Author:

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Shelton, CT

Use of NIR Spectroscopy and Adulterant Screen for the Detection of Common Adulterants in Milk

Introduction

The value of milk on the open market is linked to its protein content, and standard methods for protein analysis rely on a simple nitrogen assay, with the protein concentration inferred from the nitrogen content. Consequently, the addition of chemicals rich in nitrogen, such as urea, can artificially increase the apparent protein content and thus the price demanded. Urea occurs naturally in milk and is typically present at levels of about 0.02% - 0.05%. Higher levels of urea in milk are present only in cases of adulteration. Cane sugar is another known milk adulterant used to increase its carbohydrate content and weight. This allows extra water to be added into the milk without detection from a standard lactometer test for milk quality.

NIR spectroscopy coupled with PerkinElmer's Adulterant Screen™ is shown here to be capable of detecting adulterants intentionally or accidentally added to milk.

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APPLICATION NOTE

Liquid Chromatography/
Mass Spectrometry

Authors:

Robert Seward

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PerkinElmer, Inc.
Waltham, MA

Differentiation of Cultivars of Spanish Olive Oils Using Multiple LC/MS Analysis Methods

Introduction

For high value olive oil, the provenance may have a dramatic effect on the price. Falsifying the origin of oils is financially an attractive proposition to fraudsters. The patterns of organic components in olive oils relate to the geographic origin, cultivar and processing methods for the olives. In this study a statistical analysis of the chemical components of authentic monovariatal virgin olive oils from Spain was used to determine markers for the cultivars. These markers could be used to confirm the provenance and origins of olive oil samples.

Different LC/MS methods were used to measure the levels of a large number of polar and non-polar organic components in the oils. Results from the separate analyses were combined with data fusion. A statistical comparison with the combined results improved the discrimination of cultivar groups, when compared to a single LC/MS analytical method.

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APPLICATION NOTE

Liquid Chromatography/ Mass Spectrometry

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Markers for Spanish Olive Oil Cultivars – Statistical Analysis of Polar Compounds from LC/MS Results

Introduction

For high value olive oil, the country of origin and type of olive cultivar has a dramatic effect on the price. Falsifying the provenance of such oils is an attractive proposition to fraudulent suppliers.

Polar components such as phenolic acids and lignans are present in olive oils.[1] Secoiridoids; phenolics which include an elenolic acid moiety, are unique to the *Olearaceae* family. These compounds are known to have beneficial health effects due to their antioxidant properties and are associated with the organoleptic properties. Measuring the levels of these polar components in oils from different cultivars and geographic origins is of interest for nutritional and authenticity reasons.

In this study a statistical analysis of the polar chemical components of olive oils was used to determine potential markers for olive cultivars. These markers could be used to confirm the provenance of unknown samples.

APPLICATION NOTE

Mass Spectrometry

Author:

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Shelton, CT

Rapid Measurement of Olive Oil Adulteration with Seed Oils with Minimal Sample Preparation Using DSA/TOF

Introduction

Olive oil is a valuable product that is traditionally produced in Mediterranean countries and now in the United States (particularly California), and has nutritional advantages concerning cardiovascular disease prevention[1,2]. Due to its high nutritional value, it is offered at a higher price than other seed oils. For this reason, it is sometimes adulterated with other cheaper seed oils.

Olive oil and other oils are composed mainly of triacylglycerols. These molecules are derived from the esterification of three fatty acid molecules with a glycerol molecule. The main triglyceride in olive oil is triolein (OOO); whereas the main triglyceride in seed oils (soybean, corn and sunflower) is trilinolein (LLL). Therefore, the measurement of ratio of trilinolein to triolein in olive oil can be used as a way to detect its adulteration with soybean and other seed oils, such as corn and sunflower, which have a higher content of trilinolein and lower amount of triolein in comparison to olive oil[3]. Using this strategy with a Direct Sample Analysis™ Time-of-Flight mass spectrometry system (DSA/TOF), we detected adulteration of olive oil with soybean and corn oil. In the past, methods employing gas chromatography/mass spectrometry (GC/MS) and high performance liquid chromatography (HPLC) hyphenated to MS (HPLC/MS) were implemented for this purpose[4-7]. These methods are time consuming, expensive and require extensive sample preparation, method development and derivatization. In this work, we demonstrated that the AxION® Direct Sample Analysis (DSA™) system integrated with the AxION 2 Time-of-Flight (TOF) mass spectrometer can be used for rapid screening of adulteration of olive oil with seed oils such as soybean and corn oil with minimal sample preparation.

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APPLICATION NOTE

Near-Infrared Spectroscopy

Authors:

Jorge Puente

Ben Perston

PerkinElmer, Inc.
Shelton, CT

Quality Control of Olives by Near-Infrared Spectroscopy and AssureID Software

Introduction

Olive oil is an increasingly popular food product worldwide, with global production exceeding 3.0 million tons in 2011 and showing steady annual growth. Despite these huge volumes, however, margins are relatively small in olive oil production so quick and easy analysis of oil quality is vital to maintain process efficiency. Rapid, reliable analysis can contribute to process and quality improvements in numerous ways. For example,

- Assessment of raw olive acceptability. If the olives have been collected from the ground rather than fresh from the tree, they may be of poor quality with high acidity and hence lower value.
- Measurement of water and oil content. These parameters determine the price of the olives, with those having a greater oil content commanding a higher price.
- Process optimization. After extracting the oil, the remaining pulp or by-product (called alperujo in Spain) should have only minimal oil content, typically around 2% or less. If the oil exceeds this level, a problem with the process is indicated.

This note describes how a PerkinElmer Frontier™ FT-NIR spectrometer and AssureID software have been used by an olive oil producer in Spain to improve productivity by implementing the above analyses within their routine production.

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APPLICATION NOTE

UV/Vis Spectroscopy

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Determination of Olive Oil Purity and Degree of Oxidation using the LAMBDA XLS

Introduction

Olive oil is well established in the food industry. Demand continues to grow not only because of its distinct flavor, but also because of an increased awareness of its health benefits. In fact, the FDA allows producers of olive oil to place a health claim on their products because there is some scientific evidence to support a risk reduction of coronary heart disease by consuming a higher proportion of monounsaturated fat in one's diet. This is significant because olive oil is considerably rich in monounsaturated fats, most notably oleic acid. It is therefore of interest to producers to know the quality of the oil, its state of preservation, and changes brought about in it by technological processes.

The quality of the olive oil is studied by measuring the characteristics of the absorption bands between 200 and 300 nm. These are frequencies related to conjugated diene and triene systems. A low absorption in this region is indicative of a high-quality extra virgin olive oil, whereas adulterated/refined oils show a greater level of absorptions in this region.

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APPLICATION NOTE

Gas Chromatography/
Mass Spectrometry

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The Determination of Low Levels of Benzene, Toluene, Ethylbenzene, Xylenes and Styrene in Olive Oil Using a TurboMatrix HS and a Clarus SQ 8 GC/MS

Introduction

Levels of benzene, toluene, ethylbenzene, xylenes and styrene (BTEXS) are a concern in olive oil. These compounds find their way into olive trees and hence into the olives and olive oil mainly as a result of emissions from vehicles, bonfires, and paints into ambient air near the orchards.

Various methods have been developed to detect and quantify these compounds down to levels of 5 ng/g (5 ppb w/w). This application note describes an easy to perform method using PerkinElmer® Clarus® SQ 8 GC/MS with a TurboMatrix™ 110 headspace sampler to achieve detection limits below 0.5 ng/g.

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APPLICATION NOTE

Mass Spectrometry

Author:

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Shelton, CT USA

Rapid Measurement of Olive Oil Adulteration with Soybean Oil with Minimal Sample Preparation Using DSA/TOF

Introduction

Among edible oils, olive oil shows important and outstanding characteristics due to its differentiated sensorial qualities (taste and flavor) and higher nutritional value. It is an important oil that is high in nutritional value due to its high content of antioxidants (including vitamin E)[1]. Several health benefits, such as its ability to lower LDL cholesterol and its anti-inflammatory activity, associated with its consumption were initially observed among Mediterranean people[2,3]. Olive oil is one of the most adulterated food products of the world due to its relatively low production and higher prices as compared to vegetable and seed oils.

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APPLICATION NOTE

Liquid Chromatography/
Mass Spectrometry

Authors:

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Catherine Stacey

PerkinElmer, Inc.
Waltham, MA

Differentiation of Olive, Vegetable and Seed Oils by LC/MS Analysis of Triacylglycerides

Introduction

Extra virgin olive oil is known for its nutritional value, high antioxidant content and low saturated fat content when compared to other oils.[1] This expensive oil has been adulterated throughout history with less expensive oils such as soybean or hazelnut oil. Detecting adulteration is important for maintaining both the safety and consumer confidence of this product.

This study reports on the measurement of the triacylglyceride composition of a large number of olive oils and other seed and vegetable oils using a simple LC/MS method, together with statistical analysis of the results to find indicators for different oil types.

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APPLICATION NOTE

Mass Spectrometry

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Rapid Measurement of Extra Virgin Olive Oil Adulteration with Olive Pomace Oil with No Sample Preparation Using DSA/TOF

Introduction

Food adulteration or food crime occurs when an ingredient is replaced partially or fully with something different – without the knowledge of the consumer. Most of the time, food adulteration occurs to improve profits by diluting higher value products with lower value materials. The price of extra virgin olive oil (EVOO) is about 3-5 times higher than food grade olive pomace oil and therefore can be found to be blended with lower quality olive pomace oil. EVOO is extracted by mechanical pressing of high quality olive fruit, while olive pomace oil is the residue oil that is extracted by chemical solvents from previously pressed olive mash. Further, olive pomace oil is highly refined to remove chemical impurities. Both EVOO and olive pomace oil have similar triglyceride composition and therefore it is difficult to distinguish them on this basis. In the past, time consuming GC methods with sample preparation have been used to show that the average amount of ethyl ester of oleic acid (EEOA) in extra virgin olive oil and refined lampante olive oil (similar to refined olive pomace oil) is 27.9 mg/kg and 770 mg/kg, respectively[1]. It has also been reported that the ethyl esters of fatty acids in extra virgin olive oil were less than 32 mg/kg, whereas the ethyl esters of fatty acids in olive pomace oil were far higher; in the range of 500-40000 mg/kg[2]. The higher amounts of EEOA in olive pomace oil can be attributed either to heat treatment in processing or to lower quality of olive fruits. Both EVOO and olive pomace oils have similar amounts of free oleic acid (OA)[3]. Therefore, the measurement of ratio of EEOA to oleic acid in EVOO can be used as a way to detect its adulteration with olive pomace oils and other lower quality olive oils, which have a higher content of EEOA in them. In this work using a Direct Sample Analysis™ Time-of-Flight mass spectrometry system (DSA/TOF), we developed a method with no sample preparation to determine adulteration of EVOO with olive pomace oil by measuring the ratio of EEOA to OA.

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APPLICATION NOTE

Differential Scanning Calorimetry

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Detecting the Adulteration of Extra Virgin Olive Oil by Controlled Cooling DSC

Introduction

Food adulteration normally makes the news with cases like melamine in milk[1]. However, high-value products are often subjected to adulteration by lower-value materials and this can be difficult to detect. As a high-priced produce, a pint of extra virgin olive oil (EVOO) is close in cost to that of a half gallon of food-grade olive pomace oil. University of California at Davis has reported that the majority of the extra virgin olive oils sold in California fail the tests for the same (EVOO), using a variety of techniques (ultraviolet and visible spectroscopy [UV/Vis], gas chromatography [GC], liquid chromatography [LC]), and wet methods[2]. However, considering the way EVOO is made, one would expect a relationship to its thermal properties.

Differential scanning calorimetry (DSC) is commonly used to analyze foods in both quality control and research labs[3, 4]. DSC is often used to compare materials on heating, but cooling studies often give more information as materials can respond more thermodynamically under controlled cooling[5].

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FT-IR Spectroscopy

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Advantages of Adulterant Screen for Detection of Olive Oil Adulteration by Attenuated Total Reflectance (ATR) FT-IR

Introduction

Olive oil is an increasingly popular food product worldwide, with consumption in the U.S. alone having increased by about 50% in the last 10 years. Over three million tons annually of olive oil are produced worldwide, with approximately 75% of this being produced in Spain, Italy, and Greece. The U.S. now imports over 300,000 tons of olive oil annually.

Olive oil is considered to be healthy edible oil and is linked to the low incidence of heart disease associated with a Mediterranean diet. It is low in Saturated Fatty Acid (SFA) and Polyunsaturated fats (PUFA) but high in the healthier Monounsaturated fats (MUFA), known to lower cholesterol.

Extra Virgin Olive Oil (EVOO) is a premium product that can command a higher price than "standard" olive oils. This makes it highly susceptible to fraudulent activity. A report by the E.U. Committee on the Environment, Public Health, and Food Safety says olive oil is among the products most prone to food fraud. There were 267 oil adulteration incidents reported to the U.S. Pharmaceutical Food Fraud Database, with the vast majority occurring over the past three years.

Adulteration of EVOO with lower quality olive oils, or other lower cost edible oils, is frequently reported in the media. The most common adulterants include: hazelnut oil, sunflower oil, soybean oil, corn oil, rapeseed oil, and olive pomace oil. Fraudulent activities, such as dilution or even substitution with other lower cost oils containing additional chemicals, that enable the oil to appear to be of higher quality oil and pass routine screening tests are on the rise.

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APPLICATION NOTE

Liquid Chromatography

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Analysis of Phenolic Antioxidants in Edible Oil/Shortening Using the PerkinElmer Altus UPLC System with PDA Detection

Introduction

Phenolic antioxidants are commonly used in food to prevent the oxidation of oils. Oxidized oil and fats cause foul odor and rancidity in food products, which is a major cause for concern to the food industry. Globally, regulations vary, but current maximum allowable levels are as low as 100 µg/g (100 ppm).

This application note presents a UHPLC method for the analysis of the ten most common phenolic antioxidants that may be found in such products. The application was carried out with minor modifications to the AOAC Official Method 983.15 (1). This method applies to the analysis of finished food products. A 2.7-µm SPP (superficially porous particle) C18 column was used, allowing one to achieve very high throughput at a back-pressure considerably lower than that for UHPLC columns.

This method was then applied to a commercial vegetable shortening product, which per label claim, was reported to contain at least one of the antioxidants being analyzed.

Method conditions and performance data, including linearity and repeatability, are presented.

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APPLICATION NOTE

Liquid Chromatography

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Analysis of Common Antioxidants in Edible Oil with the PerkinElmer Flexar FX-15 System Equipped with a PDA Detector

Introduction

Phenolic antioxidants and ascorbyl palmitate (Figure 1, Page 2) are commonly used in food to prevent the oxidation of oils. Oxidized oils cause foul odor and rancidity in food products. This application note will present a UHPLC analysis of edible oils to determine the type and amount of ten different antioxidants.

The method was developed with a 1.9 µm particle size column to achieve very high throughput at a low flow rate, reducing solvent consumption. The throughput of an HPLC method with a 5 µm particle size column will be compared with that of a UHPLC method with a 1.9 µm particle size column. In addition to throughput comparisons, method conditions and performance data, including precision and linearity are presented. The results of the method applied to a spiked oil sample and a sample of vegetable shortening are reported.

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APPLICATION NOTE

Liquid Chromatography

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Rapid UHPLC Determination of Common Antioxidants in Edible Oils

Introduction

Phenolic antioxidants and ascorbyl palmitate (Figure 1 – Page 2) are commonly used in food to prevent the oxidation of oils. Oxidized oils will cause foul odor and rancidity in food products. This application note will present a UHPLC analysis of edible oils to determine the type and amount of ten different antioxidants.

The method was developed with a 1.9 µm column to achieve very high throughput at a low flow rate, reducing solvent consumption. The throughput of an HPLC method with a 5 µm particle-size column will be compared with that of a UHPLC method and 1.9 µm particle-size column. In addition to throughput comparisons, method conditions and performance data, including precision, linearity, and recovery from spiked samples, will be presented.

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APPLICATION NOTE

Atomic Absorption

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Trace Elemental Characterization of Edible Oils with Graphite Furnace Atomic Absorption Spectrophotometer

Introduction

The determination of the inorganic profile of oils is important because of the metabolic role of some elements in the human organism. On the one hand, there is knowledge of the food's nutritional value, which refers to major and minor elements. On the other hand, there is the concern to verify that the food does not contain some minerals in quantities toxic for the health of the consumers, regardless whether this presence of minerals is naturally occurring or is due to contamination during the production processes. Oil characterization is the basis for further nutritional and food technological investigations such as adulteration detection[1]. The most common adulteration is an addition of a cheaper vegetable oil to expensive oil. Authenticity is a very important quality criterion for edible oils and fats, because there is a big difference in prices of different types of oil and fat products. Adulteration detection is possible by determining the ratio of the contents of some chemical constituents and assuming these ratios as constant for particular oil. In regard to adulteration detection, approaches based on atomic spectroscopy can be attractive[2]. The quality of edible oils with regard to freshness, storability and toxicity can be evaluated by the determination of metals. Trace levels of metals like Fe, Cu, Ca, Mg, Co, Ni and Mn are known to increase the rate of oil oxidation. Metals like As, Cd, Cr, Se etc. are known for their toxicities. The development of rapid and accurate analytical methods for trace elements determination in edible oil has been a challenge in quality control and food analysis. However, sample pretreatment procedures are required in order to eliminate the organic matrix. These include wet, dry or microwave digestion, dilution with organic solvent and extraction methods[3]. The content of metals and their species (chemical forms) in edible seed oils depends on several factors. The metals can be incorporated into the oil from the soil or be introduced during the production process. Hydrogenation of edible seed oils and fats has been

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APPLICATION NOTE

Atomic Absorption

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Toxic Trace Metals in Edible Oils by Graphite Furnace Atomic Absorption Spectrophotometry

Introduction

Graphite furnace atomic absorption spectrophotometry (GFAAS) has been widely applied to the determination of trace elements in food due to its selectivity, simplicity, high sensitivity, and its capability for accurate determinations in a wide variety of matrices. Edible oils are generally low in trace element concentrations, however, metals such as arsenic (As), lead (Pb), cadmium (Cd), chromium (Cr), and selenium (Se) can be found and are known for their toxicities which affect the health of consumers. The determination of toxic elements from naturally occurring or production-contamination sources in oils can be determined by using GFAAS or inductively coupled plasma mass spectrometry (ICP-MS). When only a few elements are being analyzed, GFAAS is the preferred choice. It is easy to learn, faster in setting up, and simpler to use than ICP-MS. GFAAS is also lower in initial capital investment and has a lower operating and maintenance cost. Sample pretreatment procedures for edible oils are normally required prior to instrumental analysis in order to eliminate the organic matrix. Wet, dry or microwave digestion, dilution with organic solvent, and extraction methods can be time consuming and require more operator training than a direct analysis method.

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APPLICATION NOTE

UV/Vis Spectroscopy

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Measurement of Quality of Crude Palm Oils used in Margarine Production by UV/Visible Spectroscopy

Crude Palm Oil (CPO) is a raw material used in the production of margarine and other vegetable oil based food products. CPO is traded and there are quality specifications based on free fatty acids (FFAs) as well as moisture and impurities.[1,2,3]

Margarine manufacturers also want to assess the CPO's 'fitness for refining' which is measured by the Deterioration of Bleachability Index (DOBI). A DOBI index of less than 1.8 indicates a poor quality oil; a DOBI index > 3 indicates a high quality oil

The DOBI index is defined as the absorbance ratio $A_{446\ nm}$ / $A_{269\ nm}$ of around 0.04 g oil dissolved in 25 mL of hexane or 2,2,4Trimethylpentane (iso-octane).

Rather than simply measuring the DOBI at fixed wavelengths, there are advantages in measuring the spectrum between 220 and 500 nm as it means that it is also possible to calculate the carotene content by measuring the CPOs primary and secondary oxidation products. In addition, any adulterants added to enhance the DOBI can be detected by examining the spectrum in more detail.

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APPLICATION BRIEF

ICP - Mass Spectrometry

Authors:

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The Elemental Analysis of Meat and Seafood with the NexION 300/350 ICP-MS

Introduction

The elemental content of food products is very important, both in relation to nutritional and toxic elements. Nutritional elements can either be native to the food substance or can be added to enhance the health benefits. Toxic elements can enter food either through the environment or processing during production. Hopefully, toxic elements will be present at extremely low levels, while nutritional elements will be present at optimal levels: if too high, they may be toxic; if too low, the food will not provide the necessary nutrition. Therefore, the elemental analysis of food requires the ability to measure both trace and high levels.

The elemental capabilities and dynamic range of inductively coupled plasma mass spectrometry (ICP-MS) make it ideally suited for the analysis of food materials. The ultratrace detection limits of ICP-MS permit the determination of low-level contaminants, such as Pb, As, Se, and Hg, while the macro-level nutritional elements, such as Ca, Mg, K, and Na, can be quantified using the extended dynamic range capability of ICP-MS which provides the ability to measure concentrations over nine orders of magnitude. However, there are still a number of challenges to overcome, including complex sample matrices, high levels of dissolved solids, and interferences. With the proper ICP-MS instrumental conditions and design, all of these issues can be overcome, allowing for the successful analysis of food samples, as described elsewhere[1]. This work will focus on the analysis of meat and seafood; foods such as these are high in protein content which is important for body growth and repair.

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APPLICATION NOTE

Gas Chromatography/
Mass Spectrometry

Authors

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The Preparation and Analysis of Polycyclic Aromatic Hydrocarbons in Meat by GC/MS

Introduction

Polycyclic aromatic hydrocarbons (PAHs) are hydrocarbon molecules containing two or more aromatic rings. Some PAHs, such as benzo[a]pyrene, are classified as carcinogens; PAHs are commonly found in the environment as a result of partially burned organic materials, such as petroleum, plastics, rubber, lubricants and wood. In addition to environmental concerns, there are concerns about PAHs in food, especially in grilled meats.

The European Union (EU) introduced legislation in early 2005 in response to food-contamination data collected by the Scientific Committee on Food (SCF). The SCF identified 15 PAHs that possess carcinogenic properties. Directive 2005/69/EC of the European Parliament and of the Council of 16 November 2005 identified an additional PAH as probably carcinogenic.[1] The joint set of PAHs was recognized as the 15 + 1 EU priority PAHs. It is necessary to accurately determine the level of 15 + 1 EU priority PAHs in food to respond to European legislation and ensure food safety.

This application note will present a method developed to measure 15 + 1 EU priority PAHs at low levels using gas chromatography mass spectrometry (GC/MS). It will also describe a reliable procedure for extraction and purification of PAHs from meat samples. The sample preparation will focus on benzo[a]pyrene. In addition to method optimization and calibration, a variety of meat samples are analyzed and the amount of PAHs determined.

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APPLICATION NOTE

Differential Scanning Calorimetry

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Glass Transition Measurement of Undried Fish Gelatin with Fast Scan DSC Technique

Background

Glass transition (Tg) measurement of undried bio-polymers is challenging using the conventional DSC technique. The glass transition (Tg) is frequently masked by the moisture vaporization event. This is particularly troublesome in biomaterial where transitions near 100 ˚C and 0 ˚C are common. HyperDSC® separates two overlapping events with different kinetic properties, therefore Fast Scan DSC techniques (HyperDSC) allow measurements beyond the range of conventional DSC. Double Furnace DSC has superior performance, such as extremely short equilibration time, high sensitivity and high resolution. These support the HyperDSC technique. HyperDSC expands the DSC capabilities beyond the limitations of the conventional slow scan DSC technique by allowing us to separate the overlapping events with different kinetics and to amplify weak thermal events. In this paper, we demonstrate the HyperDSC capability to measure the Tg event of the undried fish gelatin samples with a very short test cycle time.

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APPLICATION NOTE

Atomic Absorption

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Analysis of Fish and Seafoods with AAnalyst 800 Atomic Absorption Spectrophotometer for Trace Metal Contamination, in Accordance with AOAC Methods 999.10 and 999.11

Introduction

Increased knowledge about the nutrient content of biological organisms is essential for a thorough understanding of ecological stoichiometry and nutrient transport in and among ecosystems. As a result of water pollution in coastal area, many problems in food safety like heavy metal accumulation have been recognized in farmed fish, which is one of the important fishery food resources. The heavy metals accumulated in fish not only have a bad influence on fish but they also affect the health of human beings through the food chain. It is pointed out that remarkable heavy metals were contained in fish meals that are used as major raw materials for aquaculture feeds. The Itai-itai disease of the Toyama Jintsu River area in Japan was the documented case of mass cadmium poisoning. Itai-itai disease is known as one of the Four Big Pollution Diseases of Japan.

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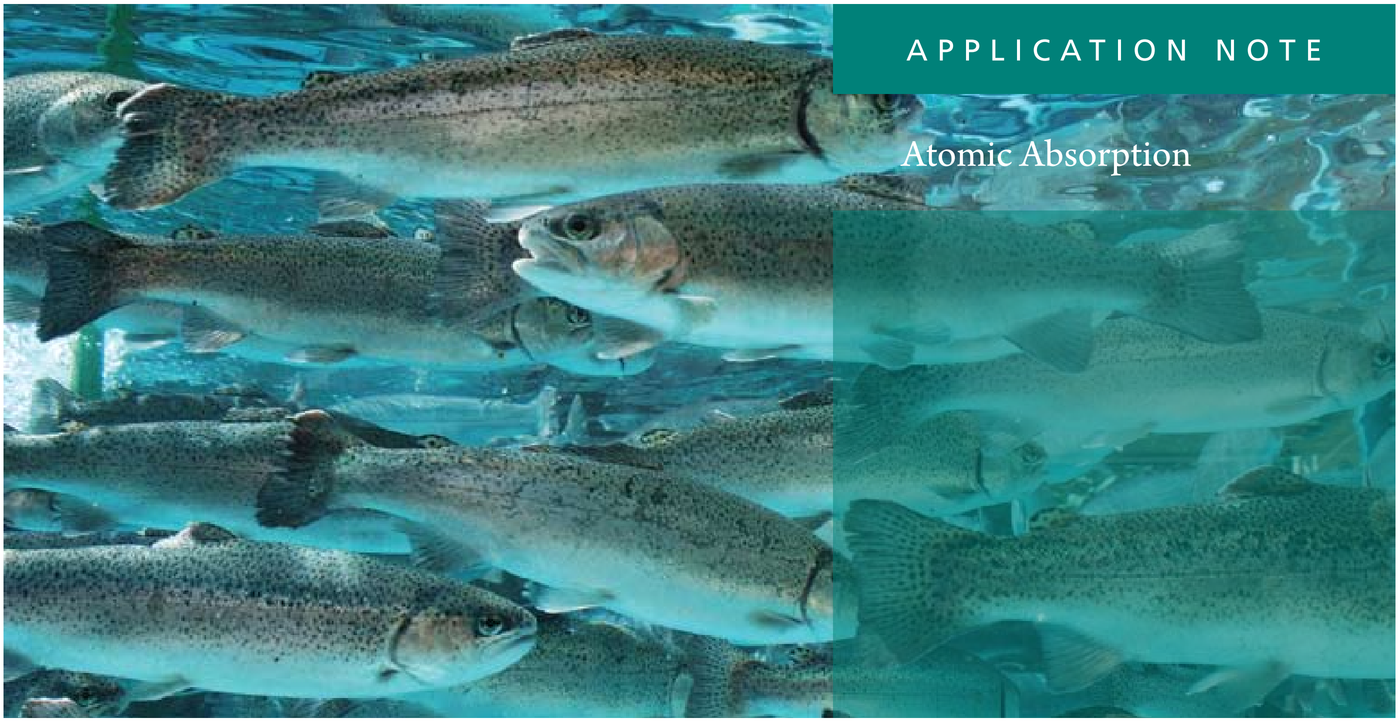

APPLICATION NOTE

Atomic Absorption

The Determination of Total Mercury in Fish and Agricultural Plant Materials Using Thermal Decomposition and Amalgamation Coupled with Atomic Absorption

Summary

Mercury has long been recognized as a serious global pollutant that has a significant impact upon our ecosystem. Unlike most other pollutants, it is highly mobile, non-biodegradable, and bio-accumulative and as a result has to be closely monitored to ensure its harmful effects on local populations are minimized. Approximately 50 tons of mercury particulates are emitted into the atmosphere every year by a variety of different man-made and natural sources including coal-fired power plants, solid waste incineration plants, volcanoes and forest fires. When the mercury falls back to earth it is deposited on the land and gets into the soil, river sediments and water ecosystems, where it is converted into the highly toxic organo mercury compound, methyl mercury (CH_3Hg^+). This toxicant enters both the plant and aquatic system food chain, and eventually ends up in the crops, vegetables and seafood we consume.

This application note will focus on a rapid test method for determining mercury directly in food materials and agricultural crops using the principles of thermal decomposition, amalgamation and detection by atomic absorption described in EPA Method 7473 and ASTM Method 6722-01. Because there is no sample dissolution required, this novel approach can determine the total mercury content in these types of samples in less than five minutes, which is significantly faster than the traditional wet chemical reduction method for quantifying mercury.

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CASE STUDY

Food

FT-IR Analysis Critical to Fast Characterisation of Unknown Contaminants in Food

Advances in analytical chemistry mean there are a now variety of techniques that can be used in the identification of unknown contaminants. The challenge for testing laboratories is to balance the use of established methods, such as IR, with more specific techniques such as LC/MS. The goal being efficient and fast sample testing. This enables clients to benefit from rapid sample throughput (from processing to analysis and final reporting), and have the ability to respond swiftly to any urgent requests for investigation.

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APPLICATION NOTE

Differential Scanning Calorimetry

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Practical Food Applications of Differential Scanning Calorimetry (DSC)

Abstract

This note describes a number of important food applications utilising the PerkinElmer DSC demonstrating the versatility of the technique as a tool in the food industry.

Introduction

Food is often a complex system including various compositions and structures. The characterization of food can therefore be challenging. Many analytical methods have been used to study food, including differential scanning calorimetry (DSC).[1] DSC is a thermal analysis technique to measure the temperature and heat flows associated with phase transitions in materials, as a function of time and temperature. Such measurements can provide both quantitative and qualitative information concerning physical and chemical changes that involve endothermic (energy consuming) and exothermic (energy producing) processes, or changes in heat capacity.

DSC is particularly suitable for analysis of food systems because they are often subject to heating or cooling during processing. The calorimetric information from DSC can be directly used to understand the thermal transitions that the food system may undergo during processing or storage. DSC is easy to operate and in most cases no special sample preparation is required. With a wide range of DSC sample pans available, both liquid and solid food samples can be studied. Typical food samples and the type of information that can be obtained by DSC are listed in Table 1. These tests can be used for both QC and R&D purposes. DSC applications are used from troubleshooting up to new product developments.

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FIELD APPLICATION REPORT

ICP-Mass Spectrometry

The Determination of Iodine in Food with the ELAN DRC-e ICP-MS

Authors

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Acknowledgements

The method development has been done by Fabien Bolle and Khalid Boutakhrit of the Belgian Scientific Institute of Public Health.

ntroduction

odine is essential for the production of thyroid hormones. These hormones stimulate metabolism in the body, as well as mental growth and evelopment. The recommended daily iodine intake is 0.1-0.2 mg, with he most common sources of iodine being fish, seafood, milk and food upplements. The determination of iodine in food has always been diffiult due to the low concentrations (mg/kg), difficult sample preparation nd the volatility of iodine.

This work demonstrates the ability of the ELAN® ICP-MS to measure odine in food samples.

xperimental

ample Preparation

amples consisted of three certified reference materials: skimmed milk owder, cod, and mussel. Prior to weighing, the samples were mixed or lightly crushed; 0.25-0.5 g of sample was then added to PFA tubes, ollowed by 4.5 mL H_20 (Milli-Q) and 1 mL TMAH (25%).

After capping, the tubes were placed in a drying oven at 90 °C for hours. After cooling, Milli-Q® water was added to a final volume of 0 mL. These solutions were then centrifuged at 3000 rpm for 15 minutes. f any visible particulates remained after centrifuging, the samples were hen filtered. The resulting solutions can then be analyzed directly or ith an extra dilution if high matrix concentrations are present.

Instrumental Conditions

The instrument used for this analysis was an ELAN DRC™ II ICP-MS. Instrumental operating parameters are shown in Table 1. All measurements were done in standard mode.

Calibration standards ranging from 5 to 20 µg/L were used, prepared in 0.5% (v/v) TMAH. After each standard and sample were analyzed, a 45 second rinse with of 0.5% TMAH was performed.

Table 1. **Instrumental Conditions.**

Spray chamber	Cyclonic
Nebulizer	Meinhard®
Sample Uptake Rate	1 mL/min
RF Power	1100 W
Plasma Gas Flow (L/min)	15
Nebulizer Gas Flow (L/min)	0.93
Auxiliary Gas Flow (L/min)	1.2
Dwell Time (ms)	50
Sweeps per reading	25
Replicates	3
Delay Time (s)	50
Wash Time (s)	45

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APPLICATION NOTE

GC-Mass Spectrometry
and Headspace Sampling

Author

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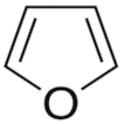
Determination of Furan in Food by Gas Chromatography-Mass Spectrometry and Headspace Sampling

Introduction

Furan is naturally occurring at low levels in many foods and drinks.[1] Furan consumption is of concern because it has been classified by the International Agency for Research on Cancer (IARC) as possibly carcinogenic to humans, based on studies with laboratory animals. The U.S. FDA has recently published a report on the occurrence of furan in a large number of thermally processed foods, especially canned and jarred foods, including baby foods and infant formulas. The primary source of furan in food is considered to be thermal degradation of carbohydrates, such as glucose, lactose and fructose.

Of all the foods tested in various papers, coffee contained the largest amount of furans.[1] Furan is a colorless, volatile and lipophilic organic compound. It has a molecular weight of 68 and a low boiling point (31 °C). Due to its high volatility, furan levels in foods are easily determined, with high accuracy, by headspace methods.

This application note will demonstrate a rapid method for the identification and quantification of furan in food samples, using gas chromatography with headspace sampling and mass spectrometry. In addition to method optimization and standard analysis, we will analyze a number of food samples for furan. We chose to test coffee containing drinks, sauces, and canned foods, as previous studies demonstrated high levels of furan in these foods. The samples were randomly collected from the local market.



Synonyms: furfuran, oxole, tetrole, divinylene oxide, oxacyclopentadiene
Formula: C_4H_4O
MW: 68.07
MP: -85.6 ºC
BP: 31 ºC

Figure 1. Structure and physical properties of furan.

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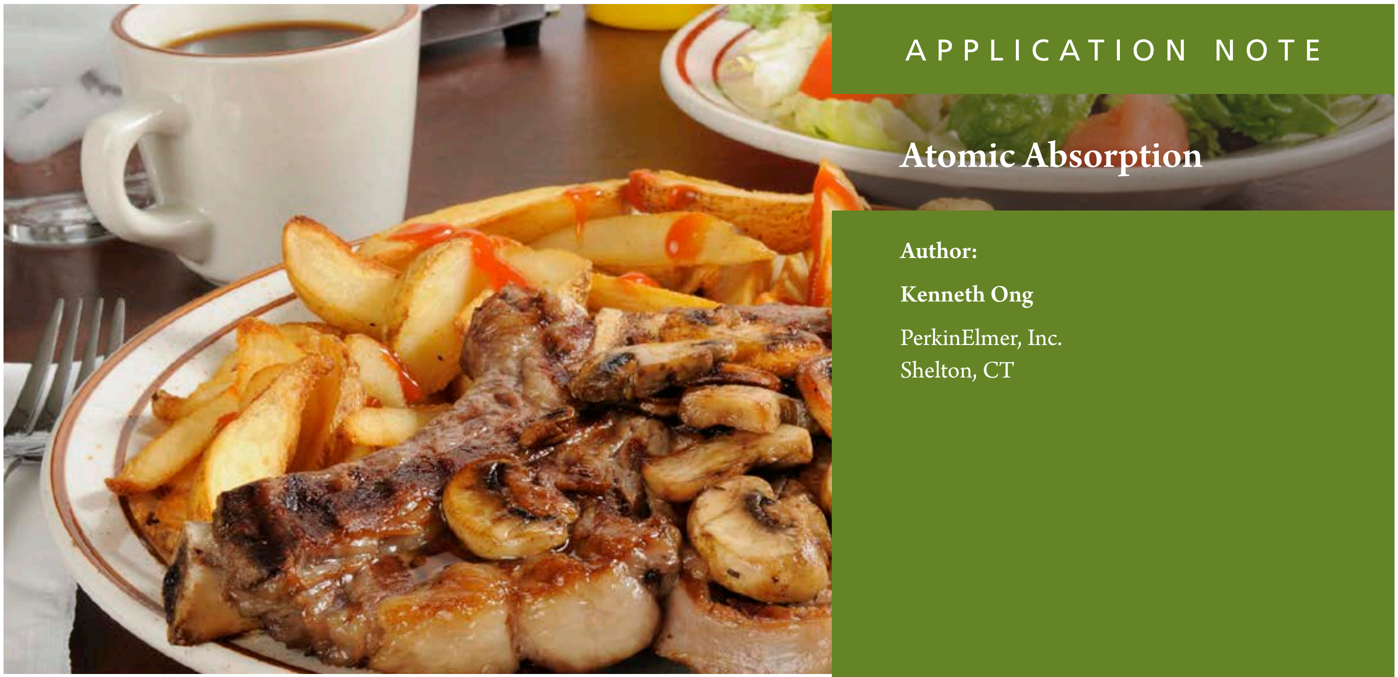

Atomic Absorption

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Determination of Lead and Cadmium in Foods by Graphite Furnace Atomic Absorption Spectroscopy

Background

Humans can be exposed to heavy metals through a variety of means, including consumption of contaminated food. Although heavy metals are usually present in foods at very low levels, long-term exposure can have negative health impacts. Two of the more important toxic elements that must be monitored are cadmium (Cd) and lead (Pb), which can enter food either through environmental processes or through contamination in processing and/or packaging. As a result, it is very important to accurately measure low levels of Cd and Pb in a variety of food matrices.

A major challenge in the analysis of food samples is the extremely low analyte levels and the very high matrix levels. For many years, graphite furnace atomic absorption spectroscopy (GFAAS) has been a reliable technique and the preferred method for this analysis, especially for the determination of Cd and Pb.

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APPLICATION NOTE

Gas Chromatography/
Mass Spectrometry

Author

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Analysis of Butylated Hydroxytoluene in Food with Headspace Trap-GC/MS

Introduction

Butylated hydroxytoluene (BHT, 2,6-di-tert-butyl-4-methylphenol) is a common food additive. BHT is found in many types of food including butter, meats, cereals, chewing gum, baked goods, snack foods, dehydrated potatoes and beverages. It is used to preserve food odor, color and flavor. BHT is oxidized preferentially in fats or oils, protecting the foods from spoilage.

Concern exists that long-term human consumption of BHT may have potential health risks. It has undergone the additive application and review process required by the U.S. Food and Drug Administration (FDA); the committee concluded that no evidence in the available information on BHT demonstrates a hazard to the public when it is used at levels that are now current and in the manner now practiced. However, uncertainties exist requiring that additional studies should be conducted.[1] The chemical properties which make BHT an excellent preservative may also be implicated in health effects. The oxidative characteristics and metabolites of BHT may contribute to carcinogenicity. Some people may have difficulty metabolizing BHT, resulting in health and behavioral changes.

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APPLICATION NOTE

Atomic Absorption

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The Determination of Toxic, Trace, and Essential Elements in Food Matrices using THGA Coupled with Longitudinal Zeeman Background Correction

Introduction

Ingestion of trace elements from food can be linked to nutrition, disease, and physiological development. Whether they are needed for proper nutritional value or contain toxic elements, the presence of major and minor elements in food needs to be verified to help determine health effects for the consumer. Contamination of food products may result from metals present during cultivation and/or processing. Acute or chronic exposure to heavy metals can lead to damaged nervous system function and have detrimental effects on vital organs. Food safety laboratories performing these analyses are often high-throughput facilities and require a detection tool that is efficient and cost effective.

Unlike flame atomic absorption spectrophotometry (FAAS) where the ground state atoms quickly diffuse into surrounding air, graphite furnace atomic absorption spectrophotometry (GFAAS), being a total consumption technique, offers the ability to dry and atomize the entire pipetted sample in a more controlled environment within the graphite tube. This significantly increases sensitivity and provides superior detection limits with microliter (µL) sample volumes. Only ICP-MS can provide the same level of detection as GFAAS, however GFAAS is more cost efficient, simpler to operate and has fewer laboratory facility requirements.

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APPLICATION NOTE

ICP – Mass Spectrometry

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PerkinElmer, Inc.
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The Determination of Toxic, Essential, and Nutritional Elements in Food Matrices Using the NexION 300/350 ICP-MS

Introduction

The elemental and dynamic range of inductively coupled plasma-mass spectrometry (ICP-MS) makes it ideally suited for the analysis of food materials. The ultratrace detection limits of ICP-MS permit the determination of low-level contaminants such as Pb, As, Se, and Hg, while the macro-level nutritional elements such as Ca, Mg, K, and Na can be quantified using the extended dynamic range capability of ICP-MS which provides 9-orders of magnitude. However, there are still a number of challenges to overcome, which makes the routine analysis of foods difficult unless the sample dissolution procedure is well thought out and instrumental conditions are optimized for complex sample matrices.

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APPLICATION NOTE

Liquid Chromatography

Authors

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Safeguarding Food from Pesticides by UHPLC After Extraction with the QuEChERS Method

Introduction

The detection of pesticides in food matrices, such as fruit and vegetables, requires an ever increasing use of new technologies. Chromatographic techniques, both gas (GC) and liquid (LC), offer a suitable means of addressing the analytical requirements. They enable screening to be carried out rapidly both in gaseous and liquid phases – on samples prepared using the "QuEChERS" (Quick, Easy, Cheap, Effective, Rugged, Safe) system, a fast and effective multi-residue extraction method.

Employing UHPLC (Ultra High Performance Liquid Chromatography) with a PDA (Photo Diode Array) detector offers considerable advantages over standard LC analysis. Indeed, it provides a very fast means of monitoring phytodrugs which are permitted in concentrations of up to 10 µg/Kg. The use of a PDA detector, together with the development of a library of spectra, enables each substance to be analyzed and identified correctly.

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Determination of Nickel in Fats and Oils

Scope

Triglyceride-based vegetable fats and oils can be transformed through partial or complete hydrogenation to fats and oils of greater molecular weight. The hydrogenation process involves sparging the oil at high temperature and pressure with hydrogen in the presence of a catalyst, typically a powdered nickel compound. Atomic Absorption Spectrometry is commonly used to estimate the amount of nickel left in the vegetable oils.

Typical Analytical Procedure

Materials and Methods

The following reagents and equipment are used for the measurement:

- Atomic absorption spectrometer
- Nickel metal
- Conc. nitric acid
- Conc. hydrochloric acid
- Double distilled water

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APPLICATION NOTE

Differential Scanning Calorimetry

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Investigating the Destabilization of Solid Emulsions Using Differential Scanning Calorimetry (DSC)

Introduction

Emulsions are systems that consist of two or more liquid phases that are partially or completely immiscible with one liquid being dispersed in the other in the form of droplets. Emulsions constitute an important product class in various industries including the food, chemical and pharmaceutical industries. Emulsions are inherently unstable since the two immiscible liquids have a tendency to phase separate over time in order to minimize the thermodynamically unfavorable interaction between the two or more molecular species. As such, the kinetic stability of emulsions that is their ability to resist phase separation due to destabilization processes such as flocculation, coalescence, or Ostwald ripening is of key importance to manufacturers.

There are a number of methods available to determine the stability of emulsions, with each method having certain advantages and disadvantages. Light scattering is a very common method to determine the stability of emulsions, because it can be used to assess changes in the droplet size distribution of the emulsion. Unfortunately, light scattering does not yield information about differences in composition of individual particles in the emulsions, and therefore, it cannot give specific information on emulsion

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APPLICATION BRIEF

ICP - Mass Spectrometry

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The Elemental Analysis of Grains with the NexION 300/350 ICP-MS

Introduction

Trace elemental analysis of grains can provide associations between air pollution sources and soil variables. The elements themselves are distributed unevenly throughout the cereal grain, with the germ and the outer layers having the highest concentrations. Therefore, the elemental analysis requires the ability to measure both trace and high levels.

The elemental capabilities and dynamic range of inductively coupled plasma mass spectrometry (ICP-MS) make it ideally suited for the analysis of food materials. The ultratrace detection limits of ICP-MS permit the determination of low-level contaminants, such as Pb, As, Se, and Hg, while the macro-level nutritional elements, such as Ca, Mg, K, and Na, can be quantified using the extended dynamic range capability of ICP-MS which provides the ability to measure concentrations over nine orders of magnitude. However, there are still a number of challenges to overcome, including complex sample matrices, high levels of dissolved solids, and interferences. With the proper ICP-MS instrumental conditions and design, all of these issues can be overcome, allowing for the successful analysis of food samples, as described elsewhere[1]. This work will focus on the analysis of grains.

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APPLICATION NOTE

ICP - Mass Spectrometry

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Characterization of Silver Nanoparticles in Dietary Supplements by Single Particle ICP-Mass Spectrometry

Introduction

A nanopartcile is defined as a small object, between 1 and 100 nanometers in size, that behaves as a whole unit with respect to its transport and properties. Because of their small size and large surface area, nanoparticles can exhibit different chemical and physical properties from the bulk material. Nanoparticles have found their way into a large number of consumer products. As of 2013, it is estimated that there are over 1300 different consumer products which feature nanoparticles. Silver nanoparticles (AgNPs) are the most frequently found element in all varieties of consumer products (>23%)[1]. Manufacturers of consumer products use AgNPs due primarily to their known antimicrobial properties. Because of their very small size, AgNPs have high surface areas yielding high reaction rates, increasing the efficacy of silver as an antimicrobial.

The use of colloidal and nano silver is directly marketed to the public in such forms as odorless clothing, mildew-resistant shower curtains, food containers and food cutting boards and are even being promoted for direct human consumption as dietary supplements to fortify one's immune system.

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APPLICATION NOTE

Liquid Chromatography

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The Qualitative and Quantitative Analysis of Water-Soluble B Vitamins by HPLC-PDA in Various Multivitamin Tablets

Introduction

Vitamins are essential nutritional elements in a human diet. Though we are able to synthesize certain vitamins, such as Vitamin D when exposed to sunlight, we mainly depend on our diet to supply our vitamins.

Since Ancient Egypt, humans have recognized that eating certain foods guarded us from certain diseases. But only in 1912 did Polish biochemist Kazimierz Funk establish the term *"Vitamine"*, derived from the compound words *Vital* and *Amine,* together meaning *amine of life.* Today, the term *"Vitamin"* is described as an organic compound serving as a vital nutrient that an organism requires in limited amounts.[1]

The food industry routinely fortifies many foods with vitamins to enhance their nutritional value and to help with deficiencies in dietary requirements. However, to meet legal requirements, the manufacturers must label their products in accordance to the specific regulations pertaining to the country in which the products are sold and consumed.

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APPLICATION NOTE

Liquid Chromatography

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Fast Analysis of Fat-Soluble Vitamins Using Flexar FX-10 and Chromera CDS

Introduction

Vitamins are essential nutrients and used in numerous systems throughout the human body. Vitamins are divided into two groups: water-soluble (B-complex and C) and fat-soluble (A, D, E and K). Water-soluble vitamins have limited retention in the body and need regular replacement. Fat-soluble vitamins are stored in the liver and fatty tissues, allowing accumulation and consumption over time.

The determination of vitamin content in foods is important in quality, labeling and marketing. Europe and the United States require food products to be clearly labeled with nutritional information; the vitamin content is required on the label.

Supplements and functional foods are intended to provide health benefits to consumers–these products often contain vitamins. Vitamins, D and E are commonly marketed in supplements because of their role in calcium absorption of bones and antioxidant properties, respectively.

A reliable and fast determination of fat-soluble vitamins in food and supplement products is important in complying with regulations and controlling the quality and safety of products. This application note will present a fast and robust method for the determination of fat-soluble vitamins using UHPLC. An optimized separation of vitamins A acetate, K2, D2, D3, E succinate, E acetate and K1 using the PerkinElmer® Flexar™ UHPLC system is presented. Calibration of the UHPLC system and analysis of the vitamins in a commercial supplement will be presented.

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APPLICATION NOTE

Liquid Chromatography

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Analysis of Capsaicin and Dihydrocapsaicin in Chili Peppers Using the PerkinElmer Altus HPLC System with PDA Detection

Introduction

Capsaicinoids are the compounds that produce the pungency, aroma and flavor of chili peppers. The two most abundant capsaicinoids in chili peppers are capsaicin (8-methyl-N-vanillyl-trans-6-nonenamide) and dihydrocapsaicin. Combined, these two make up close to 90% of the most pungent varieties of capsaicinoids, with capsaisin making up about 71%.[1] The capsaicin content of peppers is one of the parameters that determine their commercial quality. The amount of capsaicin can vary, depending on the light intensity and temperature at which the plant is grown, the age of the fruit, and the position of the fruit in the plant.[2]

Besides their widespread uses in foods, capsaicinoids are increasingly being used as the active component in pharmaceuticals and have been used as an analgesic against arthritis pain and inflammation.[3] They have also been reported to be active against neurogenic inflammation (as in pepper sprays) and have shown protective effects against high cholesterol levels and obesity.[4, 5]

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APPLICATION NOTE

Atomic Absorption

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The Determination of Minerals and Metals in Multi-Mineral/Multi-Vitamin Tablets by Flame Atomic Absorption Spectroscopy

Introduction

There are many mineral dietary supplements available in today's marketplace to ensure that mineral deficiencies do not occur in one's diet. The mineral content of these products must be verified for quality control (QC) purposes. Furthermore, the Nutritional Labeling and Education Act of 1990 mandates accurate labeling of all food supplements sold in the U.S. which means accurate testing of the products is mandatory. In many labs, this task is accomplished by the technique of flame atomic absorption spectroscopy (FAAS). FAAS has the advantages of lower initial cost, low cost per analysis, and requires less operator training than many other trace elemental techniques. The objective of this work is to demonstrate the applicability of FAAS using the PerkinElmer® PinAAcle™ 900T to accomplish this task. Seven elements are determined in two commercially available multi-mineral tablets, a NIST® Standard Reference Material, and a commercial reference material which simulates a mixed food diet.

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APPLICATION NOTE

Liquid Chromatography/
Mass Spectrometry

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Targeted Screening of 130 Pesticides in QuEChERS Extracts of Lettuce Leaves Using UHPLC-TOF and High Throughput Screening Software

Introduction:

The Food Quality protection Act (FQPA) in the United States (US) and the European Union (EU) directive 91/414/EEC require that if pesticides are present in food they are below agreed levels due to the health risk posed by pesticides [1, 2]. With the advent of large scale agricultural production, hundreds of pesticides have been synthesized in the last century and used widely to protect crops. Newer pesticides continue to be synthesized for crop usage which makes it important to analyze both targeted (or expected analytes) and non-targeted pesticides in food and in the environment. Unlike a triple quadrupole instrument that only measures targeted analytes (defined by selected multiple reaction monitoring of analyte ions or MRMs), the time-of-flight (TOF) mass spectrometer can measure both targeted and non-targeted analytes[3]. TOF mass spectrometers collect full spectrum information and hence the data can be re-examined for the presence of these "non-targeted" analytes. We present a study of pesticide analysis in a lettuce leaves extract that was obtained by the QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) method of food extraction. The lettuce extract was spiked with varying concentrations of a mix of 130 pesticides and analyzed by Ultra-High Pressure Liquid Chromatography-Mass Spectrometry (UHPLC-MS) with a PerkinElmer AxION® 2 TOF MS as the detector. We could detect the majority of the pesticides well within the EU limit of detection (LOD) requirement range of 10 ppb. The data was further analyzed using AxION Solo™ high throughput software. The presence of each of the analytes when detected above the 10 ppb threshold was given a specific color code which helped to rapidly screen for presence/absence of all 130 analytes in each sample. A combination of short run times and powerful screening software helped simplify analysis and also reduce the time of analysis.

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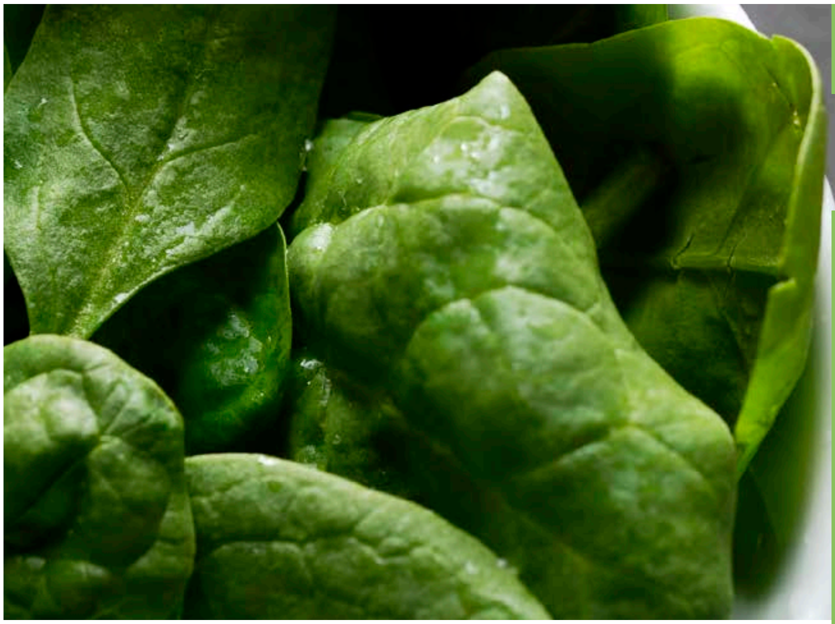

APPLICATION BRIEF

ICP - Mass Spectrometry

Authors:

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PerkinElmer, Inc.
Shelton, CT

The Elemental Analysis of Spinach with the NexION 300/350 ICP-MS

Introduction

Trace metals in food can be highly toxic or nutritionally beneficial, depending on the type of metal present and its concentration. Naturally present in many foods, some elements are added to boost the nutritional value and enhance consumers' well-being, while others may be toxic at higher concentrations. Therefore, the elemental analysis of food requires the ability to measure both trace and high levels.

The elemental capabilities and dynamic range of inductively coupled plasma mass spectrometry (ICP-MS) make it ideally suited for the analysis of food materials. The ultratrace detection limits of ICP-MS permit the determination of low-level contaminants, such as Pb, As, Se, and Hg, while the macro-level nutritional elements, such as Ca, Mg, K, and Na, can be quantified using the extended dynamic range capability of ICP-MS which provides the ability to measure concentrations over nine orders of magnitude. However, there are still a number of challenges to overcome, including complex sample matrices, high levels of dissolved solids, and interferences. With the proper ICP-MS instrumental conditions and design, all of these issues can be overcome, allowing for the successful analysis of food samples, as described elsewhere[1]. This work will focus on the analysis of spinach, an important food staple, especially in the developing world.

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APPLICATION NOTE

Mass Spectrometry

Author:

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Rapid Screening and Quantitation of Postharvest Fungicides on Citrus Fruits Using AxION DSA/TOF and Flexar SQ MS

Introduction

Thiabendazole and Imazalil are the major post-harvest fungicides used on surface of citrus fruits such as oranges, grapefruits and lemons to prevent mold formation and control postharvest decay by various fungal pathogens in citrus fruits[1]. Figure 1 shows the structure of two fungicides. These substances have toxicity in higher doses, with effects such as liver and intestinal disorders and carcinogenic effects. The MRL (maximum residue limits) of imazalil and thiabendazole for conventional citrus fruits are 10 ppm and 5 ppm in the U.S. and Europe, respectively[2,3]. The use of these fungicides on organic citrus fruits is prohibited. The maximum residue limits for these compounds on conventional citrus fruits and possible toxic effects require that these compounds should be monitored before consumption of fruit to ensure food safety. Previously, these fungicides on citrus fruits have been measured by different LC/MS techniques with varying sample preparation techniques such as liquid-liquid extraction[4,5] and QUECHERS[6,7].

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APPLICATION NOTE

Gas Chromatography/
Mass Spectrometry

Authors

Stephen Davidowski

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The Extraction and Quantification of Limonene from Citrus Rinds Using GC/MS

Introduction

D-Limonene, shown in Figure 1, is a common naturally occurring compound with a citrus scent. It is often used as an additive in food products and fragrances, and is classified by the U.S. Food and Drug Administration (FDA) as Generally Recognized as Safe (GRAS)[1]. It has also been approved by the U.S. Environmental Protection Agency (EPA) for usage as a natural pesticide and insect repellent[1]. Limonene has also been studied for its anti-carcinogenic properties[2]. Orange oil, which contains a considerable amount of limonene, has numerous applications including a combustant in engines[3], a powerful degreaser in cleaning applications, and a natural pesticide[4]. These uses may require a known concentration of limonene with a limited amount of impurities. This exemplifies the need for a reliable method of extraction of limonene from its natural source, citrus rinds, followed by a quantitative analysis of the extract for limonene and possible impurities.

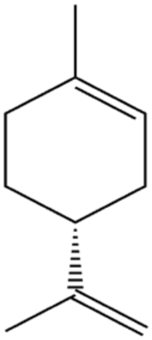

Figure 1. Molecular structure of limonene.

A method for the extraction and quantification of limonene from citrus fruit peels is discussed in this applications note. Beyond demonstrating the use of GC/MS in the analysis of citrus fruit for limonene content, this application demonstrates a simple, inexpensive technique to introduce students to method development, calibration and quantification using a chromatographic technique. The analysis of citrus fruit for limonene may be an ideal laboratory assignment at the undergraduate level. The techniques used are safe, simple and easy.

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APPLICATION NOTE

FT-IR Spectroscopy

Authors

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Rapid Authentication of Larch Fiber Dietary Supplement Ingredient by FT-IR Using Diamond Single Bounce uATR Sampling Device

Introduction

Larch fiber is a complex botanical material rich in arabinogalactin containing polymers that has shown promise as an immune-stimulating and pre-biotic dietary supplement. However, due to the highly complex nature of the material, it does not lend itself to easy identification using microscopy or thin layer chromatography; two common methods used to authenticate dietary supplements. In order to comply with the new Dietary Supplement current Good Manufacturing Practices (cGMP's), all dietary ingredients must be identified using a valid scientific method. The Spectrum™ 400 equipped with a single-bounce diamond uATR sampling accessory allows for rapid identification of larch fibers of various grades. Even though there is some variance among samples, the fingerprint region has a consistent profile that is unique and easily recognized. Differentiation between authentic larch fiber and common potential economic adulterants (cellulose and carageenan) is achieved using this method. FT-IR is a very well established methodology for identification of chemical compounds and can be utilized to comply with the 21 CFR Part 111 cGMP's for Dietary Supplements. Using the Enhanced Security software meets the requirements of 21 CFR Part 11 which is also called for in the Dietary Supplement cGMP's.

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APPLICATION NOTE

Atomic Absorption

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The Determination of Minerals and Metals in Multi-Mineral/Multi-Vitamin Tablets by Flame Atomic Absorption Spectroscopy

Introduction

There are many mineral dietary supplements available in today's marketplace to ensure that mineral deficiencies do not occur in one's diet. The mineral content of these products must be verified for quality control (QC) purposes. Furthermore, the Nutritional Labeling and Education Act of 1990 mandates accurate labeling of all food supplements sold in the U.S. which means accurate testing of the products is mandatory. In many labs, this task is accomplished by the technique of flame atomic absorption spectroscopy (FAAS). FAAS has the advantages of lower initial cost, low cost per analysis, and requires less operator training than many other trace elemental techniques. The objective of this work is to demonstrate the applicability of FAAS using the PerkinElmer® PinAAcle™ 900T to accomplish this task. Seven elements are determined in two commercially available multi-mineral tablets, a NIST® Standard Reference Material, and a commercial reference material which simulates a mixed food diet.

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APPLICATION NOTE

FT-IR Spectroscopy

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Classification of Samples of the Traditional Remedy "Chinese Goldthread" by FT-IR Spectrometry and AssureID software

Introduction

Coptidis Rhizoma (the root of the Coptis chinensis plant), which is also known as goldthread, is one of the most commonly used traditional Chinese medicines (TCM) in China. It can clear heat and reduce fire and is usually used for diarrhoea, vomiting, jaundice, fever, toothache, conjunctivitis and some other conditions.[1]

In a separate note,[2] we have shown that FT-IR spectrometry can be used to identify the presence of the marker compound (berberine) in Coptidis Rhizoma samples, and importantly, to distinguish it from other berberine-containing materials (Phellodendri Chinensis Cortex and Phellodendri Amurensis Cortex). Second-derivative processing and peak location methods were used for the identification.

TCM materials are normally processed before being used clinically, and different processing methods lead to different medicinal effects. The Chinese Pharmacopoeia[1] lists three processing methods for Coptidis Rhizoma. Coptidis Rhizoma processed by rice wine is used for toothache and conjunctivitis, while that processed by ginger juice or the extract of Corni Fructus is used as an anti-emetic and to comfort the stomach. These different processing methods do not bring about an obvious difference in the appearance of the herb, so it is important to develop an analytical method that is capable of distinguishing these materials. In terms of chemical composition, the differences between the raw and the processed herbs may be not as significant as those between different kinds of herbs, so we can expect to require a more careful analysis of the spectra using chemometric tools.

In this note, we show that FT-IR spectrometry combined with AssureID™ software and the COMPARE™ and SIMCA algorithms can be used to distinguish the raw and processed Coptidis Rhizoma samples.

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APPLICATION NOTE

FT-IR Spectroscopy

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Rapid Quality Control of the Traditional Remedy "Chinese Goldthread" by FT-IR Spectrometry

Introduction

Coptidis Rhizoma (the root of the *Coptis chinensis* plant), which is also known as goldthread, is one of the most commonly used traditional Chinese medicines (TCM). It can clear heat and reduce fire and is usually used for diarrhoea, vomiting, jaundice, fever, toothache, conjunctivitis, and some other conditions.[1]

In the Chinese Pharmacopoeia, the isoquinoline alkaloid berberine is considered as the marker component of Coptidis Rhizoma and is used for the identification of this herb.[1] However, berberine is also present in some other herbs, such as Phellodendri Chinensis Cortex and Phellodendri Amurensis Cortex, which are used to treat different conditions.[2,3]

In general, the strategy of relying on a single marker compound to identify a herb is clearly not reliable, as there may be other herbs containing the same compound. Robust identification requires consideration of the entire chemical composition of the sample. Among analytical methods that can provide this information, infrared spectroscopy offers a unique combination of specificity, sensitivity, speed, and convenience of sampling. In recent years, numerous academic papers have been published describing the use of infrared spectroscopy for characterisation of herbal medicines.[4-6] Practical guidelines for successful implementation of the method have recently been published.[7]

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FT-IR Spectroscopy

Authors

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Rapid Quality Control for the Traditional Chinese Remedy "Red Flower Oil" by FT-IR Spectroscopy

Introduction

Red flower oil is a popular remedy in China and Southeast Asia. It is used for the treatment of muscle and joint pain, sprains, and bruising. It is prepared by blending several natural essential oils: principally wintergreen, clove, cinnamon, and turpentine, plus other oils or herbal extracts in smaller quantities.

The most important chemical components in red flower oil are methyl salicylate (from wintergreen oil), eugenol (from clove or cinnamon), and α-pinene (from turpentine). This is recognized in the publication WS3-B-2699-97[1] from the Ministry of Health of the People's Republic of China, which states that these three compounds must be quantified by a gas chromatography (GC) method before the product is approved for sale to the public. According to this document, the minimum contents of methyl salicylate and eugenol are 33.5% v/v and 38% v/v, respectively. Poor quality red flower oil typically has less eugenol, as clove and cinnamon essential oils are more expensive than wintergreen, and natural oil of wintergreen is more easily substituted by industrial methyl salicylate. Another possible source of poor quality is adulteration of the product by addition of excess amounts of inert vegetable or paraffin oil.

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APPLICATION NOTE

UHPLC

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Analysis of Ginsenosides in Ginseng Root with the PerkinElmer Flexar FX-15 System Equipped with a PDA Detector

Introduction

The root of the panax genus plant (also called Ginseng) has been used as an herbal medicine in Asia for over two thousand years for its purported various health benefits, including (but not limited to), antioxidant, anticarcinogenic, anti-inflammatory, antihypertensive and anti-diabetic. The pharmacologically active compounds behind the claims of ginseng's efficacy are ginsenosides; their underlying mechanism of action although not entirely elucidated appears to be similar to that of steroid hormones. There are a number of ginseng species, and each has its own set of ginsenosides. In fact, more than forty different ginsenosides have been identified. Ginsenosides are a diverse group of steroidal saponins with a four ring-like steroid structure with sugar moieties (Figure 1); they are found exclusively in ginseng plants and are in higher concentration in their roots. There are two main groups of ginsenosides: the panaxadiol group or Rb1 group that includes Rb1, Rb2, Rc, Rd, Rg3, Rh2, and Rh3; and the panaxatriol group or Rg1 group that includes Rg1, Re, Rf, Rg2 and Rh1.

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APPLICATION NOTE

Thermal Analysis

Importance of DSC Rapid Cooling for the Analysis of Plastic Microwave Food Trays

DSC 8500

Introduction

Differential scanning calorimetry (DSC) is widely used to characterize the thermophysical properties of polymers. DSC can measure important thermoplastic properties including:

- Melting temperature
- Heat of melting
- Percent crystallinity
- Tg or softening
- Crystallization
- Presence of recyclates/regrinds
- Nucleating agents
- Plasticizers
- Polymer blends (presence, composition and compatibility)

Most DSC experiments on polymers are conducted by heating from ambient conditions to above the melting temperature. But, for some thermoplastics, which do exhibit differences during processing, standard heating DSC may not show any significant differences. A more sensitive test, for detecting subtle, but important differences between different batches of a given thermoplastic, is the DSC isothermal crystallization test.

During the manufacture of plastic products, such as bottles, fibers, films, containers, housings, pipes and trays, the thermoplastic is melted, cooled, thermoformed and crystallized. The complete

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Mass Spectrometry

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Shelton, CT USA

Quantitation of BADGE: An Epoxyphenol-based Food Can Coating in Canned Tuna Extracts Using UHPLC-TOF

Introduction

Metal cans are often coated with a resin barrier to prevent contact between food and the can. Components from these coatings can migrate into the food affecting its safety and quality. Polyepoxyphenol coatings on the inside of cans based on bisphenol A epoxy resins can release the epoxy monomer bisphenol A diglycidyl ether (BADGE) into food (1,2). Bisphenol A and its derivatives are considered as endocrine disruptors (3). Both Europe and the U.S. have set regulations on the limit of BADGE migration into food at 1 mg/Kg. Using the quantitative capability of the AxION® 2 Time-of-Flight (TOF) mass spectrometer, we were able to set up a calibration curve and quantitate BADGE in a tuna extract. In addition, the high mass accuracy capability of the TOF along with the proprietary AxION EC ID software, allowed us to identify an unknown impurity cyclo-di-BADGE without having an authentic standard of this compound.

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APPLICATION NOTE

Gas Chromatography/
Mass Spectrometry

Authors

Miles Snow

Heidi Grecsek

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Shelton, CT 06484 USA

Analysis of Food-Packaging Film by Headspace-GC/MS

Introduction

Food-packaging material is typically manufactured as a thin film and coated with inks which usually contain multiple, harmful, volatile organics. Therefore, they must be carefully monitored and quantitated to ensure that the amounts are limited.

Traditionally, the test for solvent materials in food-packaging film was performed using a technique of heating a square meter of the film material inside a mason jar. This jar is then opened and tested (by smell) for volatile organic compounds. Later, this test was expanded to extract a headspace sample out of the mason jar by syringe and then injected into a gas chromatograph (GC) for quantitative analysis. This produced significantly better results and provided laboratories with a quantitative number. This process is still very time-consuming and labor intensive as a result of the number of manual steps involved. The manual process of cutting food packaging, placing it in a mason jar, heating the jar, and manually collecting a sample for GC analysis dramatically limits the number of samples that can be analyzed each day. The technique demonstrated here will greatly improve the efficiency and throughput of this analysis.

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APPLICATION NOTE

Gas Chromatography

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Monza, Italy

Determination of Residual Solvents in Flexible Packaging According to EN 13628-2:2004

Introduction

The reference standards for food contact materials are rapidly evolving in favor of increasing consumer protection.

The Commission Regulation (EC) No. 1935/2004 is the main reference legislation in the European community. This regulation establishes that any materials that come into contact with food must not release chemicals in quantities which could:

- Pose a danger to the health of consumers
- Result in an unacceptable change in the composition of food
- Change the organoleptic properties

Part 2 of the regulation focuses the attention of food contact material producers on the need to operate in terms of quality assurance. The Commission Regulation (EC No. 2023/2006) has made it mandatory to adopt a system of Good Manufacturing Practice (GMP); with GMP referring to the set of actions to ensure a consistently high quality both in production and control process. This requires not only a deep knowledge of the materials used but also of the entire production and control process.

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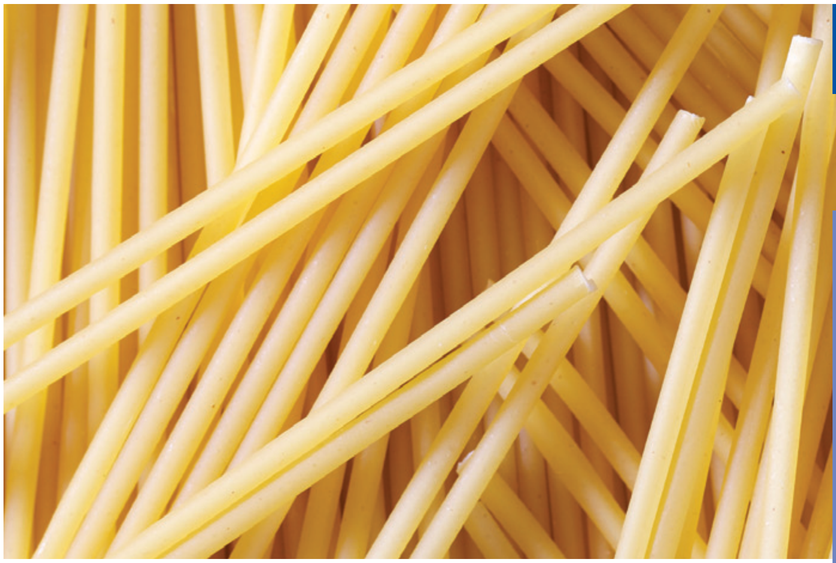
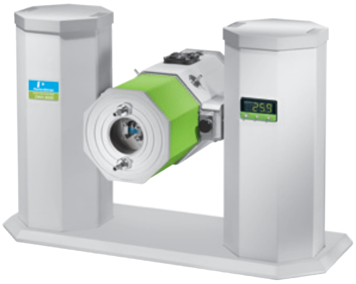


APPLICATION NOTE

Thermal Analysis

Effect of Humidity on Mechanical Properties of Pasta



Summary

The mechanical properties of pasta are very important. In its dried form, it is very brittle and quite stiff. If introduced into a humid environment and heated, the properties of the pasta change dramatically. This application note will investigate the effect of humidity on pasta under isothermal and temperature scanning conditions.

Introduction

Dynamic Mechanical Analysis (DMA) measures the stiffness of a material. The stiffness of pasta is a good indication as to the stability of the dried product. Dried pasta from the packet is quite stiff and humidity and temperature will decrease the stiffness as the material softens. The modulus gives an indication of the stiffness as it decreases and tan δ indicates when the material becomes more viscous and less elastic.

DMA works by applying an oscillating force to the material and the resultant displacement of the sample is measured. From this, the stiffness can be determined and the modulus and tan δ can be calculated. Tan δ is the ratio of the loss modulus to the storage modulus. By measuring the phase lag in the displacement compared to the applied force it is possible to determine the damping properties of the material. Tan δ is plotted against temperature and a glass transition is normally observed as a peak since the material will absorb energy as it passes through the glass transition.

Dried pasta can be stored for long periods without risk of biological decay. When exposed to water, either by immersion or humidity, the pasta will absorb the water and start to hydrate. The humidity and temperature affect the hydration rate significantly as will be demonstrated.

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APPLICATION NOTE

HPLC/ICP - MS

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Arsenic Speciation Analysis in Brown Rice by HPLC/ICP-MS Using the NexION 300D/350D

Introduction

Arsenic (As) is a well-known toxic element which has been highly regulated, especially for drinking water. Although regulatory limits have been for total arsenic, its toxicity varies widely and is dependent on its chemical form. For example, inorganic forms of arsenic are highly toxic and carcinogenic. However, organic forms (such as monomethylarsonic acid, dimethylarsinic acid, and arsenobetaine) are recognized as non-toxic or as having low toxicity.

The Joint Expert Committee on Food and Additives (JECFA) recognizes the importance of monitoring inorganic arsenic intake. In 1988, they established a provisional tolerable weekly intake (PTWI) of 0.015 mg/kg body weight inorganic arsenic. However, this recommendation was withdrawn in 2010.

In Japan, the average total arsenic intake/person/day is divided between seafood (53.6%), vegetables and seaweed (35.4%), rice (7.1%), and other sources.[1]
It is known that the majority of arsenic in marine organisms is in the form of arsenobetaine, which is non-toxic. However, because of the large quantities of rice consumed in Japan, it is important to know what forms of arsenic are present in rice.

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APPLICATION NOTE

HPLC/ICP - MS

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Arsenic Speciation Analysis in White Rice by HPLC/ICP-MS Using the NexION 300D/350D

Introduction

There has been a rising concern about the presence of arsenic in rice, especially in societies which consume large quantities of rice. Arsenic can enter rice naturally through the environment or through the application of pesticides. Because not all arsenic species are toxic, the ability to measure the different forms is important.

In recent years, it has become common to measure different forms of arsenic using HPLC/ICP-MS: HPLC separates the forms and ICP-MS detects them as they elute from the column. The advantage of ICP-MS as an HPLC detector is that it is very sensitive and can measure trace levels, as demonstrated by its use to measure impurities in a wide range of electronic materials and environmental samples.

This work demonstrates the ability to measure various arsenic forms in white rice, building upon previous work.[1,2]

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APPLICATION NOTE

Mass Spectrometry

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Rapid Detection of Vanilla Bean Extract Adulteration with Tonka Bean Extract with No Chromatography or Sample Preparation

Introduction

Vanilla is the second most expensive spice, and is widely used as a flavoring ingredient in the food, beverage, cosmetic, pharmaceutical and tobacco industries. Pure vanilla bean extract is made by soaking at least 13.35 ounces of vanilla beans in a gallon solution containing a minimum of 35% ethyl alcohol in water.

The production of vanilla beans is quite expensive, since it is a very labor intensive process and harvesting takes place two to three years after planting. Due to this, the price of natural vanilla bean extract is quite expensive. It is quite often adulterated with cheaper tonka bean extract, which smells and tastes like vanilla bean extract due to the presence of a compound called coumarin[1]. Since coumarin is absent in vanilla bean extracts, it can be used as a marker compound to detect its adulteration with tonka bean extracts.

Tonka bean extract is banned for human consumption by the FDA due to its adverse health effects caused by the presence of coumarin. Coumarin is banned in foods based on histological evidence of hepatoxicity in animal experiments. Coumarin is toxic to the liver and kidneys and causes thinning of the blood. This is particularly dangerous for people taking blood thinning drugs because the interaction of coumarin and blood thinners can increase the likelihood of bleeding[2].

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APPLICATION NOTE

Mass Spectrometry

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Rapid Differentiation Between Natural and Artificial Vanilla Flavorings for Determining Food Fraud Using AxION DSA/TOF with No Chromatography or Sample Preparation

Introduction

Vanilla is the second most expensive spice, next to saffron, and is widely used as a flavoring ingredient in the food, beverage, cosmetic, pharmaceutical and tobacco industries. Vanilla extract is the most common form of vanilla used today, and comes in 2 forms, natural & artificial. Pure vanilla extract is made by soaking at least 13.35 ounces of vanilla beans in a gallon solution containing a minimum of 35 % ethyl alcohol in water. There are two types of artificial vanilla flavorings, which always contain vanillin that is synthesized from cheap raw material such as guaiacol, eugenol or lignin, a natural polymer found in wood; and/or ethyl vanillin that is added, which is another artificially produced vanilla compound that has three times the flavor strength of vanillin[1].

The production of vanilla beans is quite expensive, since it is a very labor intensive process and harvesting takes place two to three years after planting. This drives the price of natural vanilla extract to about three to five times higher than artificial vanilla preparations. Due to quality, price concerns and economically motivated frauds, it is important to differentiate between natural and artificial forms of vanilla extracts. Apart from vanillin, natural vanilla extracts have 4-hydroxybenzaldehyde, which is absent in artificial vanilla flavorings. This compound can be used as a marker ion to rapidly differentiate between natural and artificial vanilla preparations[2].

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APPLICATION NOTE

FT-IR NIR Spectrometry

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Determination of Levels of Spice Adulteration using Near-infrared Spectroscopy

Introduction

Since the late 1800's scientists have been testing spice samples and discovering that they are adulterated. Some spices are high-value products that can be adulterated with lower-value commodities for commercial gain by unscrupulous suppliers. Some common adulterants of spices range from talc powder, ground walnut shells, cassia bark and sand, to wheat starch, saw dust, millet, buckwheat, and cornstarch. Commonly adulterated spices include garlic powder, black pepper, and cinnamon. Fourier Transform Near-Infrared Spectroscopy (FT-NIR) is shown here to be an effective and rapid technique to determine if these types of spices have been adulterated.

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APPLICATION NOTE

Atomic Absorption

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Analysis of Total Mercury in Chinese Spice Mixtures using Flow Injection Cold Vapor Atomic Absorption Spectrophotometry

Introduction

Spices are often used as dietary components to improve color, aroma, palatability and acceptability of food. Most spices are fragrant, aromatic and pungent.[1] Spices and herbs, grown widely in various regions of the world, have been used for several purposes since ancient times – in folk medicine as antiscorbutic, antispasmodic, tonic, carminative agents against bronchitis, ulcers and as diuretics, depuratives and vermifuges. Also, some species are used as tea flavoring agents in several regions.

Natural food spices such as pepper and mustard have been reported to contain significant quantities of some trace metals.[2] These trace metals in spices and medicinal plants play a vital role as structural and functional components of metallo proteins and enzymes in living cells.[1] Heavy metals have important positive and negative roles in human life. Some of the heavy metals are considered essential including iron, zinc and copper,[3] whereas some other metals, like mercury, have toxic roles in biochemical reactions in our body. Mercury is distributed throughout the environment in a number of different forms – it exists mainly as elemental mercury vapor in the atmosphere, while most of the mercury found in water, sediments, soil, plants, and animals is in the inorganic and organic forms (for example methylmercury) of the element.[4] The habitual addition of mercury-contaminated spices

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APPLICATION NOTE

Atomic Absorption

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Analysis of Arsenic, Cadmium and Lead in Chinese Spice Mixtures using Graphite Furnace Atomic Absorption Spectrophotometry

Introduction

Spices are dried parts of plants. Together with herbs, these plants grow widely in various regions of the world and have been used for several purposes since ancient times. Most are fragrant, aromatic and pungent and are used for culinary purposes to improve color, aroma, palatability and acceptability of food.[1] In addition, they are also used in folk medicine as antiscorbutic, antispasmodic, tonic, carminative agents against bronchitis, ulcers and as diuretics, depuratives and vermifuges. Also, some species are used as tea-flavoring agents in several regions. Natural food spices, such as pepper and mustard, have been reported to contain significant quantities of some heavy metals, including cadmium, lead and arsenic. Exposure to trace and heavy metals above the permissible affects human health and may result in illness to human fetus, abortion and preterm labor, as well as mental retardation to children. Adults also may experience high blood pressure, fatigue and kidney and neurological disorders.[1] Contamination with heavy metals may be accidental (e.g. contamination of environment during plant cultivation) or deliberate – in some cultures, according to traditional belief, specially treated heavy metals are associated with health benefits and are thus an intentional ingredient of traditional remedies. Spices and herbal plants may contain heavy-metal ions over a wide range of concentrations.[2,3]

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APPLICATION NOTE

Atomic Absorption

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Quantification of Essential Metals in Spice Mixtures for Regulatory Compliance Using Flame Atomic Absorption Spectrophotometry

Introduction

Foods, together with water, provide the major proportion of the total daily intake of trace elements by humans. Spices and vegetables are some of the most common foods in the human diet around the world. Besides polluted soil and water, foods can also be contaminated with trace metals by the introduction of mechanized farming, the increasing use of chemicals, food processing and packaging, etc. In order to minimize adverse impact, it is important to measure and continuously monitor the levels of trace elements in various kinds of food materials. Trace element food composition data are also important for both consumers and health professionals. In recent years, food labeling legislation has enforced this requirement. Trace element determination in complex matrices, such as food, often requires sample preparation prior to determination by instrumental techniques.[1]

Cobalt (Co), copper (Cu), manganese (Mn), nickel (Ni) and zinc (Zn) are all essential elements, not only for mammals, but also for plants. They play important roles in many biological processes including carbohydrate and lipid metabolism.[2] For example, a daily copper intake of 1.5 - 2.0 mg is essential and copper at nearly 40 ng/mL is required for normal metabolism of many living organisms.[3] However, copper at higher levels is toxic to the circulatory system and kidneys. The trace element content of food items for all the essential elements mentioned above must be controlled on a daily basis.

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Analysis of Pb, Cd and As in Spice Mixtures using Graphite Furnace Atomic Absorption Spectrophotometry

Introduction

The toxicity and effect of trace heavy metals on human health and the environment has attracted considerable attention and concern in recent years. With an inherent toxicity, a tendency to accumulate in the food chain and a particularly low removal rate through excretion,[1] lead (Pb), cadmium (Cd) and arsenic (As) cause harm to humans even at low concentrations. Exposure to trace and heavy metals above the permissible level affects human health and may result in teratogenicity (reproductive effects). Individuals may also experience high blood pressure, fatigue, as well as kidney and neurological disorders.

Spices, the dried parts of plants, grow widely in various regions of the world, are produced either on small farmlands or naturally grown, and have been used for several purposes since ancient times. Most are fragrant and flavorful and are used for culinary purposes to improve the quality of food.[2] Natural food spices, such as pepper, have been reported to contain significant quantities of some heavy metals, including Pb, Cd and As. Contamination with heavy metals may be accidental (e.g. contamination of the environment during plant cultivation) or deliberate – in some cultures, according to traditional belief, specially treated heavy metals are associated with health benefits and are thus an intentional ingredient of traditional remedies. Spices and herbal plants may contain heavy metal ions over a wide range of concentrations.[3,4] There is often little information available about the safety of those plants and their products in respect to heavy metal contamination. Due to the significant amount of spices consumed, it is important to know the toxic metal concentrations in them.[5]

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UHPLC

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Analysis of Ginsenosides in Ginseng Root with the PerkinElmer Flexar FX-15 System Equipped with a PDA Detector

Introduction

The root of the panax genus plant (also called Ginseng) has been used as an herbal medicine in Asia for over two thousand years for its purported various health benefits, including (but not limited to), antioxidant, anticarcinogenic, anti-inflammatory, antihypertensive and anti-diabetic. The pharmacologically active compounds behind the claims of ginseng's efficacy are ginsenosides; their underlying mechanism of action although not entirely elucidated appears to be similar to that of steroid hormones. There are a number of ginseng species, and each has its own set of ginsenosides. In fact, more than forty different ginsenosides have been identified. Ginsenosides are a diverse group of steroidal saponins with a four ring-like steroid structure with sugar moieties (Figure 1); they are found exclusively in ginseng plants and are in higher concentration in their roots. There are two main groups of ginsenosides: the panaxadiol group or Rb1 group that includes Rb1, Rb2, Rc, Rd, Rg3, Rh2, and Rh3; and the panaxatriol group or Rg1 group that includes Rg1, Re, Rf, Rg2 and Rh1.

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Liquid Chromatography/ Mass Spectrometry

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Analysis of Polyphenols in Saffron Petal Extracts with UHPLC/UV/MS

Introduction

Saffron is one of the most expensive spices by weight and is savored around the world. It is derived from the flowers of the crocus plant *Crocus sativus,* which is mostly cultivated in the Mediterranean and Middle East regions. Saffron has recently received additional focus for its naturally-containing carotenoids and polyphenols, both of which have been reported to have nutraceutical/medicinal value, particularly for their antioxidant and anti-inflammatory characteristics[1].

With the above-mentioned interest in mind, this particular work focused on the characterization of the major polyphenolic compounds that are naturally present in saffron petal extract, using UHPLC (ultra-high pressure liquid chromatography) and both UV/Vis and ESI-MS (electrospray ionization mass spectrometer) detectors. The UV/Vis detector was used for the initial detection of the chromatographically-separated analytes while the complimentary ESI-MS was primarily used for analyte identification/confirmation.

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