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Food contact material and food packaging applications

Solutions for food testing

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Food contact material analysis

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Food packaging and food contact materials (FCM) are used throughout the food and beverage industry. Inevitably, chemical compounds migrate or transfer from these materials into the products themselves. Any food packaging component should not release chemicals that can accumulate in a food or beverage product in quantities sufficient to present a risk for consumers. Regulations are complex; however, a diversity of analytical techniques are available to provide effective tools for monitoring and control.

Intentionally added substances (IAS) are known and permitted added substances that are found within food and beverage products and are rigorously controlled.

IAS are found from the beginning to the end of the food chain, starting at the farm, during factory processing and packaging, and eventually being used in the kitchen for preparation and cooking of food. The materials are diverse, including plastics, paper and board, rubber, ceramics, metal, glass, wood, and cork.

Food contact materials include any type of material that comes into direct or indirect contact with food or beverages at any point in the food chain, from the farm gate to the kitchen (from farm-to-fork). For example, fruit and vegetables are harvested on the farm, then stored and transported in plastic crates. Food factories might use conveyor belts, pipes, valves, mixing vessels, mechanical equipment and different types of containers for storage and transport. Cookware and items such as spatulas, cutlery, kitchen surfaces, and chopping boards are all manufactured from various types of food contact materials including wood. All of these items must comply with regulations establishing their suitability for use in contact with food.

Food packaging materials are also subject to stringent regulations; these include items such as plastic laminates, plastics coatings on paperboard and coatings on metal cans, as well as different materials being used for primary and secondary packaging of food. Adhesives and printing inks found on packaging materials may not be in direct contact with food, but can nevertheless also contribute to the burden of chemicals migrating into food.

Non-intentionally added substances (NIAS) are compounds such as impurities, reaction intermediates, breakdown products of polymer/additives, and contaminants from recycling. When investigating NIAS in food and beverages, the analysis can be challenging as there is very little information on the potential chemicals involved.

Therefore, for NIAS, the approach taken needs to be as nonselective as possible to determine the maximum amount of information, both quantitative and qualitative, to, in turn identify known and unapproved substances—those that can be detected and not identified and those that are simply not detected and enter the food chain. For the detection of both known and unknown NIAS compounds, more robust techniques must be employed to test stimulants, packaging, or foodstuffs in the targeted and non-targeted analysis.

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Accelerated solvent extraction

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The Thermo Scientific™ Dionex™ ASE™ 350 Accelerated Solvent Extractor is often used when testing food packaging materials, food contact materials, and food samples, and analyzing for semi-volatile and non-volatile compounds. It is a particularly efficient technique to reliably extract compounds from polymeric materials. Conditions can be carefully controlled to ensure that the material is not deformed or damaged during the extraction process.

Dionex ASE 350 Accelerated Solvent Extractor

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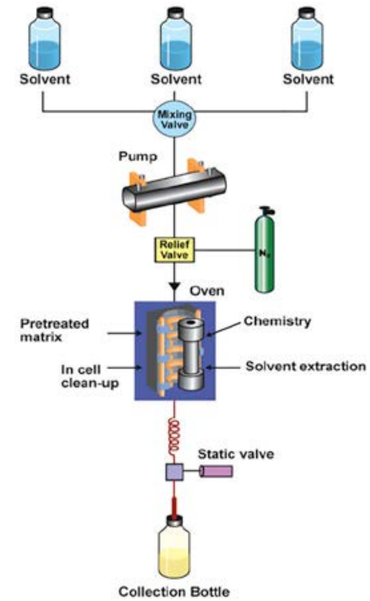
Comparison of Soxhlet and accelerated solvent extraction for analysis of packing material

This application note compares Soxhlet and accelerated solvent extraction techniques in packing material extractable studies. The accelerated solvent extraction technique is an automated technique with several advantages, including efficient extraction, reduced extraction time (<0.5 h/sample), reduced solvent use (<30 mL/sample), and flexibility in solvent selection. The accelerated solvent extraction technique delivers comparable or more efficient extractions than the traditional Soxhlet extraction method using less time and less solvent. Extractions using two solvents of varying polarity are necessary for Controlled Extraction Studies because different solvents can provide additional information on extractables.



Schematic of the accelerated solvent extraction technique and a Thermo Scientific™ Dionex™ ASE™ 350 Accelerated Solvent Extractor.

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Volatile analysis

Meet your stringent requirements for performance, reliability, and value in volatiles analysis with Thermo Scientific™ gas chromatograph (GC) and gas chromatography-mass spectrometer (GC-MS) systems. Combine powerful GC-MS instruments with productivity-enhancing software to create complete solutions for the most challenging food contact material testing.

Thermo Scientific GC instruments provide outstanding performance for routine analyses, advanced capabilities, and the flexibility to increase sample throughput. Instant-connect injectors and detectors enable you to change modules in minutes to reconfigure the instrument for new workflows, develop new methods, and eliminate maintenance downtime. Current developments in GC-MS triple quadrupole technology deliver high sensitivity and selectivity in the small molecule mass range and allow the detection of compounds at low concentrations, even in complex matrix samples. A simple and standard approach using electron impact ionization (EI) enables a very straightforward method for low-level analysis.

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Thermo Scientific™ TSQ™ 8000 Evo Triple Quadrupole GC-MS/MS system

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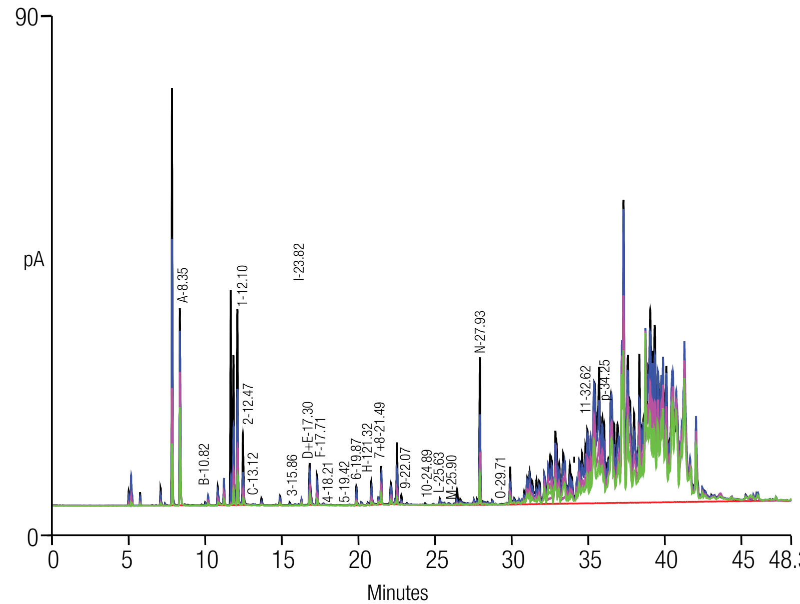
Residual solvents in packaging

Food manufacturers need to ensure the quality of their food for commercial success and to comply with government food safety standards. For this and other applications with non-food products, headspace (HS) gas chromatography is the recommended method of analysis. The method for the quantitative determination of residual solvents in flexible packaging by static headspace is reported in the European Standard absolute method EN 13628-1: 2002. In particular, EN 13628-1: 2002 specifies methods for the quantitative determination of residual solvents in flexible packaging by static headspace chromatography where the chemical identities of the residual solvents are known before commencing analysis. EN 13628-1: 2002 applies to flexible packaging materials that may consist of monolayer or multilayer plastic films, paper or board, foil or combinations thereof and does not apply to residual solvents with amounts lower than 0.5 mg/m². The application note demonstrates, with use of a high-throughput headspace autosampler, the analysis of real samples such as flexible packaging material (plastic film/paper) which shows that residual solvents are present.



Chromatograms of MHE extractions from real samples of transparent plastic film.

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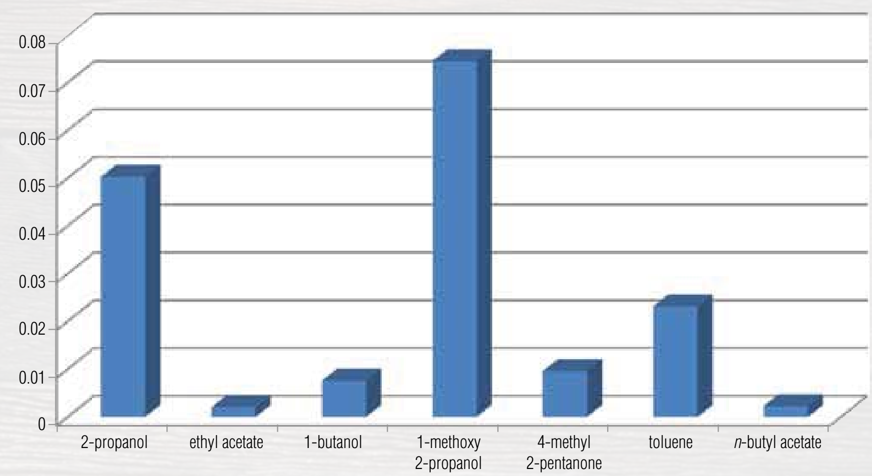
VOCs in food packaging

Accelerated solvent extraction is a high-temperature, high-pressure extraction technique that is widely used for sample extractions in the environmental, chemical and food analysis industries. Extractions at higher temperatures and pressures allow faster extraction of analytes relative to conventional solid/liquid-base extraction techniques such as Soxhlet. Spiked oyster samples were either treated with Dionex ASE Prep MAP and Dionex ASE Prep DE (1:1) or by using sodium sulfate as the drying agent prior to in-cell extraction in the Dionex ASE 350 Accelerated Solvent Extractor. The data shows that Dionex ASE Prep DE is an effective drying agent for wet oyster samples with excellent recoveries for the six OCPs.



VOCs detected in croissant packaging and their relative distribution.

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Determination of phthalates

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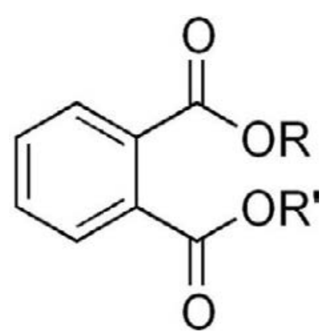
Phthalate plasticizers migrate from plastic containers or closures into soft drinks and alcoholic beverages. They are chemically inert, have high density, low to medium volatility, high solubility in organic solvents, and are easily released to the environment during aging of polymer materials. Phthalate residues in food and beverages are regulated internationally. The China Ministry of Health issued a public notice on June 1, 2011, that phthalate esters are clearly prohibited as non-food substances for use in food. Phthalate acid esters (PAEs) are introduced into the food chain primarily through food packaging material. Alcoholic beverages in plastic containers are a particular risk, because the ethanol provides very good solubility for PAEs. Consequently, the PAEs leach into the beverages from the plastic contact materials.

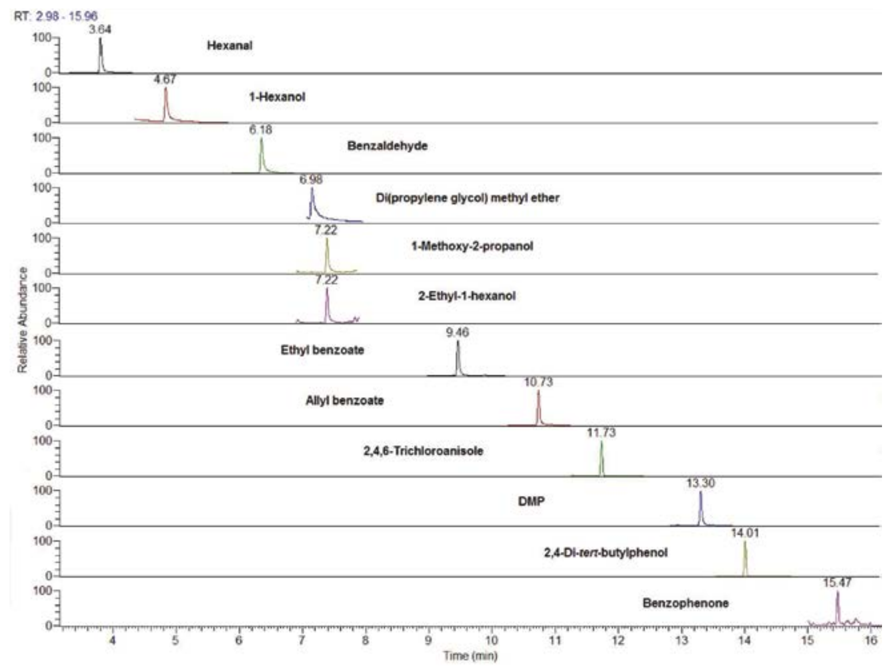
This study follows the China regulation GB/T 21911-2008 for the determining of phthalates in food. The method is sensitive, rapid, accurate, and covers a wide linear range to meet the need for trace level detection of phthalate esters in different types of beverages.



Download the application note

Migration contaminants in paperboard

The packaging of foods is a key activity of the food industry. In this application note, a GC-MS/MS method for developing and validating the determination of packaging migrants in paperboard is discussed. Paperboard can be produced from virgin paper, recycled paper, or a mixture of both. Recycled paperboard is more likely to contain a wide range of dangerous contaminants derived from the degradation of paperboard components, including printing inks, coatings, and adhesives. However, virgin material must also be monitored for unwanted compounds produced during the manufacturing process. The technique used for this analysis includes solid phase micro-extraction (SPME) coupled to gas chromatography triple quadrupole mass spectrometry (GC-MS/MS), enabling detection of volatile and semi-volatile sample components. This technique provides a major advantage because volatile compounds are the most likely to migrate from packaging to food. Identification of the packaging migrants was based upon the presence of transition ions (quantifier and qualifier) at the retention times (± 2.5%) corresponding to known standards. The method was validated in-house using the criteria specified in European Commission Decision 2002/675/EC2 as a guideline.



Chromatogram of spiked paperboard with 12 packaging migrants (c = 0.024–30 mg/kg).

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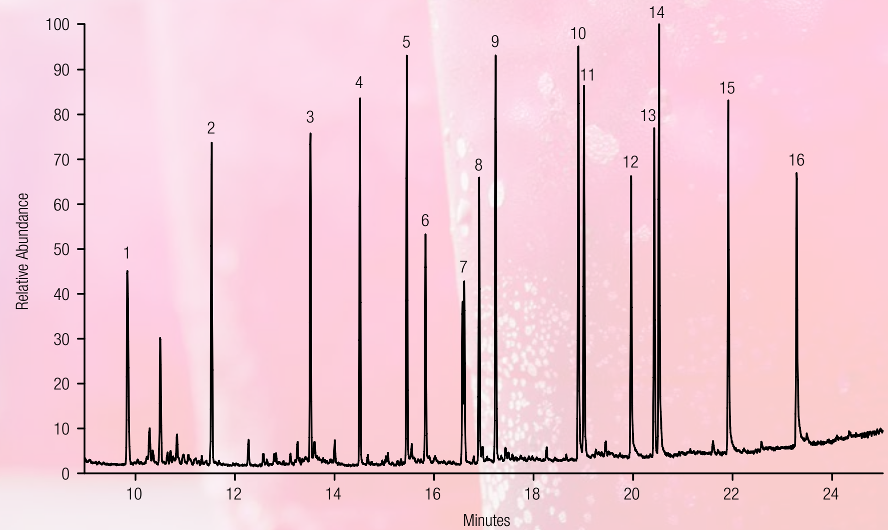
Phthalate esters in soft drinks

Phthalate esters are the main plasticizers used as softening agents in the production of PVC. These compounds are reported to act as endocrine disruptors and exposure to high levels can cause harmful effects in the human reproductive system. There have been reports from the U. S. Food and Drug Administration that certain foods and beverages, particularly fruit juices, contain high levels of phthalates. Phthalate esters are environmentally ubiquitous and may affect measured recoveries. To minimize this problem, care was taken when preparing solutions for calibration and extractions. Longer chain phthalate esters such as DNP, DNOP, and DHXP can adhere to the glassware and lower extraction recoveries were expected.



TIC of 1000 ng/mL phthalate esters standard in full scan 40–450 amu.

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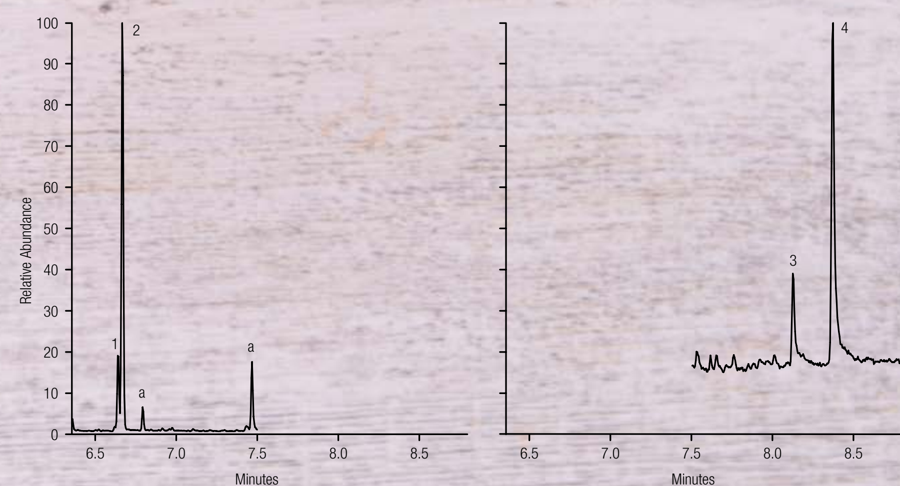
Chemical migrants in breakfast cereal

The Food Standard Agency (FSA), UK, published a method for analyzing benzophenone and 4-hydroxybenzophenone in food. Benzophenone is a chemical migrant associated with inks and coating in food packaging that can migrate to the surface of the food. Directive 2002/72/ EC has a specific migration limit (SML) for benzophenone at 0.6 mg/kg. The QuEChERS methodology has become widely used in food-safety analyses. A modified sample preparation approach, described in the European EN15662 QuEChERS procedure, was used for extracting benzophenones from breakfast cereal and analyzed using GC-MS.



SIM chromatogram for benzophenone and 4-hydroxybenzophenone spiked at 0.6 mg/kg in breakfast cereal.

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Semi-volatile analysis

Bring the power of the first-ever combination of high-resolution gas chromatography (GC) and high-resolution, accurate-mass (HRAM) Orbitrap mass spectrometry to your laboratory. The Thermo Scientific™ Q Exactive™ GC Orbitrap™ GC-MS/MS system provides a comprehensive characterization of samples in a single analysis for the highest confidence in compound discovery, identification, and quantitation. This system offers the quantitative power of a GC triple quadrupole MS combined with the high-precision, full-scan HRAM capabilities only available in combination with Thermo Scientific™ Orbitrap™ technology.

Get high-confidence discovery, identification, and quantitation of food contact materials and packaging migration samples from a single analysis. The Q Exactive GC Orbitrap GC-MS/MS system offers the quantitative power of a GC triple quadrupole MS combined with the high-precision, full-scan, high-resolution, and accurate-mass (HRAM) capabilities. This system is ideal for the analysis of non-intentionally added substances (NIAS) and routine grade performance for both targeted and non-targeted analysis, along with high confidence quantitation for the ultimate sample analysis workflow.

Learn more

Q Exactive GC Orbitrap GC-MS/MS system

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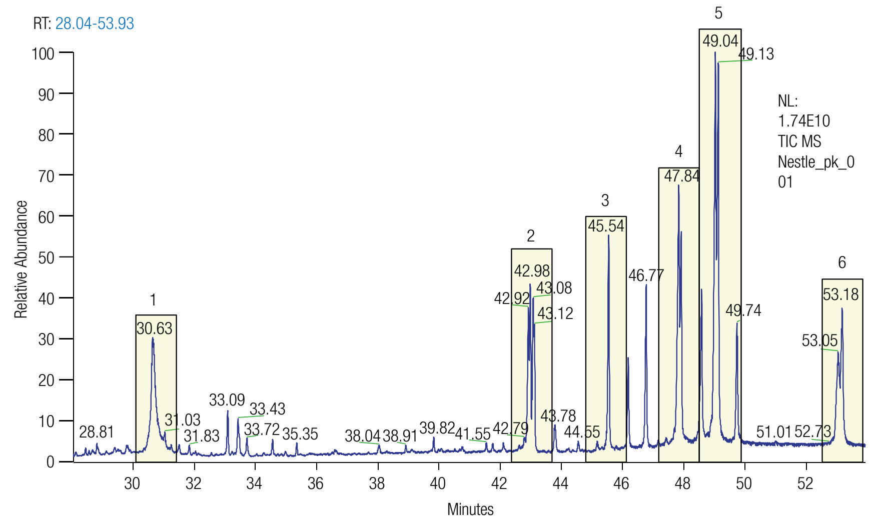
Characterizing unknowns in food packaging

Gas chromatography-mass spectrometry (GC-MS) is a popular analytical technique that has been widely used in food packaging studies. It provides analytical advantages of chromatographic resolution, reproducibility, peak capacity, and, importantly, extensive spectral libraries to aid identification. The analytes of interest are either volatile or semi-volatile (<1000 Da) in nature, and are therefore well suited to analysis by GC-MS. The primary materials, such as monomers, additives, and solvents, used in food packaging are usually well understood. However, these materials can also contain non-intentionally added substances (NIAS) such as impurities, reaction intermediates, breakdown products of polymer/additives, and contaminants from recycling. This study focused on the utilization of a new GC-MS system with high-mass resolution performance and high-mass accuracy for fast and confident identification of unknown compounds in food packaging. The study demonstrates that GC Orbitrap MS is a powerful solution for fast, confident, and comprehensive chemical characterization of food packaging samples.



Zoomed region showing the six peaks of interest in the electron impact (EI) total ion chromatogram of the packaging sample.

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Semi-volatile analysis

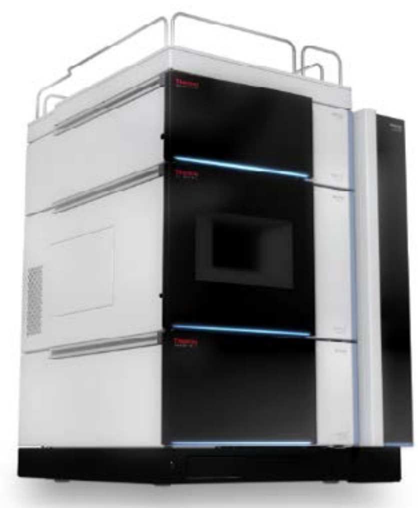
Thermo Scientific™ Vanquish™ UHPLC systems were designed from the ground up to deliver a new standard in UHPLC—more results with better separations without compromise. These easy-to-use, fully integrated, and biocompatible systems feature high-sample capacity for high-throughput workflows, industry-leading pumping performance, exceptionally high S/N and linearity, two-mode thermostatting, and both active and passive preheating in a system that's driven by the gold-standard Thermo Scientific™ Chromeleon™ 7.2 Chromatography Data System software.

- **Drive separation with more power –** The Vanquish UHPLC system was developed to offer more pressure capabilities than ever before, without any trade-off on durability or robustness. From ultra-fast to extremely shallow gradients at pressures up to 1500 bar, the industry-leading fourth generation SmartFlow™ pumping technology of the Vanquish UHPLC system provides you with unmatched retention time reproducibility and lowest baseline noise for the highest detection sensitivity.

- **Handle samples with more accuracy –** The Vanquish UHPLC system combines maximum injection precision with huge sample capacity and high-throughput capabilities for busy food analytical laboratories.

- **Control separation with more confidence –** Temperature rules retention, selectivity, and efficiency in chromatography, and the Vanquish UHPLC system is your key to pave the way to maximum efficiency and resolution through exceptional temperature control.

- **Detect analytes with more sensitivity –** Thermo Scientific™ LightPipe™ technology within the diode-array detector (DAD) provides you with an unmatched detection experience. It achieves a wide linear range and industry-leading signal-to-noise performance, even beyond the capabilities of variable wavelength detectors.



Vanquish UHPLC system

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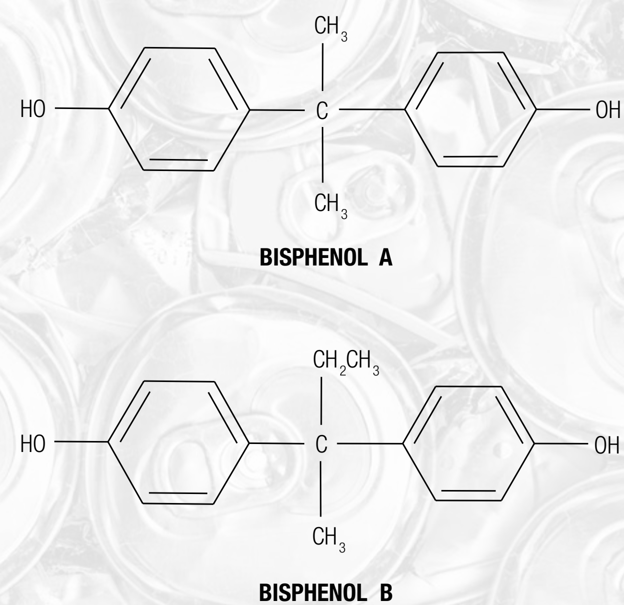
Determination of bisphenols

Bisphenols A and B are monomers used in the production of epoxy resins and plastics and are widespread in food and drink packages as coatings of metal cans and water pipes, as well as microwave susceptors (used to crispen food), and in some dental resins. Bisphenol A and liquid obtained from plastic-coated cans have been shown to be estrogenic in MCF-7 and human breast cancer cells. This method shows that the Thermo Scientific™ Dionex™ UltiMate™ 3000 CoulArray™ Coulometric Array Detector not only offers unrivaled sensitivity and selectivity but also unparalleled stability and maintenance-free routine operation. The bisphenols show characteristic oxidation patterns across the array that are of value when assaying these compounds in complex biological samples. Although it is unclear what minimum levels of these compounds are biologically active, the coulometric array detector can quantitate the bisphenols at sub 50 pg levels, which permits the study of their effects even at low levels.



Chemical structures of Bisphenols A and B.

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Determination of phthalates

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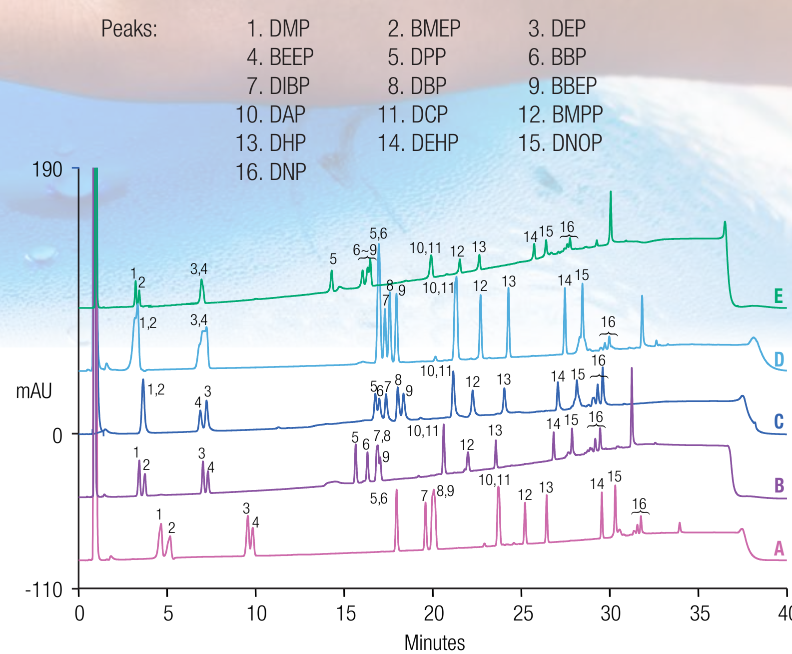
Phthalates are potentially hazardous to human health—especially to children's health—due to their classification as endocrine disruptors. This has resulted in regulations regarding the types and levels of phthalates allowable in plastic toys, water containers, textiles, and foods.

The simultaneous determination of phthalates using UHPLC that can separate all the phthalates listed in the enacted standard methods has not yet been published. In this application note, we describe a method that can separate all the phthalates listed in the standard methods and directives, for example in foods GB/T 21911-2008 and HJ/T 72-2001 in water.



Chromatograms of phthalates listed in GB/T 21911-2008 using five different columns.

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Semi-volatile analysis

MS detection has become more affordable, accessible, and even mandatory in many fields of research. Only detecting a peak is no longer sufficient; now you also must know what that particular peak is. When our pioneering Vanquish UHPLC system is combined with our best-in-class mass spectrometers, you achieve an extra level of confidence.

The Thermo Scientific™ TSQ Altis™ and TSQ Quantis™ triple quadrupole mass spectrometers are the perfect matches for a wide range of quantitation workflows that utilize selected reaction monitoring (SRM) analyses.

Vanquish UHPLC and TSQ Altis Triple Quadrupole Mass Spectrometer

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Analysis of plasticizer contaminants

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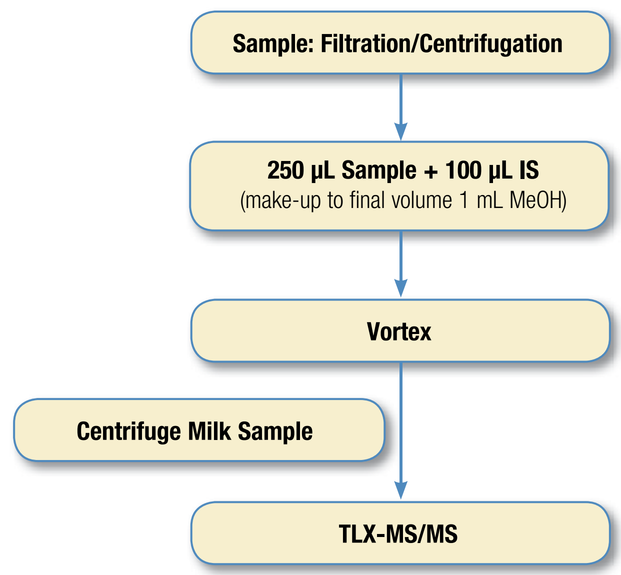
Phthalates are endocrine active chemicals used in a variety of consumer products. In some markets, restricted levels of certain phthalates are permitted for use in food contact materials, but they are not permitted as direct food additives. Phthalates have been used to deliberately adulterate beverages and sports drinks in Taiwan, and phthalates and other plasticizers are widely found as ubiquitous contaminants particularly in fatty foodstuffs. Contamination arises from numerous sources such as the environment and food packaging. Cross-contamination with phthalates can easily arise during trace analysis in the laboratory, and there are significant advantages in minimizing sample handling through online automated analysis.



Schematic of method.

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LC-HRAM

Non-volatile analysis

For polymer additives and monomers in food and food packaging analysis, identify and confirm more compounds rapidly and with confidence. The Thermo Scientific™ Vanquish™ Horizon UHPLC system is a great match for our unique Thermo Scientific™ Orbitrap™ mass analyzer technologies including the Thermo Scientific™ Exactive™ series of benchtop mass spectrometers or the revolutionary Thermo Scientific™ Orbitrap Fusion™ Tribrid™ mass spectrometer.

Take full advantage of all the benefits of high-resolution, accurate-mass detection by LC-MS to identify, characterize, and quantify unknown and known compounds within complex matrices.

Vanquish UHPLC and Thermo Scientific™ Q Exactive™ Focus Hybrid Quadrupole-Orbitrap™ MS System

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Identification of known and unknown food contact materials

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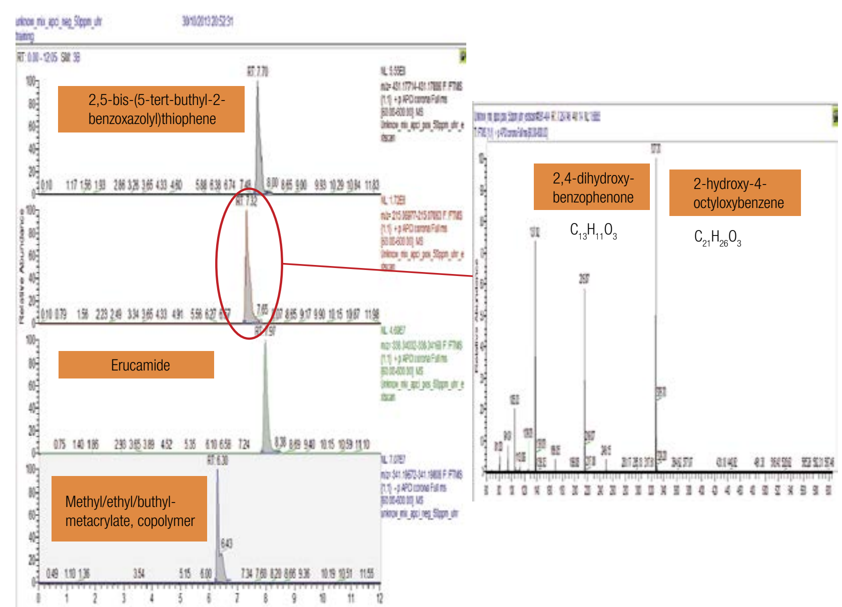
Packaging materials (PM) and non-intentionally added substances (NIAS) are currently one of the hottest research topics in food safety relevant applications. Current research focuses especially on the identification of non-expected or up-to-now unknown compounds.

This poster aims to present such approaches supported by high-resolution MS (HRMS)-based LC-MS methods for the identification of known authorized or unauthorized substances. All test compounds could be identified correctly. HRMS is essential for high confidential identification of both known and unknown compounds.



Overview of the identified four unknown compounds.

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Phthalate screening in food packaging

Phthalic acid di-esters (PAEs), also known as phthalates, are widely used in industry as plasticizers in everyday products like toys, flooring, personal care products, and food packages. These compounds can be present up to a high ratio in some materials. As substances classified as semi-volatile organic compounds (SVOC), they are evaporating into the environment over a long time. In total, 13 lid gaskets, nine milk packages, five bags and fruit containers were tested. Results of full MS analysis and HCD fragmentation of standards were compared with spectra obtained from commercial food packaging and food contact materials. Specific MS2 fragments were used to confirm the presence of banned substances. Direct analysis in real time (DART) combined with Orbitrap mass analyzer-based HRAM LC-MS/MS was shown to be a very fast and convenient way for screening for additives in food packaging and other goods.

List of analyzed phthalate standards. Exact masses of $[M+H]^+$ for precursors as well as for a selection of characteristic HCD fragments are displayed.

Compound	Elemental composition	Precursor $[M+H]^+$	Selection of characteristic HCD fragments m/z	Regulation
Di-n-butyl phthalate (DBP)	$C_{16}H_{22}O_4$	279.1591	167.0339; 205.0859; 223.0965	CA Prop 65
Diisobutyl phthalate (DiBP)	$C_{16}H_{22}O_4$	279.1591	167.0339; 205.0859	EU
Benzyl butyl phthalate (BBP)	$C_{19}H_{20}O_4$	313.1434	91.0542; 205.0859	CA Prop 65
Bis(2-ethylhexyl)phthalate (DEHP)	$C_{24}H_{38}O_4$	391.2843	167.0339; 279.1591	CA Prop 65
Di-n-octyl phthalate (DnOP)	$C_{24}H_{38}O_4$	391.2843	167.0339; 261.1485	CA Prop 65
Dioctyltere phthalate (DOTP)	$C_{24}H_{38}O_4$	391.2843	167.0339; 261.1485; 279.1591	-
Diisononyl phthalate (DiNP)	$C_{26}H_{42}O_4$	419.3156	127.1481; 275.1642; 293.1747	CA Prop 65
Diisodecyl phthalate (DiDP)	$C_{28}H_{46}O_4$	447.3469	141.1638; 289.1798; 291.1955; 307.1904	CA Prop 65

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Trace elemental analysis

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Elemental impurities

Research, production, and analytical laboratories worldwide rely on rapid, efficient, qualitative and quantitative analysis. Our innovative instruments and user-friendly software serve a range of industrial, educational, environmental, and health markets. We offer a comprehensive portfolio for the quantification and identification of trace elemental species at ppm to sub-ppt levels in addition to isotopes. From process and quality control in the lab and on the line to instant, actionable data in the field, we enable more confident analysis and better decision making.

Elemental impurities are common in printed materials, pigments, foil packaging, and delivery systems. Meet the most challenging regulatory requirements for trace metals analysis in packaging migration and food contact materials. Thermo Scientific atomic absorption spectroscopy (AAS) offers high-performance, double beam, graphite furnace and dual-atomizer double beam spectrometers for cost-effective, efficient, and accurate trace elemental analysis. Inductively coupled plasma mass spectrometry (ICP-MS) allows for a broad portfolio of spectrometers to perform accurate and reliable quantitation of inorganic elements at trace levels for routine and research analysis. Whereas, inductively coupled plasma optical emission spectrometry (ICP-OES) is a range of excellent value, multi-element spectrometers featuring advanced hardware design for excellent stability and analyte detection capability.

Thermo Scientific™ iCE™ 3300 AAS

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Tin in canned fruit juice

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Small amounts of metal can adversely affect the flavor and storage properties of food products. The metal is introduced as a contaminant during processing of food products and can accumulate during storage due to leaching from the containers. The United Food Standards Agency (UK) regulations permit a maximum guideline limit of 250 mg/kg in canned foods. However, improvements to the processing and use of new materials for canning mean food manufacturers are required to measure significantly lower concentrations. The traditional flame atomic absorption spectrometric determination of tin is relatively insensitive, and accurate quantification at low concentrations is difficult. Tin can be successfully determined by graphite furnace atomic absorption spectrometry using an accurate background correction system provided care is taken to minimize losses during the program cycle.

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The need for nanoparticle (NP) characterization has exploded in recent years due to the ever-increasing use of engineered nanoparticles (EN) in various industries and the consequent studies that investigate the environmental and consumer risk. Of the methods developed with this goal in mind, field flow fractionation (FFF) coupled to ICP-MS has proved to be one of the most promising. FFF is compatible for particle sizes in the low nm to low µm range and is thus perfectly suited to NP separation. Another promising approach for NP characterization is single particle ICP-MS (sp-ICP-MS). Through direct analysis of an appropriately diluted solution containing NPs, the NPs can be counted. The more sensitive an instrument, the smaller the particle it can detect.

Nanoparticle characterization in food faces many challenges due to the presence of complex matrices and the lack of fully developed and validated sample preparation protocols. An approach based on enzymatic extraction, followed by fractionation using FFF and final nanoparticle characterization with sp-ICP-MS was investigated for a chicken meat paste.

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Peer reviewed journal references

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Recommended resources
www.thermofisher.com/foodcontactmaterials

Peer reviewed journals:
LC and LC-MS methods

Title	Authors	Publication
Release of non-intentionally added substances (NIAS) from food contact polycarbonate: Effect of aging	Chiara Bignardi, Antonella Cavazza, Carmen Laganà, Paola Salvadeo, Claudio Corradini	Food Control, Volume 71, January 2017, Pages 329–335
UHPLC-high-resolution mass spectrometry determination of bisphenol A and plastic additives released by polycarbonate tableware: influence of aging and surface damage	Chiara Bignardi. Antonella Cavazza, Carmen Laganà, Paola Salvadeo, Claudio Corradini	Analytical and Bioanalytical Chemistry, October 2015, Volume 407, Issue 26, Pages 7917–7924
Targeted and untargeted data-dependent experiments for characterization of polycarbonate food-contact plastics by ultra high-performance chromatography coupled to quadrupole Orbitrap tandem mass spectrometry	Chiara Bignardi. Antonella Cavazza, Claudio Corradini, Paola Salvadeo	Journal of Chromatography A, Volume 1372, 12 December 2014, Pages 133–144
Liquid chromatography–full scan-high resolution mass spectrometry-based method towards the comprehensive analysis of migration of primary aromatic amines from food packaging	Monica Mattarozzi, Francesca Lambertini, Michele Suman, Maria Careri	Journal of Chromatography A, Volume 1320, 13 December 2013, Pages 96–102

Methods

Title	Authors	Publication
The challenge of identifying non-intentionally added substances from food packaging materials: A review	C. Nerin, P. Alfaro, M. Aznar, C. Domeño	Analytica Chimica Acta, Volume 775, 2 May 2013, Pages 14–24
Rapid qualitative analysis of phthalates added to food and nutraceutical products by direct analysis in real time/Orbitrap mass spectrometry	Randy L. Self, Wen-Hsin Wu	Food Control, Volume 25, Issue 1, May 2012, Pages 13–16
PCDD and PCDF levels in paper with food contact	H. Beck, A. Droß, W. Mathar	Chemosphere, Volume 25, Issues 7–10, October–November 1992, Pages 1533–1538

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