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Chromatography for Foods and Beverages

Adulteration and Authentication Applications Notebook

Analytical Methods for Effective and Efficient Confirmation

Thermo SCIENTIFIC

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Adulteration and Authentication

Introduction

Guaranteeing the authenticity of a product is integral to quality procedures, essential in the compliance of food labelling regulations and important to consumers when paying for premium products. Adulteration refers to the addition of substances to foods or substitution of components with cheaper alternatives, usually for economic gain (also called food fraud). Adulterants can lead to serious health issues. Food authenticity and adulteration testing encompasses multiple approaches and techniques that are constantly evolving to meet emerging challenges.

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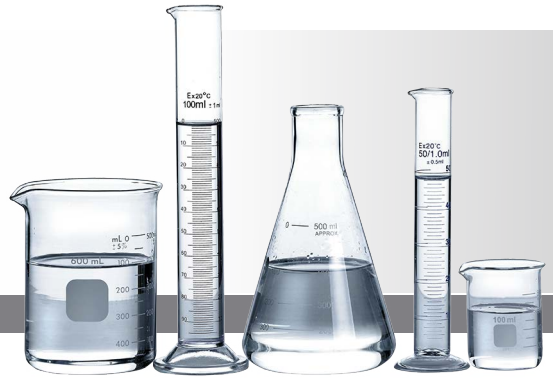

Analytical Technologies

High-Performance Liquid Chromatography

Thermo Scientific™ Vanquish™ UHPLC System and Thermo Scientific™ Dionex™ UltiMate™ 3000 UHPLC+ systems offer excellent chromatographic performance, operational simplicity and unrivaled flexibility. Choose from a wide range of standard and unique specialty detectors to extend your laboratory's analytical capabilities.

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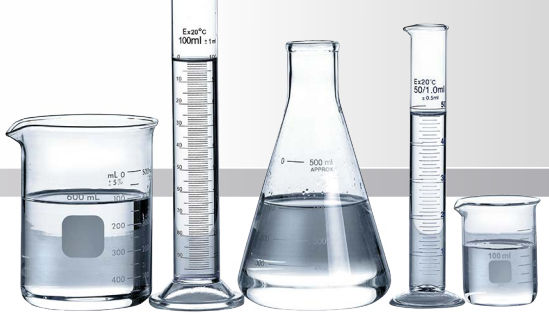

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The Vanquish UHPLC System

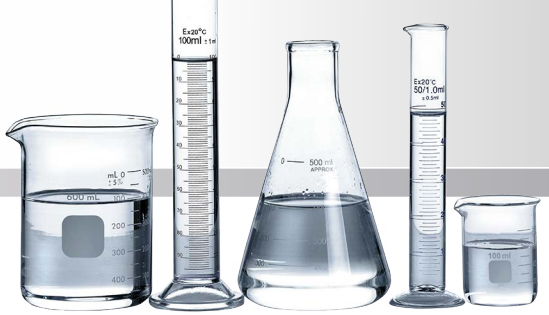
The next generation in UHPLC innovations

The Vanquish system takes high-end UHPLC to a new level, offering more resolution while meeting the throughput demands of modern laboratories. The system is delivers better separations, more results and easier interaction, simultaneously, without compromise.

Delivering the new standard in UHPLC

- More powerful separations with 1500 bar of pump pressure at flow rates up to 5 mL/min
- Industry-leading flow and gradient precision
- Excellent injections up to 100 µL in 0.01 µL increments
- Automated workflows with barcode reading for simplified setup and tracking
- Maximum sample capacity with up to 23 well plates, or 8832 samples
- More confident separations with a wide temperature range of 5 °C to 120 °C for two thermostatting modes and active column pre-heating for improved precision
- UV detection with linear response up to 3000 mAu and noise levels as low as 3 µAu
- Thermo Scientific™ LightPipe™ technology assures lowest peak dispersion with UV detection
- Available Vanquish Charged Aerosol detector for quantification of non-chromophoric compounds

Vanquish Diode Array Detector with LighPipe technology



UHPLC Portfolio

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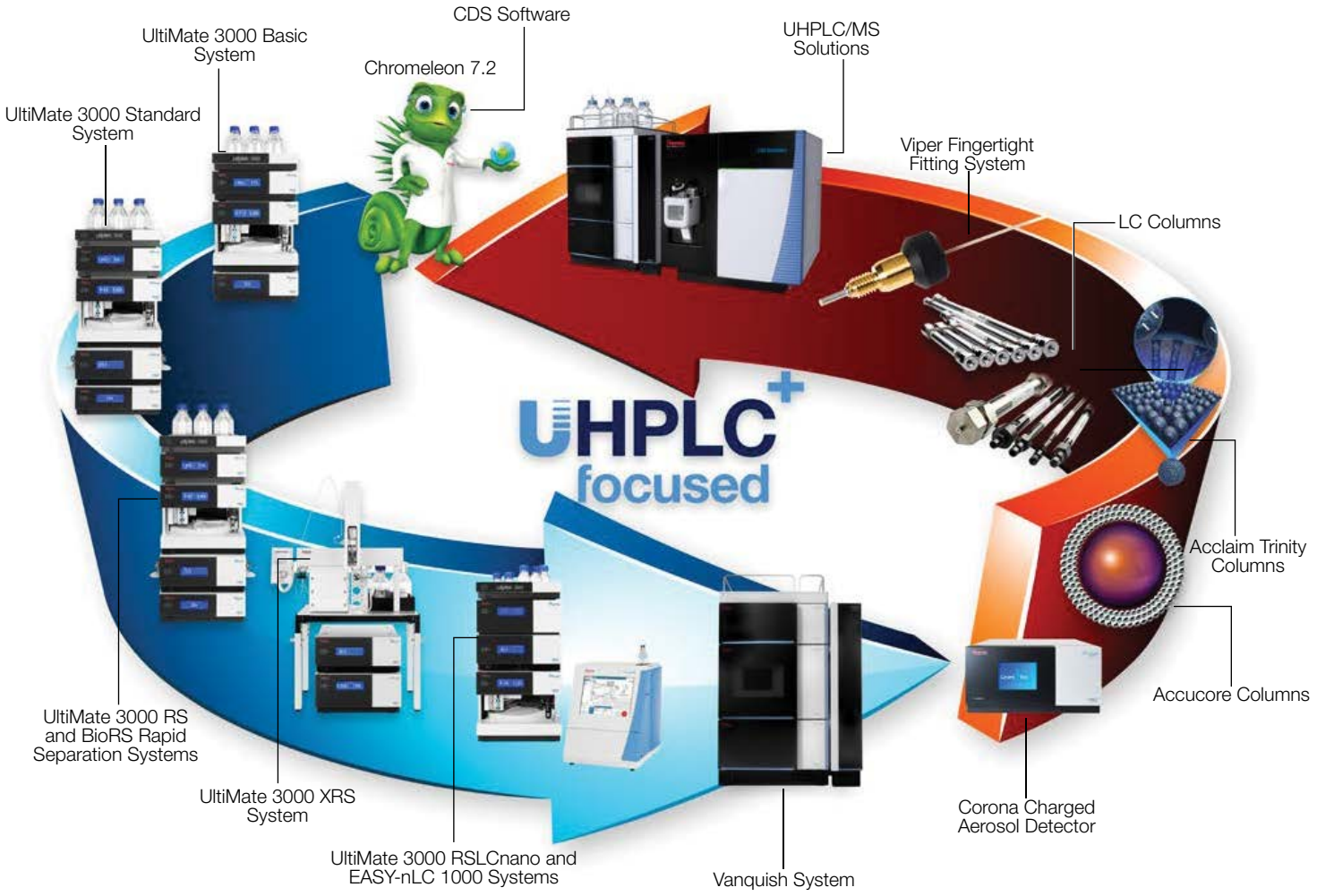

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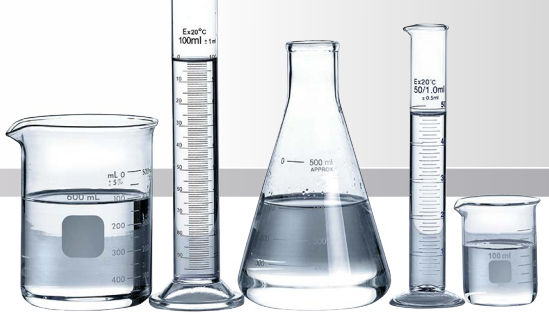

UltiMate 3000 UHPLC+ Systems

Best-in-class HPLC systems for all your chromatography needs

UltiMate 3000 UHPLC+ Systems provide excellent chromatographic performance while maintaining easy, reliable operation. The basic and standard analytical systems offer ultra HPLC (UHPLC) compatibility across all modules, ensuring maximum performance for all users and all laboratories.

Covering flow rates from 20 nL/min to 10 mL/min with an industry-leading range of pumping, sampling, and detection modules, UltiMate 3000 UHPLC+ Systems provide solutions from nano to semipreparative, from conventional LC to UHPLC.

Superior chromatographic performance

- UHPLC design philosophy throughout nano, standard analytical, and rapid separation liquid chromotography (RSLC)
- 620 bar (9,000 psi) and 100 Hz data rate set a new benchmark for basic and standard analytical systems
- RSLC systems go up to 1000 bar and data rates up to 200 Hz
- ×2 Dual System for increased productivity solutions in routine analysis
- Fully UHPLC compatible advanced chromatographic techniques
- Thermo Scientific™ Dionex™ Viper™ and nanoViper™ fingertight fittings—the first truly universal, fingertight fitting system even at UHPLC pressures

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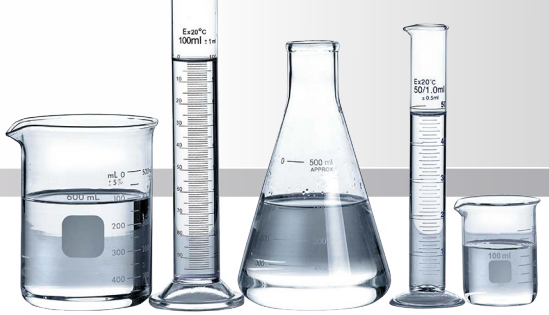

UltiMate 3000 UHPLC+ Systems

We are uniquely focused on making UHPLC technology available to all users, all laboratories, and for all analytes.

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Rapid Separation LC Systems
The extended flowpressure footprint of the RSLC system provides the performance for ultrafast high-resolution and conventional LC applications.

Standard LC Systems
Choose from a wide variety of standard LC systems for demanding LC applications at nano, capillary, micro, analytical, and semipreparative flow rates.

RSLCnano Systems
The Rapid Separation nano LC System (RSLCnano) provides the power for high resolution and fast chromatography in nano, capillary, and micro LC.

Basic LC Systems
UltiMate 3000 Basic LC Systems are UHPLC compatible and provide reliable, high performance solutions to fit your bench space and your budget.

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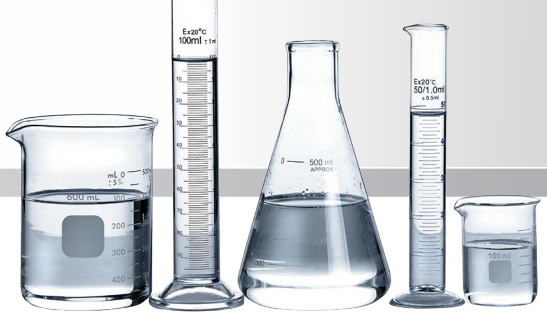

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Advanced Detection Capabilities

Charged Aerosol Detection

Charged Aerosol Detection provides near universal detection independent of chemical structure for non- or semi-volatile analytes with HPLC and UHPLC. Thermo Scientific™ Dionex™ Corona™ Veo™ and Vanquish Charged Aerosol detectors are ideally suited as a primary detector for any laboratory, while providing complementary data to UV or MS methods. No other LC detector available today can match the performance of a Corona Veo detector.

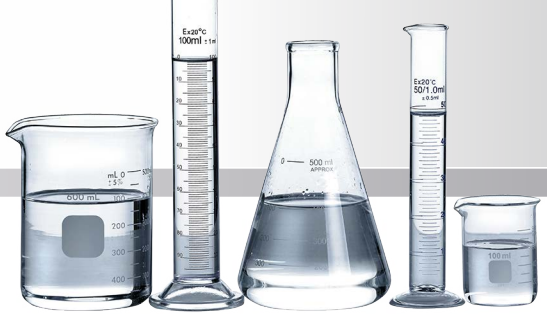
- High sensitivity – single-digit nanogram on column
- Consistent response – independent of chemical structure
- Wide dynamic range – to four orders of magnitude or greater
- Simple to use – easy to integrate with any HPLC/UHPLC system

Charged aerosol detectors give the simplicity, reproducibility and performance required for a full range of applications from basic research to manufacturing QC/QA. With charged aerosol detection you get predictable responses to measure analytes in direct proportion to their relative amounts for quantitation without actual standards.

This detector offers the flexibility to use reversed-phase gradients, as well as normal phase and HILIC modes of separation on any LC system. And, in many cases eliminates the need for derivatization or sample pre-treatment to provide real dilute-and-shoot simplicity.

Corona Veo Charged Aerosol Detector

Vanquish system with Charged Aerosol Detector



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Advanced Detection Capabilities

CoulArray Multi-electrode Array Detector

The Thermo Scientific™ Dionex™ CoulArray™ Multi-electrode Array detector is the only practical multi-channel electrochemical detection system that allows you to measure multiple analytes simultaneously, including those that are chromatographically unresolved. The CoulArray detector delivers the widest dynamic range of any available electrochemical detector with unmatched selectivity for detection of trace components in complex matrixes, even when used with aggressive gradients.

- Measures analytes from femtomole to micromole levels
- Greatly simplify sample preparation and eliminate interferences
- Simultaneously analyze multiple analytes in very complex samples
- Easily produce qualitative information for compound identification

Multiple system configurations offer 4, 8, 12, or 16 channels that can be upgraded anytime. The unique data acquisition and processing software uses automatic signal ranging and a unique patented baseline correction algorithms to provide identification and quantitation of single or multiple analytes and powerful 3D data for quick sample fingerprint confirmation with integration to pattern recognition platforms.

With the power of coulometric array technology, the CoulArray detector can give you the qualitative data of a optical PDA with 1,000 fold greater sensitivity to profile the characteristic qualities of products, determine integrity, identify adulteration and even evaluate competitors' products.

CoulArray Multi-electrode Array Detector

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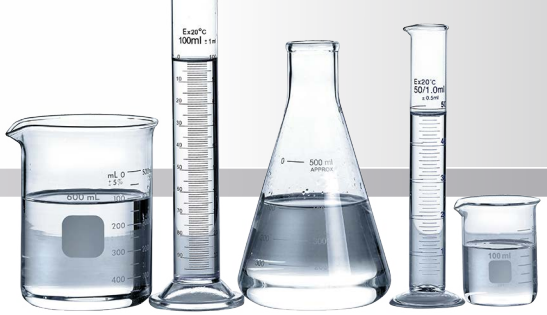

Advanced Detection Capabilities

RefractoMax 521 Refractive Index Detector

The Thermo Scientific RefractoMax 521 Refractive Index Detector from ERC Inc. This detector, in combination with the UltiMate 3000 system, is the right choice for the isocratic analysis of sugars, polymers, and fatty acids. It features fast baseline stabilization and excellent reproducibility, combined with high sensitivity. The RefractoMax 521 is fully controlled by Thermo Scientific™ Dionex™ Chromeleon™ Chromatography Data System Software (CDS), and can also operate in stand-alone mode.

- The detector is highly sensitive and applicable universally. It provides very stable baselines with a drift of 0.2 µRIU/h and a noise specification of 2.5 nRIU or less
- The optical bench, thermostatically regulated from 30 °C to 55 °C, and the superior signal-to-noise ratio ensure highly precise measurement results
- The extended flow rate range from 1 mL/min up to 10 mL/min and the operating range of 1.00 to 1.75 RIU enable the use of this detector for a wide range of applications
- Applications include the analysis of all compounds with low UV-Vis activity, such as alcohols, mono- and polysaccharides, esters, fatty acids, or polymers
- An Auto Set-up function automates purging, equilibration, autozero, and the control baseline stability and noise
- Operation with Chromeleon CDS makes the detector easy to use and ensures maximum productivity in instrument control, data processing, and reporting of results

RefractoMax 521 Refractive Index Detector

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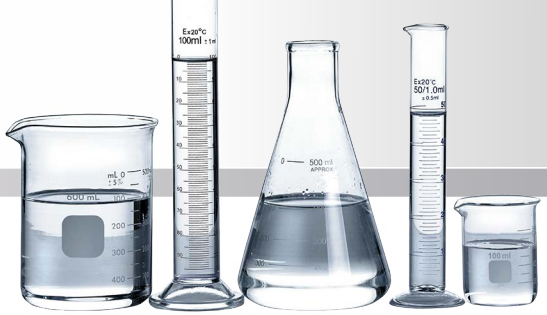

Advanced Detection Capabilities

UltiMate 3000 Diode Array and Multiple-Wavelength Detectors

The Thermo Scientific Dionex UltiMate DAD 3000 detector is a high-resolution, 1024-element diode array detector (DAD) available in Rapid Separation (200 Hz) and Standard (100 Hz) versions. It operates with Chromeleon CDS software to provide a variety of spectra views, including 3-D plotting and automated chromatogram handling. The high resolution and low-noise performance of the DAD-3000 family makes it ideal for the most sensitive and accurate library searches and peak purity analyses.

The detector is also available as a multiple wavelength detector (MWD) in Standard (100 Hz) and Rapid Separation (200 Hz) versions.

- Data collection at up to 200 Hz using a maximum of eight single-wavelength data channels and one 3-D field (3-D only with DAD-3000 (RS)) for best support of ultrafast separations
- Standard versions operate at up to 100 Hz data collection rate for optimum support of 62 MPa (9000 psi) UltiMate 3000 Standard systems
- Accurate compound confirmation with a 1024-element, high resolution photodiode array
- Flexibility in both UV and Vis applications with 190–800 nm wavelength range
- Low-noise over the full spectral range using deuterium and tungsten lamps
- Fast and accurate wavelength verification using a built-in holmium oxide filter
- The detector can be upgraded with the UltiMate PCM 3000 for accurate monitoring pH gradients
- Excellent reliability and reproducibility with low baseline drift (typically < 500 µAU/h)
- Simplified routine maintenance with front access to pre-aligned cells and lamps
- ID chips on flow cells and lamps for identification and life-span monitoring
- Chromeleon CDS software for full control and flexible data handling
- Front-panel display for easy monitoring of detector status to maximize uptime
- Flow cells for semi-micro, semi-analytical, analytical, and semi-preparative applications
- Flow cells available in stainless steel and biocompatible versions

UltiMate 3000 DAD-3000 Diode Array Detector

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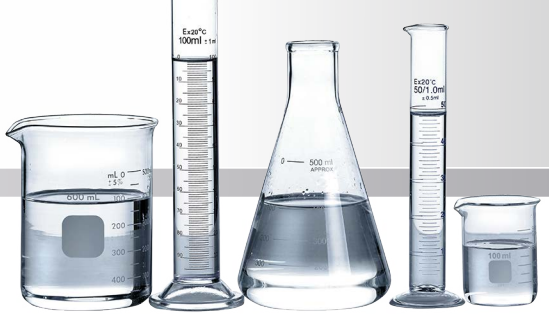

Advanced Detection Capabilities

UltiMate 3000 Electrochemical Detector

Electrochemical detection delivers high sensitivity for neurotransmitter analysis, simplicity and robustness for pharmaceutical or clinical diagnostics, and the selectivity for the characterization of complex samples such as natural products, biological tissues and fluids. For today's researcher, there is a continuing need for detecting vanishingly small quantities of analyte and often in complex samples. Because electrochemical detection measures only compounds that can undergo oxidation or reduction it is both highly sensitive and very selective.

The Thermo Scientific Dionex UltiMate 3000 Electrochemical Detector, designed by the pioneers of coulometric electrochemical detection, delivers state-of-the-art sensor technologies complete with an entire range of high performance and ultra-high performance LC systems optimized for electrochemical detection. The UltiMate 3000 ECD-3000RS takes electrochemical detection to the next level with UHPLC compatibility, total system integration, and selection of detection mode, all with unprecedented operational simplicity.

Features include:

- Detection Modes – choose from DC and PAD for optimum analyte response
- Choice of sensors – both coulometric and amperometric sensors to meet the demands of any application
- UHPLC compatibility – ultralow peak dispersion and high data acquisition rates for conventional or fast, high resolution chromatography
- Modularity – easily expandable to multiple independent sensors for unrivaled flexibility
- Autoranging – simultaneously measure both low and high levels of analytes without losing data
- SmartChip™ technology – easy operation with automatic sensor recognition, event logging and electrode protection

UltiMate 3000 Electrochemical Detector

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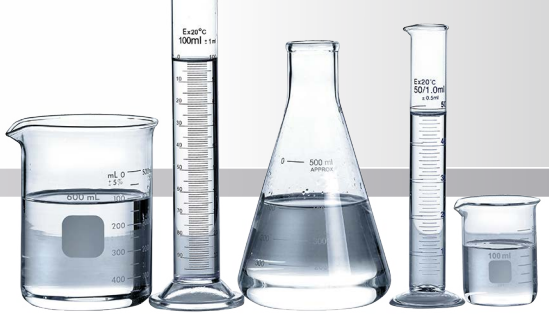

Advanced Detection Capabilities

UltiMate 3000 Fluorescence Detector

The Thermo Scientific Dionex UltiMate 3000 FLD-3000 is a high-sensitivity fluorescence detector series for UltiMate 3000 HPLC systems. It is available in Rapid Separation (RS) and Standard (SD) versions. The optics of the FLD-3000 series provide maximum stray-light suppression for best detection sensitivity. Operated with the Chromeleon CDS software, the detector provides automated qualification, various tools for method development, and instrument wellness monitoring for ease of use, maximum uptime, and the highest degree of regulatory compliance.

- Data collection at up to 200 Hz for optimal support of even the fastest UHPLC separations (FLD-3400RS)
- Standard detectors operate at up to 100 Hz data rate for optimum support of 62 MPa (9,000 psi) UltiMate 3000 standard systems
- Lowest limits of detection with a Raman signal-to-noise ratio (S/N): > 550 ASTM (> 2100 using dark signal as noise reference)
- Unsurpassed reproducibility with active flow cell temperature control for stable fluorophore activity independent of changes in ambient temperature
- Long-life xenon flash lamp for highest sensitivity and long-term operation without the need for frequent lamp changing
- Optional second photomultiplier (PMT) for unique Dual-PMT operation, offering an extended wavelength range up to 900 nm without sacrificing sensitivity in the standard wavelength range
- Two-dimensional (2D) or three dimensional (3D) excitation, emission, or synchro scans to provide the highest degree of flexibility for method development or routine sample characterization
- Innovative Variable Emission Filter for real-time compound-related sensitivity optimization (FLD-3400RS only)
- Large front-panel display for easy monitoring of the detector status
- Two flow-cell sizes for easy optimization to application requirements: the 8 µL flow cell is ideal for trace analysis, and the 2 µL flow cell offers best peak resolution with narrow-bore HPLC and UHPLC columns

Ultimate 3000 Fluorescence Detector

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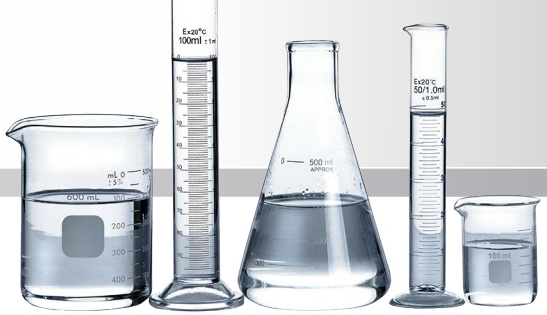

Advanced Detection Capabilities

UltiMate 3000 Variable Wavelength Detectors

The Thermo Scientific Dionex UltiMate 3000 VWD-3000 is a variable wavelength detector (VWD) series for industry leading UV-Vis detection. The forward optics design and wide range of available flow cells ensure optimal performance over a flow rate range of five orders of magnitude. Automated qualification, performance optimization, and instrument wellness monitoring deliver maximum uptime, simplify work-flow, and give you full confidence in your analytical results. The detector is available in a standard 100 Hz (VWD-3100) and a 200 Hz Rapid Separation version (VWD-3400RS) for the most challenging UHPLC applications.

High-Performance UV-Vis Detection

- The VWD-3400RS variant provides data collection rates of up to 200 Hz for optimal support of today's and tomorrow's UHPLC separations
- The VWD-3100 standard detector operates at up to 100 Hz data rate for optimum support of 62 MPa (9000 psi) UltiMate 3000 Standard systems
- Superior detection of trace analytes with low noise (< –2.0 µAU) and drift (< 100 µAU/h)
- The detector's large linearity range of up to 2.5 AU is ideal for applications with widely varying analyte concentrations
- Up to four absorption channels (VWD-3400RS) and spectral scans support effective method development
- Active temperature control of optics and electronics for data acquisition independent of ambient conditions
- Front panel access for quick and easy lamps and flow cells changes
- Automated qualification monitoring for full regulatory compliance
- Large front panel display for monitoring the detector status even from a distance
- Maximize uptime using predictive performance–based on monitoring the life cycle of detector lamps
- The detector can be upgraded with the Thermo Scientific Dionex pH/Conductivity Monitor (PCM-3000) for accurate and precise pH- and conductivity monitoring
- Unique 45 nL ultra-low dispersion UV monitor for dispersion-free UV detection in LC/MS

UltiMate 3000 VWD-3400 Variable Wavelength Detector.

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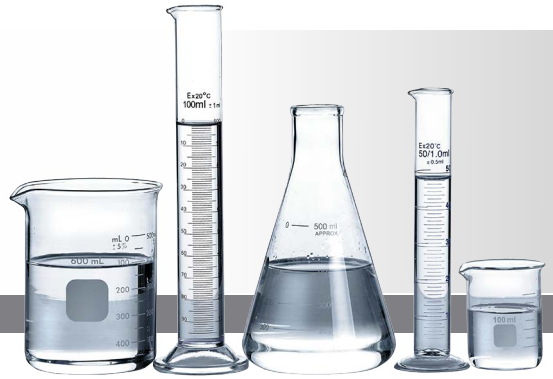

Analytical Technologies

Ion Chromatography

Thermo Scientific Dionex IC systems have led the analytical instrument industry for over 30 years with solutions that represent state-of-the art technological advancements and patented technologies.

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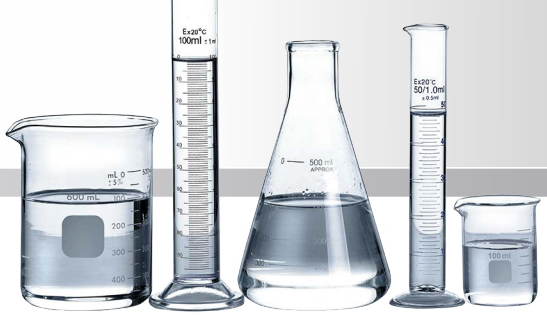

IC and RFIC Systems

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Innovative Ion Chromatography Solutions

Our High-Pressure™ Ion Chromatography (HPIC™) systems include the Thermo Scientific Dionex ICS-5000+ HPIC system, which is optimized for flexibility, modularity, and ease-of-use, combining the highest chromatographic resolution with convenience. In addition, the Thermo Scientific Dionex ICS-4000 Capillary HPIC system is the world's first commercially available dedicated capillary high-pressure Reagent-Free™ (RFIC™) IC system. The Dionex ICS-4000 system is always ready for the next analysis, delivering high-pressure IC on demand.

Reagent-Free IC systems eliminate daily tasks of eluent and regenerant preparation in turn saving time, preventing errors, and increasing convenience. RFIC-EG systems use electrolytic technologies to generate eluent on demand from deionized water, and to suppress the eluent back to pure water to deliver unmatched sensitivity. RFIC-ER systems are designed to use carbonate, carbonate/ bicarbonate, or MSA eluents for isocratic separations.

At the heart of our ion chromatography portfolio is a unique set of column chemistries that provide high selectivities and efficiencies with excellent peak shape and resolution. Thermo Scientific™ Dionex™ IonPac™ chromatography columns address a variety of chromatographic separation modes including ion exchange, ion exclusion, reversed-phase ion pairing, and ion suppression. Our column chemistries are designed to solve specific applications, and we offer a variety of selectivities and capacities for simple and complex samples. Additionally, our Dionex IonPac column line is available in standard bore, microbore and capillary formats for the ultimate application flexibility.

Thermo Scientific Dionex IC instrument family

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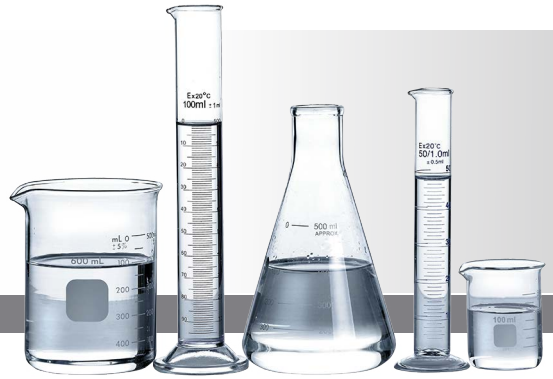

Analytical Technologies

Mass Spectrometry

We provide advanced integrated IC/MS and LC/MS solutions with superior ease-of-use and modest price and space requirements. UltiMate 3000 System Wellness technology and automatic MS calibration allow continuous operation with minimal maintenance. The Dionex ion chromatography family automatically removes mobile phase ions for effort-free transition to MS detection.

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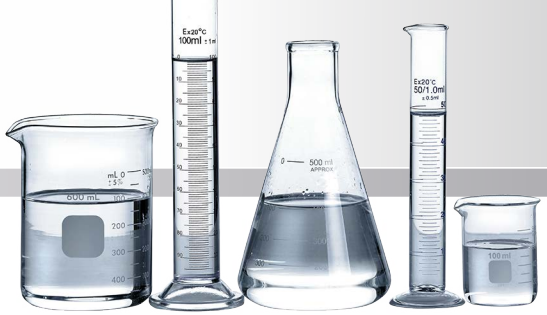

Mass Spectrometry Instruments

Single-Point Control and Automation

We provide advanced integrated IC/MS and LC/MS solutions with superior ease-of-use and modest price and space requirements. UltiMate 3000 System Wellness technology and automatic MS calibration allow continuous operation with minimal maintenance. The Dionex ion chromatography family automatically remove mobile phase ions for effort-free transition to MS detection.

- Thermo Scientific™ MSQ Plus™ mass spectrometer, the smallest and most sensitive single quadrupole on the market for LC and IC
- Self-cleaning ion source for low maintenance operation
- Chromeleon CDS software for single-point method setup, instrument control, and data management compatible with existing IC and LC methods
- The complete system includes the MSQ Plus mass spectrometer, PC data system, electrospray ionization (ESI) and atmospheric pressure chemical ionization (APCI) probe inlets, and vaccum system

Now, you no longer need two software packages to operate your LC/MS system. Chromeleon CDS software provides single-software method setup and instrument control; powerful UV, conductivity, and MS data analysis; and fully integrated reporting.

MSQ Plus Mass Spectrometer

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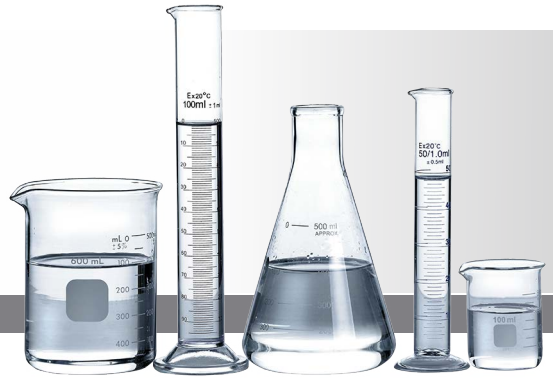

Analytical Technologies

Chromatography Data Systems

Tackle chromatography management challenges with the world's most complete chromatography software. Whether your needs are simple or complex or your scope is a single instrument, a global enterprise, or anything in between – the combination of Chromeleon CDS' scalable architecture and unparalleled ease-of use, makes your job easy and enjoyable with one Chromatography Data System for the entire lab.

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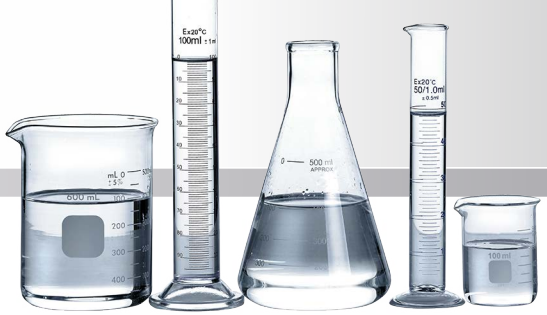

Chromeleon CDS Software

The Fastest Way from Samples to Results

The 7.2 release of Chromeleon Chromatography Data System software is the first CDS that combines separation (GC/IC/LC) and Mass Spectrometry (MS) in an enterprise (client/server) environment. By extending Chromeleon 7.2 CDS beyond chromatography into MS, lab technicians can now streamline their chromatography and MS quantitation workflows with a single software package. MS support in Chromeleon 7.2 CDS is focused on routine and quantitative workflows, which provides access to rich quantitative data processing and automation capabilities — ultimately boosting your overall lab productivity and increasing the quality of your analytical results.

- Enjoy a modern, intuitive user interface designed around the principle of operational simplicity
- Streamline laboratory processes and eliminate errors with eWorkflows™, which enable anyone to perform a complete analysis perfectly with just a few clicks
- Access your instruments, data, and eWorkflows instantly in the Chromeleon Console
- Locate and collate results quickly and easily using powerful built-in database query features
- Interpret multiple chromatograms at a glance using MiniPlots
- Find everything you need to view, analyze, and report data in the Chromatography Studio
- Accelerate analyses and learn more from your data through dynamic, interactive displays
- Deliver customized reports using the built-in Excel® compatible spreadsheet

Excel is a registered trademark of Microsoft Corporation.

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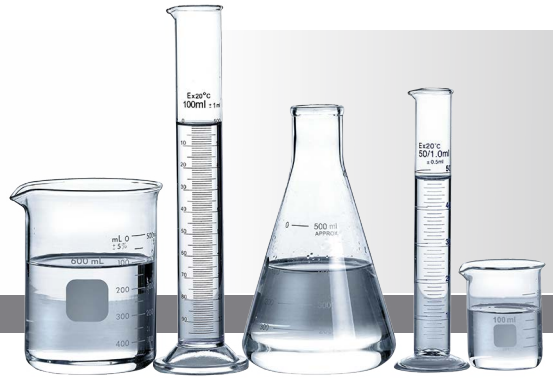

Analytical Technologies

Process Analytical Systems

Thermo Scientific Dionex process analytical systems provide timely results by moving chromatography-based measurements on-line.

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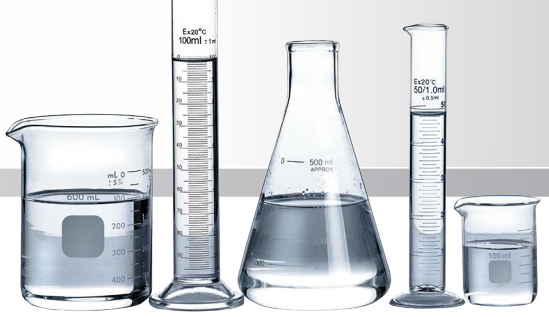

Process Analytical Systems and Software

Improved Process Monitoring with On-line Chromatography IC and LC Systems

Information from the Thermo Scientific Dionex Integral process analyzer can help reduce process variability, improve efficiency, and reduce downtime. These systems provide comprehensive, precise, accurate information faster than is possible with laboratory-based results. From the lab to the factory floor, your plant's performance will benefit from the information provided by on-line LC.

- Characterize your samples completely with multicomponent analysis
- Reduce sample collection time and resources with automated multipoint sampling
- Improve your process control with more timely results
- See more analytes with unique detection capabilities
- The Thermo Scientific Integral Migration Path approach lets you choose the systems that best meets your needs

Integral process analyzer

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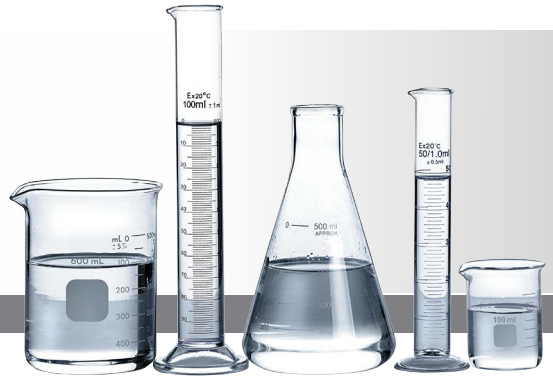

Analytical Technologies

Automated Sample Preparation

Solvent extractions that normally require labor-intensive steps are automated or performed in minutes, with reduced solvent consumption and reduced sample handling using the Thermo Scientific™ Dionex™ ASE™ Accelerated Solvent Extractor system or Thermo Scientific™ Dionex™ AutoTrace™ 280 Solid-Phase Extraction instrument.

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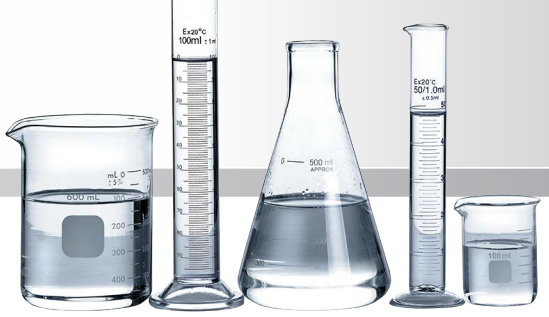

Accelerated Solvent Extractor System

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Complete Extractions in Less Time Using Less Solvent

Thermo Scientific Dionex ASE systems extract of solid and semisolid samples using common solvents at elevated temperature and pressure. The Dionex ASE 150 and 350 systems feature pH-hardened pathways with Dionium™ components to support extraction of acidic or alkaline matrices, and combine pretreatment, solvent extraction, and cleanup into one step. Dionium is zirconium that has undergone a proprietary hardening process that makes it inert to chemical attack by acids and bases at elevated temperatures.

Dionex ASE systems are dramatically faster than Soxhlet, sonication, and other extraction methods, and require significantly less solvent and labor. Accelerated solvent extraction methods are accepted and established in the environmental, pharmaceutical, foods, polymers and consumer product industries. Accelerated solvent extraction methods are accepted and used by government agencies worldwide.

Dionex ASE 150/350 and Dionex AutoTrace 280 SPE instruments

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Adulteration and Authentication

Part 1
Targeted Methods

The measurement of identified analytes as indicators of possible adulteration and to verify product authenticity.

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Adulteration and Authentication

Targeted Approaches

Coffee Adulteration

The less costly Robusta varietal is commonly used in decaffeinated coffee and as an adulterant in the preferred and more costly Arabica varietal.

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Coffee Adulteration

Organic acid profiles of coffee are used to determine Robusta content in Arabica coffee. Additionally, organic acids are of interest because they are primarily responsible for the coffee's acidity. The chromatograms shown here compare the differences in decaffeinated and caffeinated brewed coffee samples with the characteristic higher quinate (quinic acid) concentration present in the caffeinated coffee. Lower quinate and lower malate concentrations than normal would be indicative of adulteration by a Robusta varietal coffee.

Column:	Thermo Scientific™ Dionex™ IonSwift™ MAX-100G, MAX-100, 0.25 × 250 mm
Flow:	12 µL/min
Column Temp.:	30 °C
Injection Volume:	0.4 µL
Eluent Source:	Dionex EGC-KOH capillary cartridge
Gradient:	0.1 mM KOH (-10–4 min), 0.1–2 mM (4–6 min), 2–15 mM (6–12 min), 15–35 mM (12–16 min), 65 mM (17–30 min)
Detection:	Suppressed conductivity, Dionex ACES 300 suppressor, recycle mode
Sample Prep.:	1:50 dilution

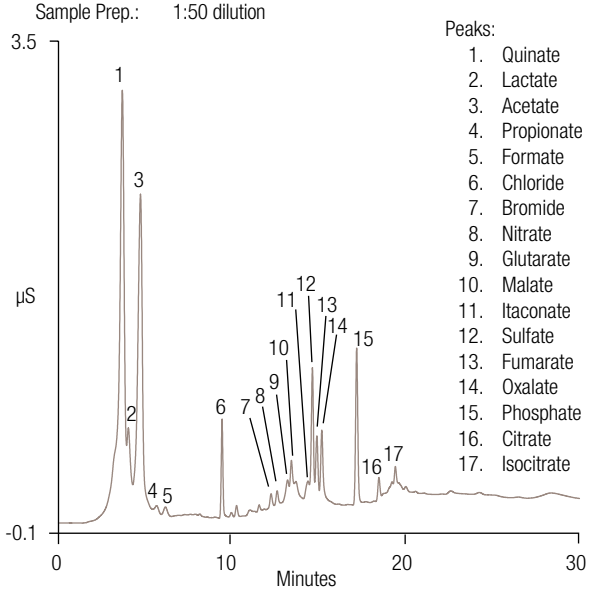

Figure 7-1. Determination of anions and organic acids in a brewed decaffeinated coffee sample more typical of the lower cost Robusta coffee.

Column:	Dionex IonSwift MAX-100G, MAX-100, 0.25 × 250 mm
Flow:	12 µL/min
Column Temp.:	30 °C
Injecton Volume:	0.4 µL
Eluent Source:	Dionex EGC-KOH capillary cartridge
Gradient:	0.1 mM KOH (-10–4 min), 0.1–2 mM (4–6 min), 2–15 mM (6–12 min), 15–35 mM (12–16 min), 65 mM (17–30 min)
Detection:	Suppressed conductivity, Dionex ACES 300 suppressor, recycle mode
Sample Prep.:	1:50 dilution

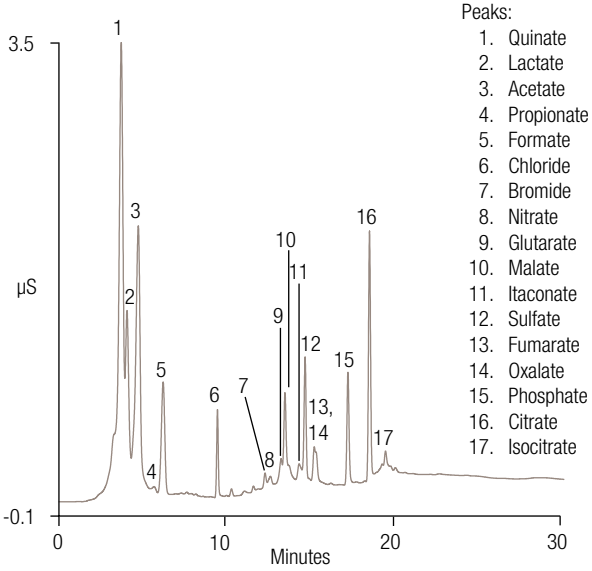

Figure 7-2. Determination of anions and organic acids in a brewed caffeinated coffee sample typical of the more costly Arabica coffee.

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Controlled Substances

The identification of controlled substances in food samples is challenging due to the wide range of drugs available and the complex nature and diversity of the sample being analyzed.

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Controlled Substances: Cannabinoids

Marijuana is the most common illegal drug in the United States, and each year U.S. law enforcement agencies seize more than two million pounds of marijuana in various forms. Seized evidence submitted to forensic laboratories is screened for marijuana by microscopic inspection and simple chemical tests such as the Duquenois-Levine test. Presumptive positive results are confirmed by using gas chromatography-mass spectrometry (GC/MS) to positively identify cannabinoids including Δ^9-tetrahydrocannabinol (THC, the main psychoactive component), cannabinol (the main degradation product of THC) and cannabidiol.

This traditional approach works fairly well for leaf marijuana, hashish, hash oil, and residue collected from smoking paraphernalia.

An alternative method to positively identify marijuana cannabinoids in complex food matrices is to use UHPLC/MS. UHPLC/MS offers a threefold benefit compared to GC/MS; simpler sample preparation, no derivatization, and less instrument clean up time. Application Note 433 demonstrates how a working forensic laboratory uses UHPLC/MS to analyze baked goods for three cannabinoids of forensic importance.

Column:	hermo Scientific™ Hypersil GOLD™ PFP (perfluorinated phenyl) 1.9 µm,100 × 2.1 mm			
Flow:	1 mL/min			
Column Temp:	45 °C			
Injection Volume:	2 µL partial loop injection, 25 µL loop size			
Mobile Phase:	A: Water with 0.06 % acetic acid			
	B: Acetonitrile (MeCN) with 0.06% acetic acid			
	C: Methanol with 0.06% acetic acid			
Gradient:	T (min)	A%	B%	C%
	0.00	95.0	0.0	5.0
	1.00	60.0	32.5	7.5
	2.00	50.0	40.0	10.0
	5.00	45.0	45.0	10.0
	6.00	25.0	60.0	15.0
	6.50	5.0	0.0	95.0
	7.50	5.0	0.0	95.0
	7.51	95.0	0.0	5.0
	8.00	95.0	0.0	5.0
Syringe Speed:	8 µL/sec			
Flush Speed: 1	100 µL/sec			
Flush Volume:	400 µL			
Wash Volume:	100 µL			
Flush/Wash Source:	Bottle with methanol			

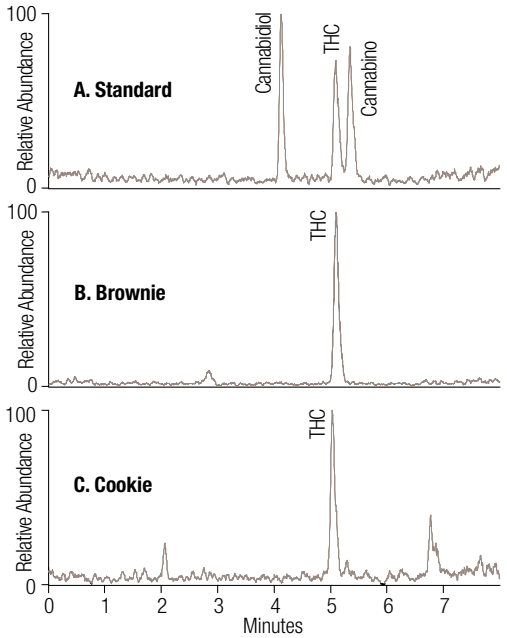

Figure 7-3. Extracted ion chromatograms (*m/z* 310.5–311.5 and 314.5–315.5) of cannabinoid standards: (A) extracts from brownie, (B) extracts from cookie, and a standard (C) by UHPLC/MS.

Download Application Note 433: Identification of Cannabinoids in Baked Goods by UHPLC/MS

Controlled Substances: Cannabinoids

MS analysis was carried out on a MSQ Plus single quadrupole LC/MS detector with Thermo Scientific™ Xcalibur™ 2.05 software.

Mass Spectrometer Conditions

Ionization:	Electrospray (ESI)
Polarity:	Positive
Probe Temperature:	500 °C
Cone Voltage:	90 V
Scan Mode:	Full scan with mass range of 50–500 *m/z*
ESI Voltage:	3.5 kV
Scan Time:	0.2 s

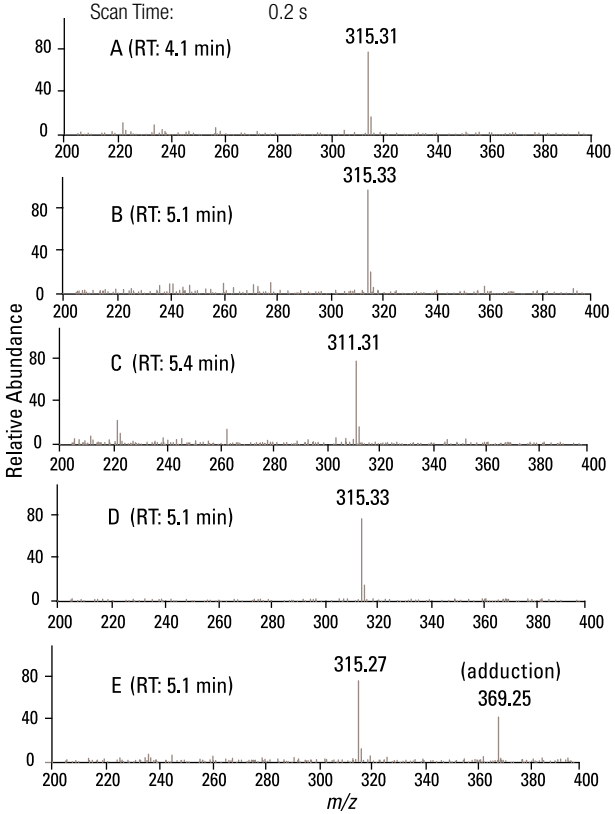

Figure 7-4. MS spectra of cannabinoid standards: cannabidiol (A), THC (B), cannabinol (C), eluted at 4.1 min, 5.1 min, and 5.4 min respectively, and extracts from brownie (D) and cookie (E), eluted at 5.1 min.

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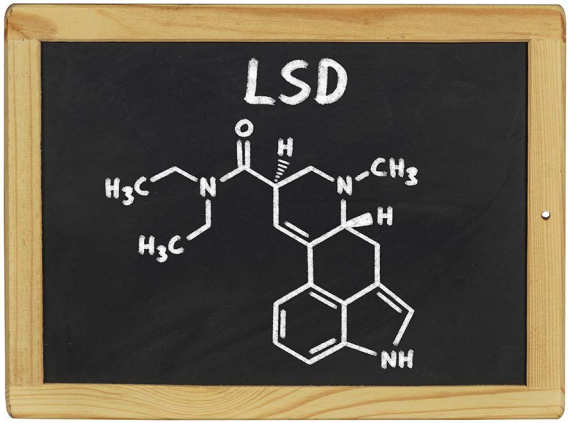
Controlled Substances: LSD

Lysergic acid diethylamide (LSD) is a controlled substance in forensic chemistry that is notorious for being difficult to identify. Its myriad evidentiary forms include paper tabs, eye drops, sugar cubes and small sugary candies such as sweet tarts, valentine hearts, or mints. Because it is such a potent hallucinogen, typical street doses require only 40 to 120 µg of LSD. The small personal-use amounts seized by state and local law enforcement often lack sufficient drug to allow both forensic analysis by traditional means and archiving of some of the evidence for follow-up testing. Most forensic laboratories confirm the presence of LSD by using gas chromatography with mass spectrometry (GC/MS).

An alternative method to positively identify LSD in complex food matrices is to use ultra high performance liquid chromatography with mass spectrometric detection (UHPLC/MS). UHPLC/MS offers a threefold benefit compared to GC/MS; simpler sample preparation, no derivatization, and less time wasted baking out or cleaning the instrument. Application Note 432 demonstrates how a working forensic laboratory uses UHPLC/MS to analyze sugar candies for LSD.



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Download Application Note 432: Identification of Lysergic Acid Diethylamide (LSD) in Candy by UHPLC/MS

Controlled Substances: LSD

Column:	Hypersil GOLD PFP (perfluorinated phenyl) 1.9 µm,100 x 2.1 mm	Syringe Speed:	8 µL/sec
Flow:	1 mL/min	Flush Speed:	100 µL/sec
Column Temp.:	45 °C	Flush Volume:	400 µL
Injection Volume:	2 µL partial loop injection, 25 µL loop size	Wash Volume:	100 µL
Mobile Phase:	A: Water with 0.06 % acetic acid	Flush/Wash Source:	Bottle with methanol
	B: Acetonitrile (MeCN) with 0.06% acetic acid		
	C: Methanol with 0.06% acetic acid		

Gradient:

T (min)	A%	B%	C%
0.00	95.0	0.0	5.0
1.00	95.0	0.0	5.0
1.50	90.0	5.0	5.0
2.70	70.0	10.0	20.0
3.00	5.0	15.0	80.0
7.00	5.0	0.0	95.0
7.10	95.0	0.0	5.0
8.00	95.0	0.0	5.0

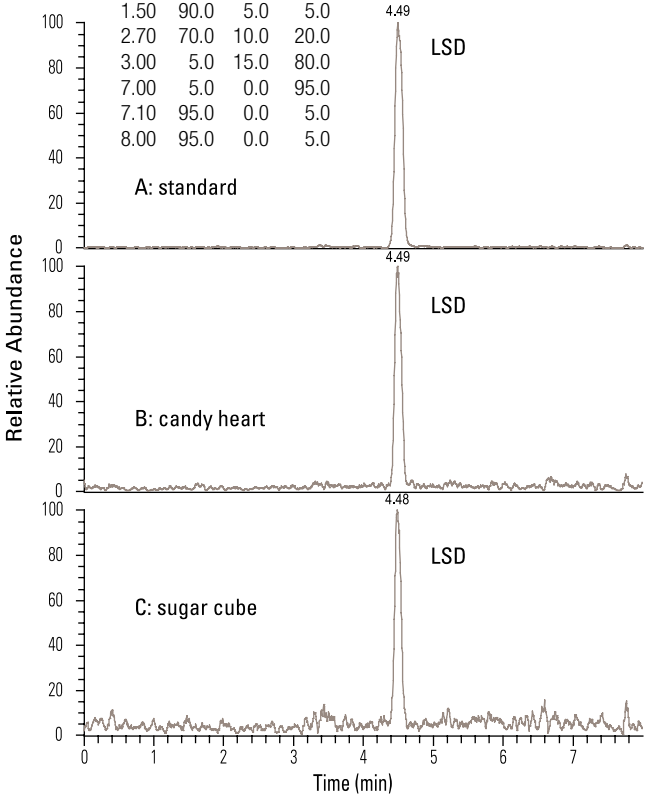


Figure 7-5. Extracted ion chromatograms at $m/z = 324 \pm 0.5$ amu obtained by UHPLC/MS on a Hypersil GOLD PFP column: (A) LSD standard; (B) methanol extract of LSD-doped candy heart; (C) methanol extract of LSD-doped sugar cube.

Mass Spectrometer Conditions

Ionization:	Electrospray (ESI)
Polarity:	Positive
Probe Temperature:	500 °C
Cone Voltage:	90 V
Scan Mode:	Full scan with mass range of m/z = 125–425 amu
ESI Voltage:	3.5 kV
Scan Time:	0.2 s

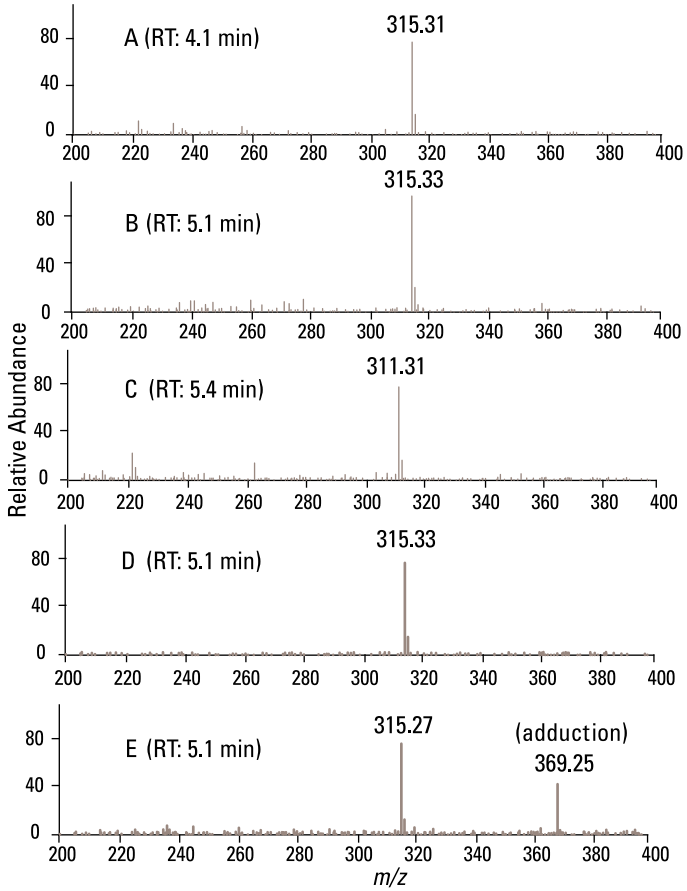


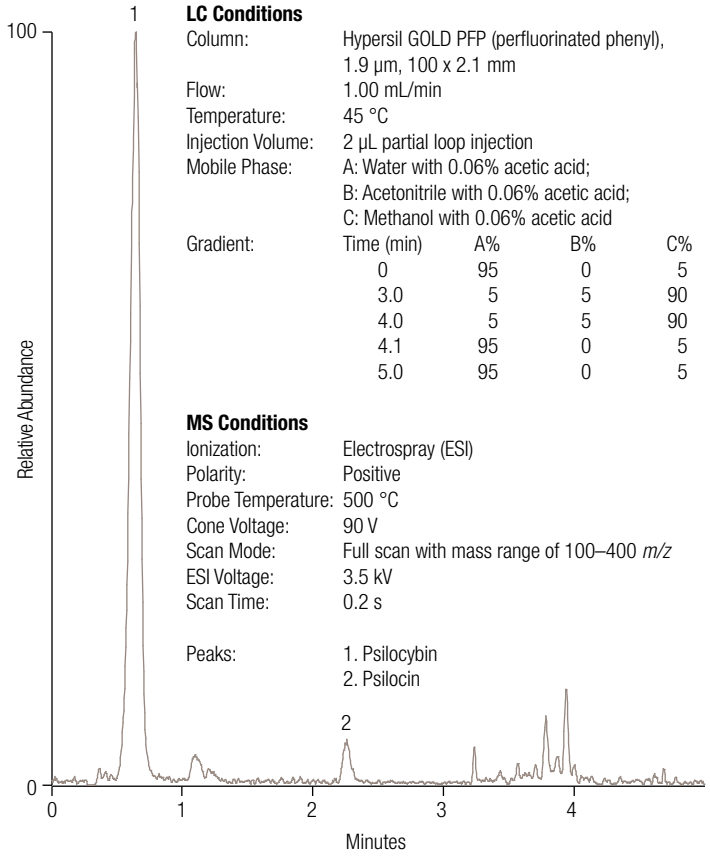
Figure 7-6. MS spectra of LSD obtained by UHPLC/MS on a Hypersil GOLD PFP column: (A) LSD standard; (B) methanol extract of LSD-doped candy heart; (C) methanol extract of LSD-doped sugar cube.

Controlled Substances: Pyschotropics

The identification of controlled substances in food matrices is a challenge for forensic laboratories. Classical techniques, such as color tests and thin-layer chromatography, do not provide molecular structural information and cannot be used as a principal means of identification. Infrared spectroscopy (IR) and GC/MS can identify controlled substances, but suffer from several shortcomings. To ameliorate these issues, extensive wet chemistry preparation methods have been developed; unfortunately, they are time-consuming and often require greater amounts of the controlled substances than are present in the evidence.

Given the increasing emphasis on instrumental methods of analysis and the limitations of the traditional instruments in the field of forensics, another solution is necessary. LC/MS holds several advantages over the traditional methods of analysis. For example, psilocybin does not decompose at the lower temperatures used in HPLC. The low concentrations of psilocybin and psilocin are not an issue due to increased sensitivity of this technique. Many matrix components that interfere in GC/MS methods do not interfere in HPLC methods because of greater differences in analyte solubility as compared to analyte volatility.

Application Note 428 describes a method that can be used to separate and confirm psilocybin and psilocin in mushroom and chocolate samples using UHPLC/MS.



LC Conditions

Column:	Hypersil GOLD PFP (perfluorinated phenyl), 1.9 µm, 100 x 2.1 mm
Flow:	1.00 mL/min
Temperature:	45 °C
Injection Volume:	2 µL partial loop injection
Mobile Phase:	A: Water with 0.06% acetic acid; B: Acetonitrile with 0.06% acetic acid; C: Methanol with 0.06% acetic acid

Gradient:

Time (min)	A%	B%	C%
0	95	0	5
3.0	5	5	90
4.0	5	5	90
4.1	95	0	5
5.0	95	0	5

MS Conditions

Ionization:	Electrospray (ESI)
Polarity:	Positive
Probe Temperature:	500 °C
Cone Voltage:	90 V
Scan Mode:	Full scan with mass range of 100–400 *m/z*
ESI Voltage:	3.5 kV
Scan Time:	0.2 s
Peaks:	1. Psilocybin 2. Psilocin

Figure 7-7. Methanol extract of chocolates containing psilocybin and psilocin.

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Download Application Note 428: Identification of Psychotropic Substances in Mushrooms and Chocolate by UHPLC/MS

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Adulteration and Authentication

Targeted Approaches

Fruit Juice Adulteration

Fruit juice adulteration presents an economic and regulatory problem. The United States orange juice industry estimates that orange juice sales gross more than one billion dollars annually. The most common forms of adulteration include simple dilution and blending of inexpensive and synthetically produced juices into the more expensive ones.

Learn more about US Fruit Juice Adulteration Regulations

Learn more about the History of Fruit and Vegetable Juice US Regulations

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Fruit Juice Adulteration: Organic Acids

The concentration of organic acids can be used to assess whether an expensive juice has been illegally adulterated with a cheaper juice. Because organic acid profiles are distinct to each type of fruit juice, evidence of tampering can be evaluated by comparing the known juice fingerprint to that of the suspected adulterated juice.

As shown in these examples, the major organic acids in grape juice are malate, tartrate, and citrate, whereas in orange juice the primary analyte is citric acid. Both lactic and acetic acids are typically low in these juice samples – elevated levels of lactic and acetic acids may be caused by microbiological spoilage, so it is important to monitor the concentrations of these organic acids as a measure of product quality.

Malate is the major constituent of apple juice, whereas in cranberry juice cocktail, there are high concentrations of succinate and quinate, which provide cranberry's tart taste.

The presence of galacturonate in cranberry juice cocktail and grape juice can be attributed to the degradation of pectins in the skins of fruit. Freshly squeezed juices generally show lower levels of galacturonate.

Peaks:

1. Quinate	55	ppm	9. Malate	116.0	
2. Fluoride	1.3		10. Malonate/Tartrate	190.0	
3. Lactate	46.2		11. Maleate	4.0	
4. Galacturonate	60		12. Sulfate	22.1	
5. Chloride	1.2		13. Oxalate	19.4	
6. Nitrate	0.6		14. Phosphate	27.0	
7. Glutarate	0.7		15. Citrate	80.0	
8. Succinate	1.2		16. Isocitrate	1.8	

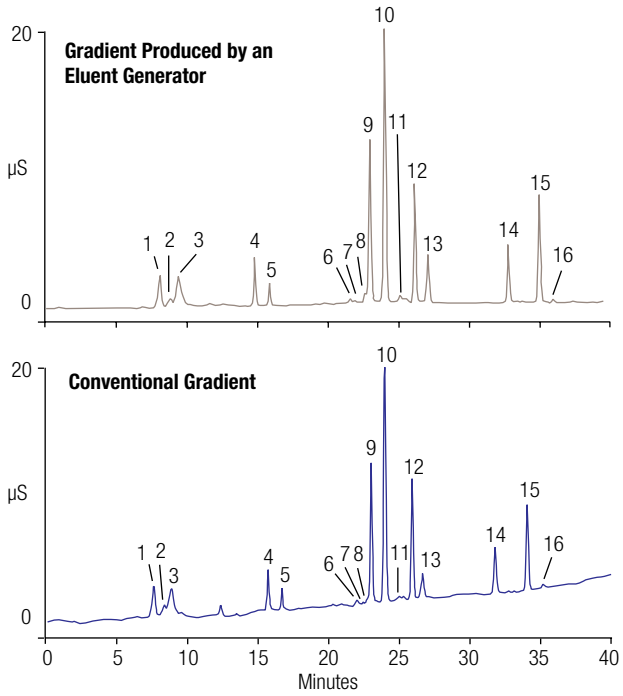

Figure 7-8. Determination of anions and organic acids in grape juice.

Download Application Note 143: Determination of Organic Acids in Fruit Juices

Fruit Juice Adulteration: Organic Acids

Peaks:					
1. Quinate	4.0	ppm	9. Succinate/Malate	161.0
2. Lactate	9.0		10. Malonate	3.0
3. Acetate	0.5		11. Sulfate	4.4
4. Propionate	1.9		12. Oxalate	13.8
5. Formate	3.0		13. Phosphate	23.0
6. Chloride	2.0		14. Citrate	400
7. Nitrate	0.1		15. Isocitrate	4.0
8. Glutarate	13.0			

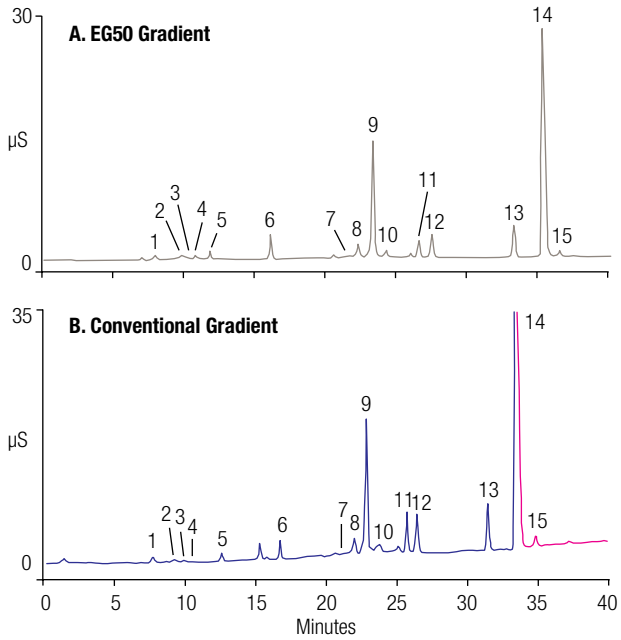

Figure 7-9. Determination of anions and organic acids in orange juice.

Peaks:				
1. Quinate	34.5	mg/L	10. Malate	250.1
2. Lactate	14.8		11. Malonate/Tartrate	0.8
3. Glycolate	0.6		12. Maleate	1.6
4. Formate	0.5		13. Sulfate	1.7
5. Pyruvate	1.2		14. Oxalate	0.5
6. Galacturonate	1.5		15. Phosphate	12.6
7. Chloride	0.4		16. Citrate	6.6
8. Nitrate	0.8		17. Isocitrate	0.4
9. Succinate	9.0		18. *Cis*-aconitate	0.4

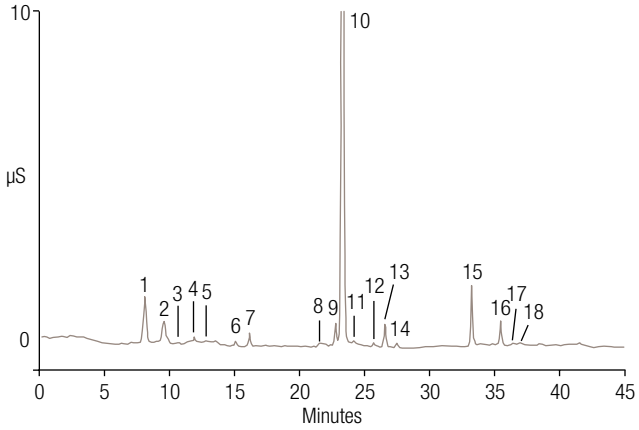

Figure 7-10. Determination of anions and organic acids in apple juice.

Fruit Juice Adulteration: Organic Acids

Peaks:					
1.	Quinate	210	mg/L	11. Succinate	257
2.	Fluoride	<0.1		12. Unknown	-
3.	Lactate/Acetate	10		13. Sulfate	10.3
4.	Glycolate	2.6		14. Oxalate	14.8
5.	Formate	3.7		15. Phosphate	1.8
6.	Pyruvate	2.1		16. Unknown	-
7.	Unknown	-		17. Citrate	163
8.	Galacturonate	16.9		18. Isocitrate	1.0
9.	Chloride	2.3		19. *Trans*-aconitate	2.7
10.	Nitrate	<0.1		20. Unknown	-

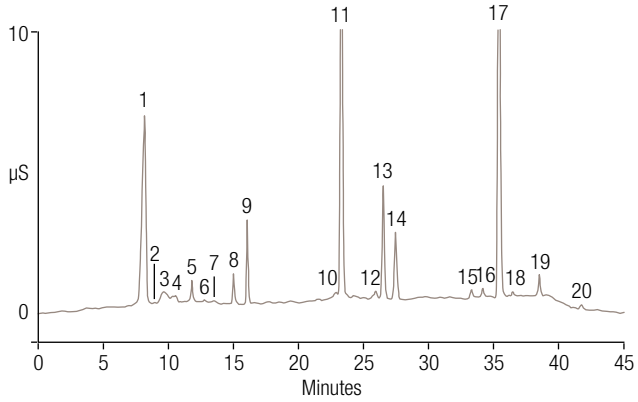

Figure 7-11. Determination of anions and organic acids in cranberry juice cocktail.

Did You Know?

There are three types of adulteration:

- Replacement (complete or partial replacement of a food ingredient with a less expensive substitute). Examples include addition of melamine to milk, and water/citric acid to lemon juice. This form of adulteration also includes false declaration of geographical, species, botanical or varietal origin.
- Addition (addition of small amounts of non-authentic substance to mask inferior quality ingredient). E.g., addition of Sudan red dyes to enhance the color of poor quality paprika.
- Removal (Removal or intentional omission of an authentic and valuable constituent). An example is filtering of poor quality honey to remove pollen or other residue in order to make it difficult to trace and identify the source of the honey.

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Fruit Juice Adulteration: Oligosaccharides

Oligosaccharides are routinely determined in food and beverage products for a variety of purposes, including quality control, verifying food-labeling claims, establishing product authenticity, and monitoring fermentation processes. Oligosaccharide profiles obtained using HPAE-PAD with the Thermo Scientific™ Dionex™ CarboPac™ PA100 column can be used to establish the "fingerprint" of food samples. Suspect samples can be analyzed and compared to the known profiles. This technique is useful in detecting adulteration and in quality control. For example, oligosaccharide profiles are used to detect adulteration of natural fruit juices, establish the geographic origin of molasses, and analyze for polysaccharides in hydrolyzed glucose syrup.

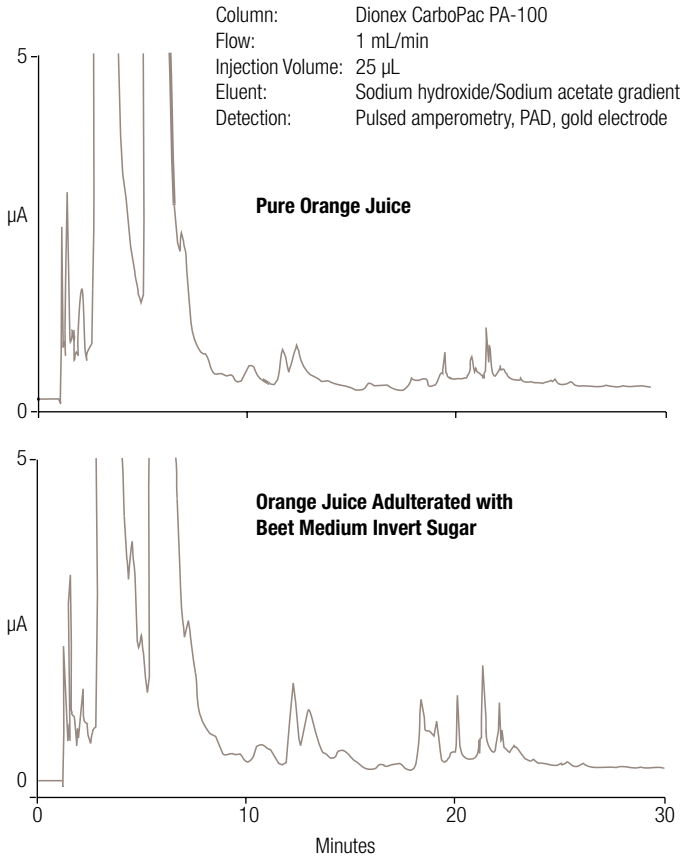

Figure 7-12. Oligosaccharide profiles of pure and adulterated orange juice. Oligosaccharide composition profiles can be used to detect the adulteration of natural fruit juices by inexpensive sweeteners such as beet sugar.

Fruit Juice Adulteration: Profiling

One adulterant currently in use is partially inverted sucrose, wherein about one-half of the sucrose has been hydrolyzed to glucose and fructose. This ratio of approximately 1:2 (glucose: fructose: sucrose) closely matches the ratio found in orange juice. When cane sugar is the of inverted sucrose, Stable Isotope Ratlo Analysis (SIRA) can be used to identify adulterated juices because the ratio of ^{13}C to ^{12}C is different for sugars in orange juice and cane sugar. Beets, on the other hand, produce sugar via a metabolic pathway different from cane and similar to many fruits, so that the ratio of ^{13}C to ^{12}C is about the same for sugars in orange juice and beet sugar. This fact renders SIRA inadequate for detecting adulteration by beet sugar.

Recently, investigators using high performance anion exchange chromatography with pulsed amperometric detection (HPAE-PAD) have discovered several components in beet medium invert sugar (BMIS) that are not present in orange juice – such as late eluting components as well as raffinose as markers of orange juice adulteration.

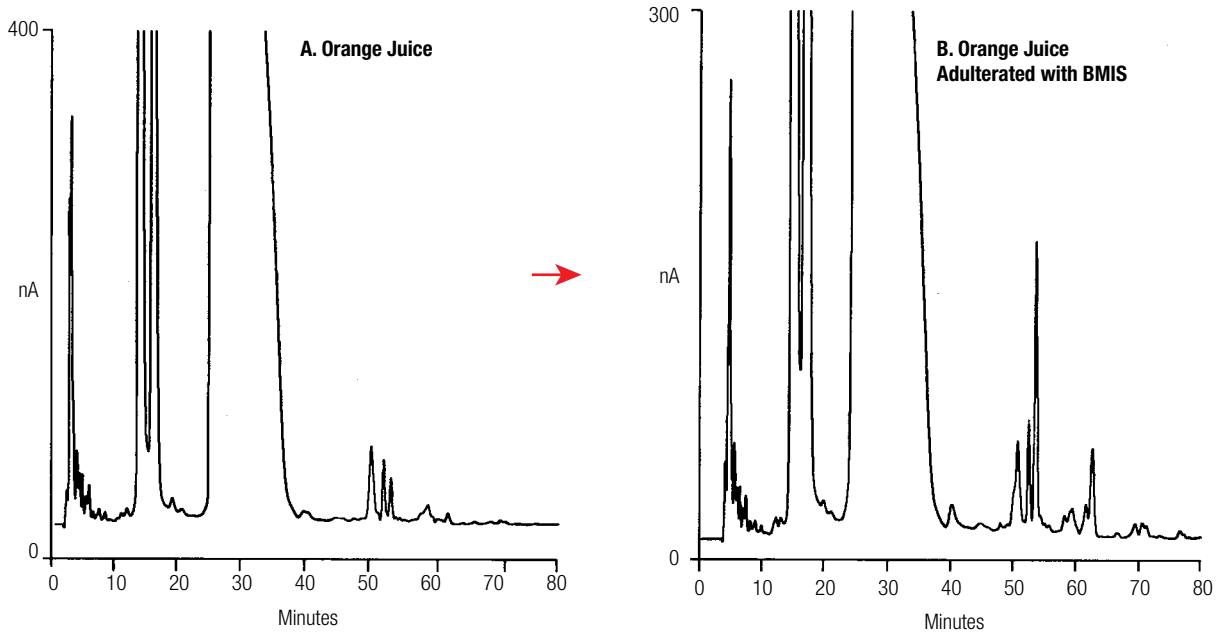

Figure 7-13. Chromatograms showing (A) unadulterated orange juice and (B) orange juice adulterated with medium invert sugar. Note the late-eluting finger-print between 50 and 60 minutes.

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Fruit Juice Adulteration: Profiling

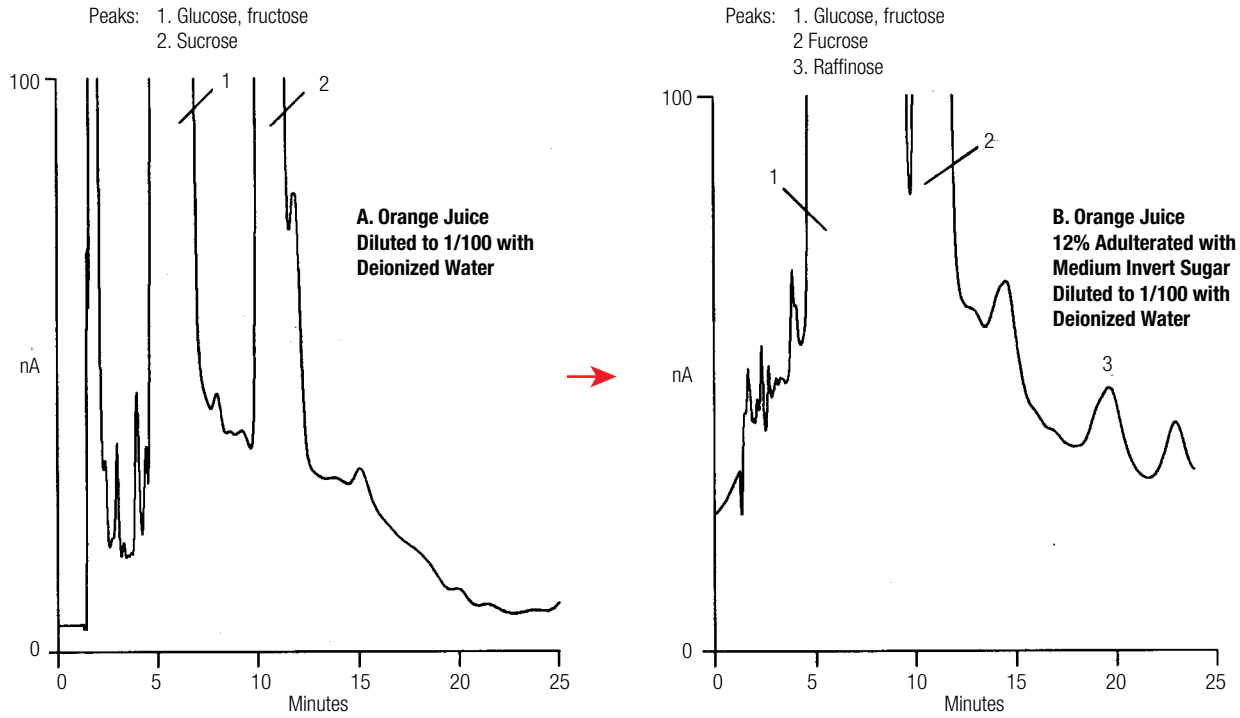

Figure 7-14. Chromatograms showing (A) unadulterated orange juice (note the lack of any peaks eluting at 20 minutes) and (B) orange juice adulterated to 12% with medium invert beet sugar Adulteration can be detected by the presence of raffinose at 20 minutes.

Fruit Juice Adulteration: Profiling

Pomegranate is extensively cultivated worldwide and has become a high-value crop for juice production. The retail market now contains numerous pomegranate-related products such as juices, smoothies, flavored waters, and sports and energy drinks. Due to the high demand for pomegranates outstripping the supply, adulteration of pomegranate juice (PJ) has become widespread. Manufacturers have attempted to extend the limited supply of PJ by blending with filler ingredients such as cane sugar, corn syrup sweeteners, and lower-quality juices containing sorbitol, malic acid, and sucrose (e.g., grape, apple, and blackberry). To establish an authentication criterion, an International Multidimensional Authenticity Specifications algorithm was developed based on the analysis of commercial juice samples from 23 manufacturers in the United States, Iran, Turkey, Azerbaijan, Syria, India, and China. There is universal agreement that the anthocyanin profile in PJ consists of a constant group of six anthocyanins, regardless of the origin. However, the anthocyanin concentrations can vary depending on the geographic source of the PJ. The anthocyanin profile is one of several chemical analyses that are required to determine the authenticity of PJ. Additional chemical profiling methods include measuring other polyphenols (i.e., ellagitannins), monosaccharides (e.g., fructose and glucose), organic acids, amino acids, and potassium in PJ samples. Determinations of monosaccharides, organic acids, and punicalagins in fruit juices have been previously described in AN 82, 143, and CAN 106, respectively

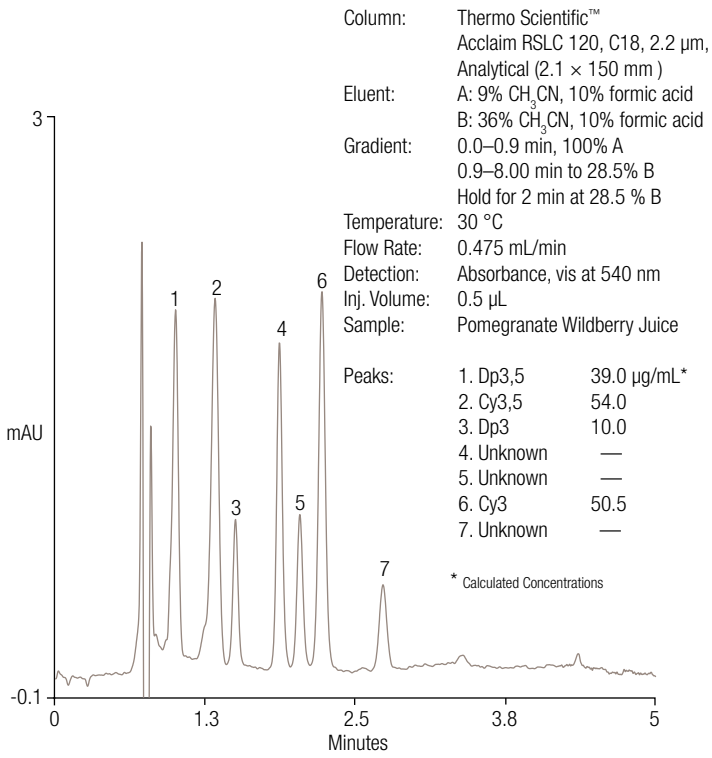

Figure 7-15. Typical profile of anthocyanins in pomegranate wildberry juice.

Download Application Note 82: Analysis of Fruit Juice Adulterated with Medium Invert Sugar from Beets

Download Application Note 143: Determination of Organic Acids in Fruit Juices

Download Customer Application Note 106: Determination of the Punicalagins Found in Pomegranate by High Performance Liquid Chromatography

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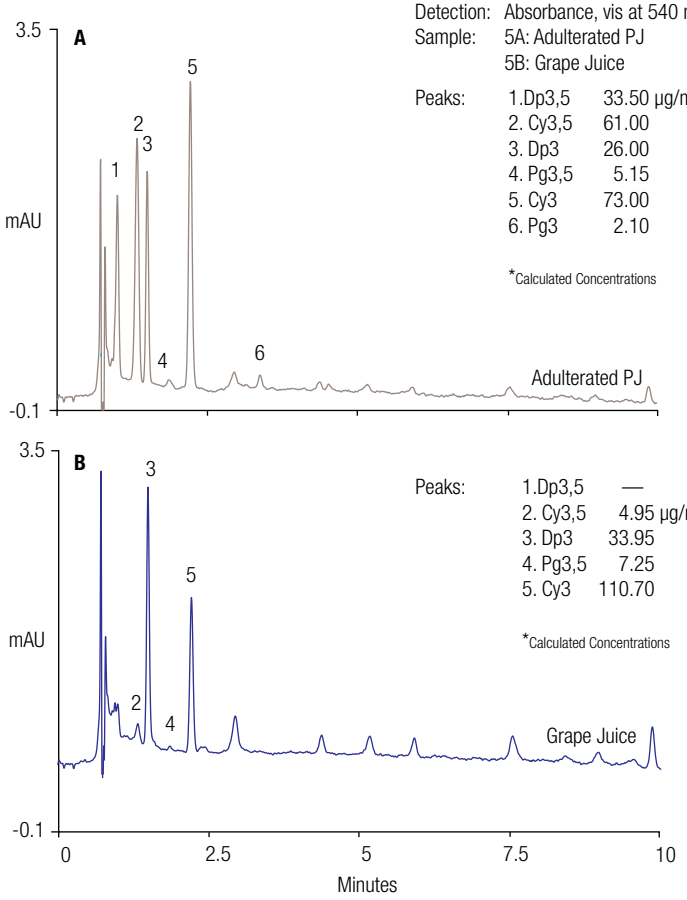
Fruit Juice Adulteration: Profiling

Grape juice contains four of the six anthocyanins present in PJ but at much lower concentrations. Simulated adulterated PJ was prepared by combining PJ and grape juice, then diluting 1:5 in mobile phase A prior to analysis. The adulterated juice shows all of the signature anthocyanins and several other late-eluting peaks not characteristic of PJ. The anthocyanin content of the adulterated juice is also lower than that of PJ, as expected.

Column: Acclaim RSLC 120, C18, 2.2 µm, Analytical (2.1 × 150 mm)
Flow: 0.475 mL/min
Temperature: 30 °C
Injection Volume: 0.5 µL

Eluent: A: 9% CH_3CN, 10% formic acid
B: 36% CH_3CN, 10% formic acid
Gradient: 0.0–0.9 min, 100% A
0.9–8.00 min to 28.5% B
Hold for 2 min at 28.5 % B
Detection: Absorbance, vis at 540 nm
Sample: 5A: Adulterated PJ
5B: Grape Juice



Peaks: 5A: Adulterated PJ

1.	Dp3,5	33.50 µg/mL*
2.	Cy3,5	61.00
3.	Dp3	26.00
4.	Pg3,5	5.15
5.	Cy3	73.00
6.	Pg3	2.10

*Calculated Concentrations

Peaks: 5B: Grape Juice

1.	Dp3,5	—
2.	Cy3,5	4.95 µg/mL*
3.	Dp3	33.95
4.	Pg3,5	7.25
5.	Cy3	110.70

*Calculated Concentrations

Figure 7-16. Separation of anthocyanins in simulated adulterated pomegranate juice (5A) overlayed with a separation of anthocyanins in grape juice (5B).

Fruit Juice Adulteration: Profiling

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Anthocyanins are water-soluble plant pigments widely present in fruits, vegetables, and flowers. Anthocyanins have gained considerable interest in the scientific community and consumer market due to their anti-inflammatory action and strong antioxidant and radiation-protection properties. Bilberries are known to have a high anthocyanin content and are, therefore, one of the most expensive botanical ingredients in the health food industry. The high price of the extract makes bilberries more susceptible to adulteration.

Analytical characterization and quantification methods for anthocyanins specific to bilberries are therefore required. This study demonstrates a rapid separation liquid chromatography-mass spectrometry (RSLC-MS) method using the MSQ Plus mass spectrometer for the determination of anthocyanins in bilberry extractand pomegranate juice based on previously developed LC separations.

Chromatographic Conditions

System:	UltiMate 3000 RSLC System
Column:	Acclaim RSLC 120 C18, 2.1 × 100 mm, 2.2 µm
Flow:	0.5 mL/min
Temperature:	40 °C
Injection Volume:	5 µL
Mobile Phase:	A) CH_3CN, B) DI water, C) 20% formic acid
Gradient:	C held constant at 10%, A from 0% to 8% from 11 to 42 min, held for 13 min, return to 0% in 5 min
Sample:	NIST Bilberry Extract Standard Reference Material

Mass Spectrometric Conditions

System:	MSQ Plus single quadrupole mass spectrometer
Ionization interface:	ESI
Probe Temperature:	500 °C
Needle Voltage:	2000 V
Nebulizer Gas:	Nitrogen at 80 psi
Detection Mode:	SIM Refer to chromatogram for SIM Acquisitions

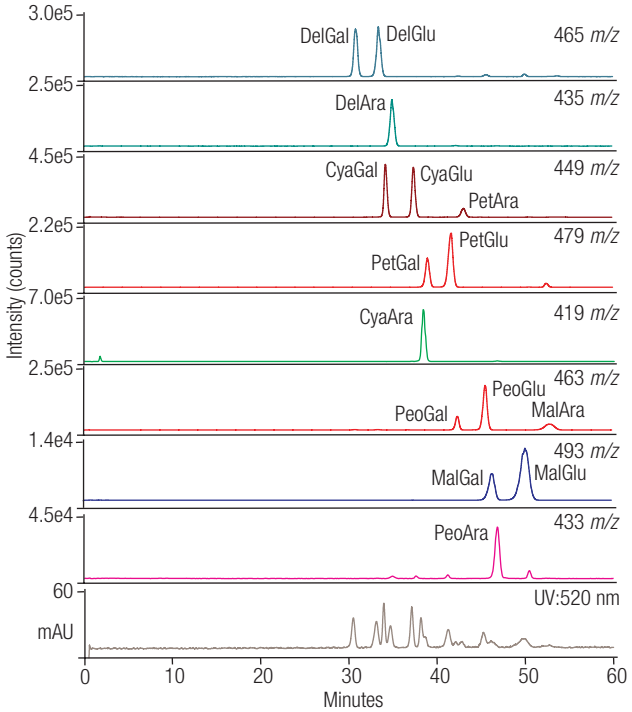

Figure 7-17. Determination of 15 anthocyanins in bilberry extract by LC-MS.

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Fruit Juice Adulteration: Profiling

The primary mission of the Office of Dietary Supplements (ODS) at the National Institutes of Health (NIH) is to promote the quality, safety, and efficacy of dietary supplements. To accomplish this mission, authentic reference materials that closely match the matrix components of the dietary supplements are needed.

Standard Reference Materials (SRMs) for vaccinium (e.g., cranberries, blueberries, and bilberries) are being developed at the National Institute of Standards and Technologies (NIST) in collaboration with the NIH-ODS to evaluate these types of dietary supplements. Several SRMs with certified values for organic acids are currently available from NIST to aid dietary supplement and juice manufacturers in their analytical method development and QA/QC operations. Further work to certify anthocyanins and anthocyanidins is under way.

The popularity of cranberries is primarily associated with their reported ability to combat urinary tract infection and their high organic acid, anthocyanin, and polyphenol content. The primary health benefits of cranberries my be derived from their antioxidant properties, and their anthocyanin profiles are important in determining good quality and authenticity of the products. Cranberry extracts are prone to adulteration with lower value products in order to offer consumers competitive prices, therefore making it important to ensure the quality, safety and efficacy of these products. Organic acids present in cranberries impart flavor, and their specific ratios can be used to detect adulteration.

Organic acids in cranberry extract were measured using suppressed conductivity detection. Ratios of quinate, malate, and citrate can be used to identify potential adulteration.

Anthocyanin content were measured by 14 participating laboratories and results were submitted to the NIST. An overall consensus of the average reported concentration was observed, indicating validity of the data and the method.

Column:	Dionex IonPac AG11-HC, AS11-HC, 2 mm
Flow:	0.38 mL/min
Temperature:	30 °C
Injection Volume:	5 µL
Eluent:	1 mM KOH from -8 to 8 min, 1—30 mM from 8 to 20 min, 30—60 mM from 20 to 30 min, 60 mM from 30—45 min
Eluent Source:	Dionex EGC II KOH with Dionex CR-ATC
Detection:	Dionex ASRS 300 suppressor. 2mm, recycle mode, 57 mA
Sample Prep:	0.1 g cranberry extract/160 mL DI water (centrifugation at 25 °C for 15 min for each 40 mL aliquot)
Peaks:	1. Quinate 11.7 mg/g 2. Malate 5.5 3. Citrate 21.7

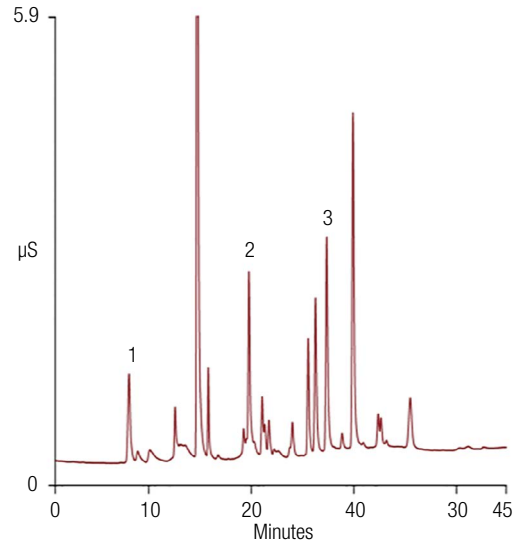

Figure 7-18 Separation of organic acids in cranberry powder.

Download Application Brief 112: Determination of Organic Acids in Cranberry and Bilberry Extracts

Fruit Juice Adulteration: Profiling

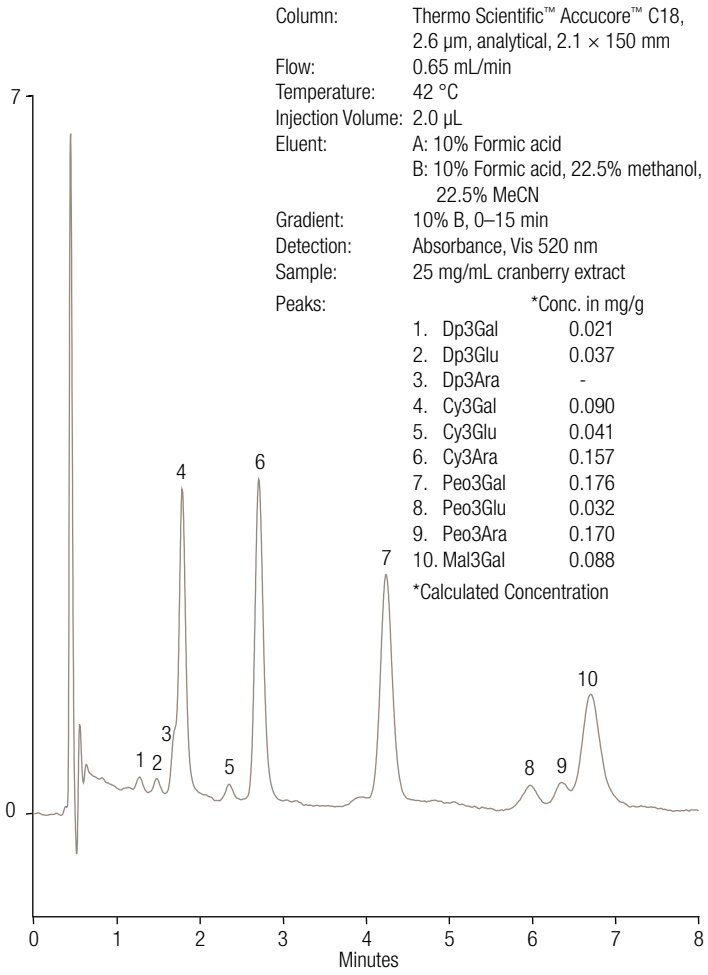

Figure 7-19. Separation of anthocyanins in cranberry powder.

Table 7-1. Comparison of the experimentally determined anthocyanin values to the average values determined by the NIST collaborative study.

Analyte	Experimental Values (mg/g) n = 3	Average Values Reported by Collaborative Study (mg/g)
Dp3Gal	0.021 ± 0.01	0.019 ± 0.03
Dp3Glu	0.097 ± 0.05	0.057 ± 0.06
Cy3Gal	0.090 ± 0.04	0.130 ± 0.04
Cy3Glu	0.041 ± 0.07	0.059 ± 0.08
Cy3Ara	0.157 ± 0.03	0.200 ± 0.06
Peo3Gal	0.176 ± 0.02	0.047 ± 0.05
Peo3Ara	0.170 ± 0.02	0.150 ± 0.05
Mal3Gal	0.088 ± 0.05	0.060 ± 0.09

Melamine Adulteration

In 2008 investigations of death and health problems of babies in China revealed that some baby foods (milk powder) were contaminated by melamine. Some manufacturers illegally used melamine as an adulterant to increase the apparent protein content.

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Melamine in Milk

Standard methods enacted by the Chinese government for determining melamine in raw milk and dairy products included HPLC-UV, LC-MS, and GC-MS methods. However, the high cost of operation and maintenance of GC/LC-MS systems as well as the labor intensive derivatization that GC-MS requires limits their use in the milk product factories. The HPLC-UV method therefore is presently the popular choice for most factories.

HPLC-UV Detection

Guard Column:	Acclaim 120 C18, 5 µm, 4.3 × 10 mm
Anal. Column:	Acclaim 120 C18, 5 µm, 4.6 × 250 mm
Flow:	1.0 mL/min
Column Temp.:	40 °C
Injection Volume:	20 µL
Eluents:	Mix of 10 mM citric acid and 10 mM sodium 1-octane sulfonate (pH 3) CH_3CN (92 : 8, v/v)
Detection:	UV at 240 nm

Traces: A. 0.2 µg/mL
B. 0.5µg/mL
C. 2.0 µg/mL
D. 20 µg/mL
E. 25 µg/mL
F. 50 µg/mL
G. 100 µg/mL

Peak: 1. Melamine

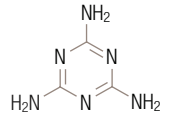

Figure 7-20. Analysis of standards.

Download Application Note 224: Determination of Melamine in Milk Powder by Reversed-Phase HPLC with UV Detection

Melamine in Milk

HPLC-UV Detection

Guard Column:	Acclaim 120 C18, 5 µm, 4.3 × 10 mm
Anal. Column:	Acclaim 120 C18, 5 µm, 4.6 × 250 mm
Flow:	1.0 mL/min
Column Temp.:	40 °C
Injection Volume:	20 µL
Eluents:	Mix of 10 mM citric acid and 10 mM sodium 1-octane sulfonate (pH 3) CH_3CN (92 : 8, v/v)
Detection:	UV at 240 nm
Peak:	1. Melamine

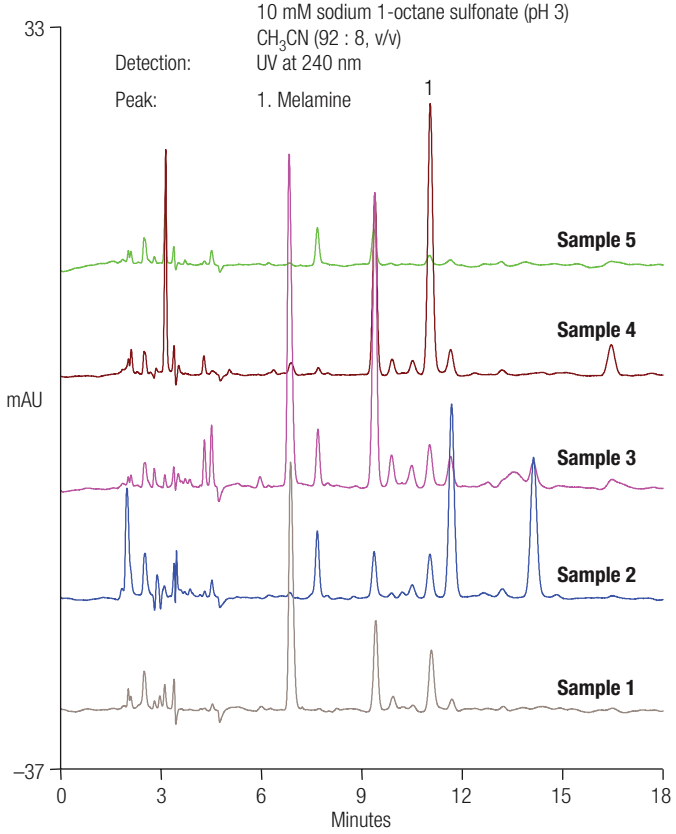

Figure 7-21. Analysis of milk powder samples.

HPLC-Charged Aerosol Detection

HPLC-Charged Aerosol Detection Parameters

Column:	Amino, 4.6 × 250, 5 µm
Flow:	1.0 mL/min
Column Temp.:	30 ºC
Injection Volume:	25 µL
Mobile Phase:	80/10/10 MeCN/MeOH/H_2O

Sample Preparation

A sample of 0.5 mL of milk was placed in a glass tube and 2.0 mL of 80% acetonitrile added to help precipitate any proteins. The tube was mixed well and centrifuge. The supernatant was then poured onto previously condition SPE columns as described below.

Solid Phase Extraction Protocol

1. Condition SPE* Column: 2.0 mL MeOH, 2.0 mL 0.1% TFA,
2. 0.5 mL milk + 2.0 mL 80% ACN, mix, centrifuge
3. Pour onto column
4. Wash: 2.0 mL 0.1% TFA,
5. 2.0 mL MeOH, vacuum dry for 30 seconds
6. Elute: 1.0 mL 5% NH3 in MeOH, Dry for 30 seconds
7. Repeat Step 6
8. Evaporate at 40 ºC
9. Reconstitute with 0.50 mL 50% CAN
10. Analyze

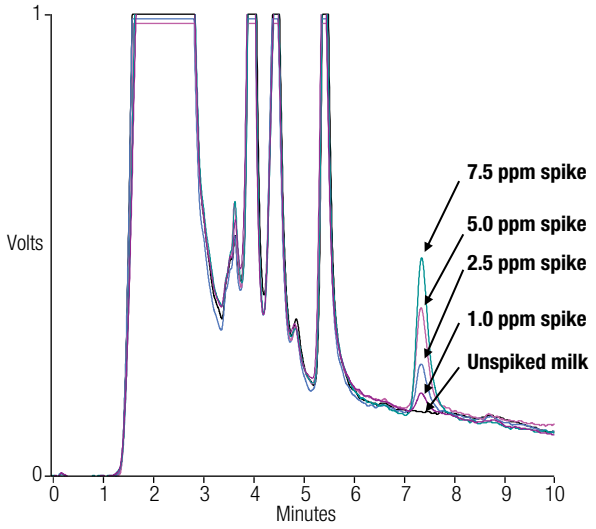

Figure 7-22. Analysis of milk spiked with melamine.

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Melamine and Cyanuric Acid

A few years ago contaminated food led to numerous pet animal deaths and health problems. The toxins were found to be melamine and cyanuric acid, which when present together form an insoluble crystal, causing kidney failure. Since contaminated wheat gluten, rice protein concentrate, and corn gluten used in animal feed can be also used in human food (for example, bread, pasta, and baby food), it is crucial to monitor the presence of melamine and cyanuric acid in raw materials and animal tissue.

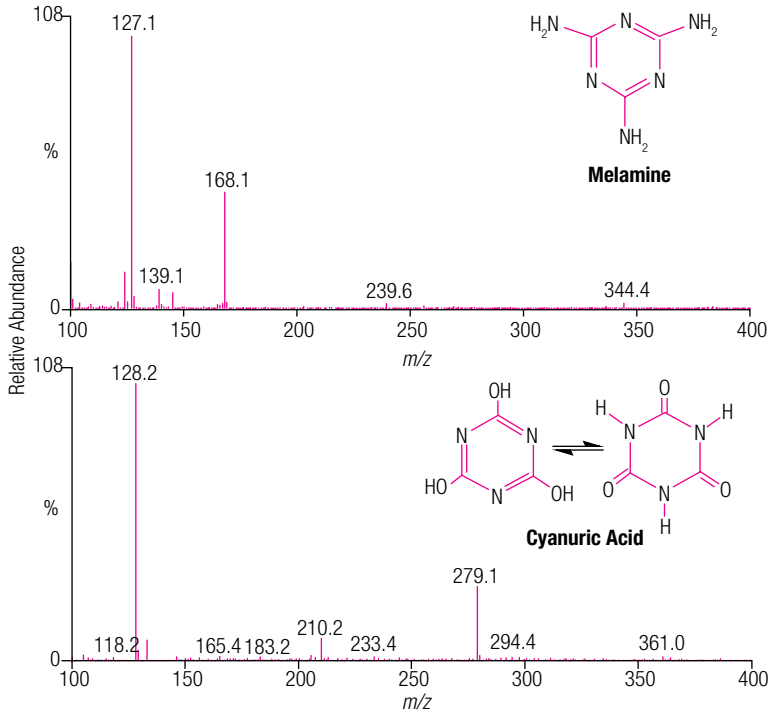

Figure 7-23. Structures of melamine and cyanuric acid and full scan MS spectra.

Column:	Acclaim Mixed-Mode WAX-1, 5 µm, 2.1 × 150 mm	Mass Spectrometric Conditions	
Flow:	0.25 mL/min	Ionization Interface:	Electrospray Ionization (ESI)
Temperature:	20 °C	Detection Mode:	Selected Ion Monitoring (SIM)
Injection Volume:	5 µL	Probe Temp.:	500 C
Eluent:	90% Acetonitrile	Needle Voltage:	2000 V
	10% Ammonium acetate buffer, 20 mM, pH 4	Cone Voltage:	50 V
		Dwell Time:	0.5 s for each SIM
Detection:	MS		

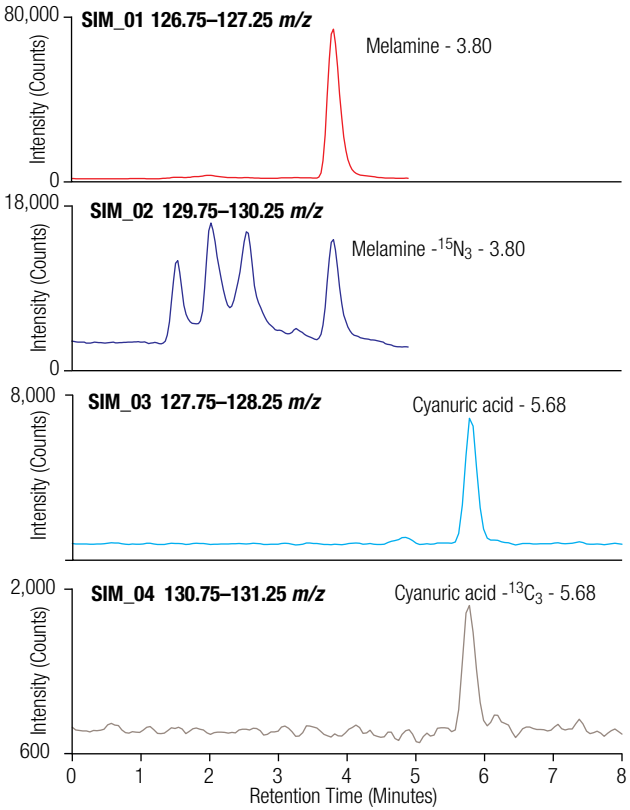

Figure 7-24. SIM chromatogram of an FDA control sample (pork) spiked with melamine, cyanuric acid, and internal standards.

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Curry powder

Mixtures of Complex Materials

Understanding the dynamics of a mixture of complex materials poses difficult analytical problems. The most common analytical approach for solving this issue is to assay for a single, hopefully unique, analyte for each of the constituents in the mixture. However, such marker peaks are not always seen by the analytical approach being used, or if found are not always known.

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Mixtures of Complex Materials

Deconvolution of a Spice Mix

Gradient HPLC with coulometric electrochemical array detection is showing promise for distinguishing components contained in complex mixtures. In Application Brief 168 this approach was used to measure the contribution of cumin and coriander to a curry powder.

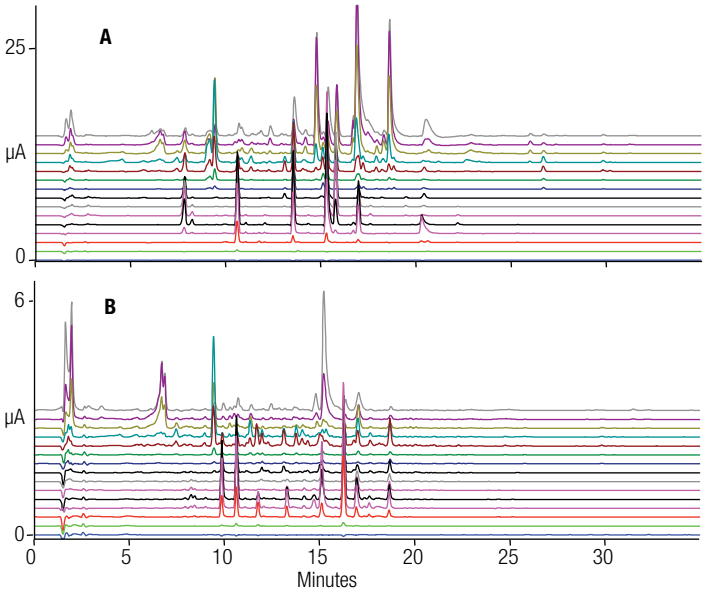


Figure 7-25. Curry powder mixture constituents (A) cumin and (B) coriander.

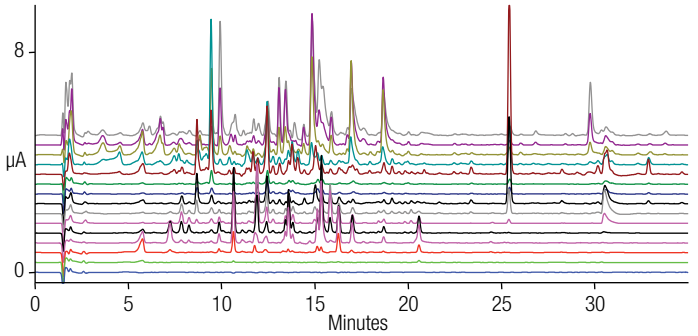


Figure 7-26. Curry powder blend.

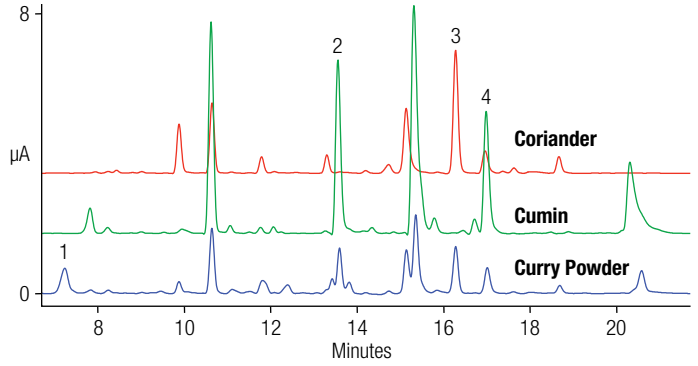


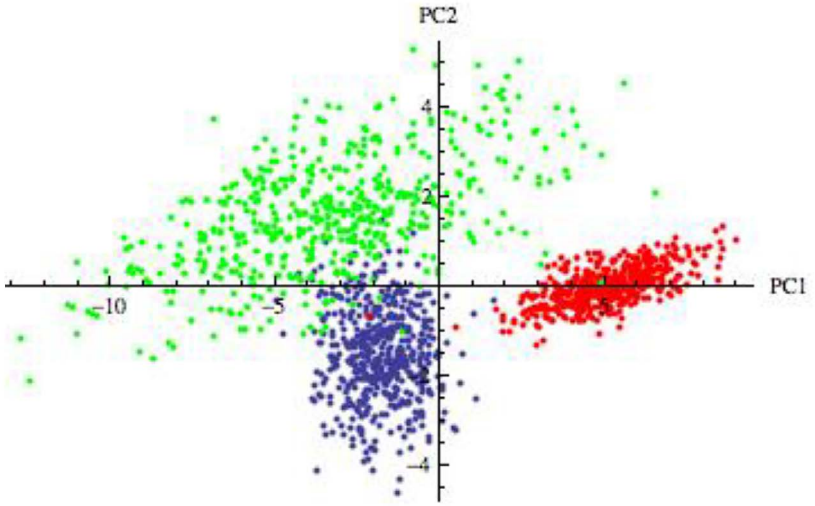
Figure 7-27. Contribution of single components to a complex mixture based on marker peaks. Peak 2: abundance of cumin in mix. Peak 3: abundance of coriander in the mix. Only a single channel is presented for clarity.

Adulteration and Authentication

Part 2

Non-targeted Methods

The measurement of patterns of both known and unknown analytes in a sample to authenticate product and to highlight possible adulteration. This is also referred to as metabolite profiling or metabolomic approaches.



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Adulteration and Authentication

Metabolomics

Screening Herbs for Authenticity and Blending with Adulterants

Herb adulteration can include both inadvertent misidentification of the herb and deliberant blending of the herb with cheaper or more available substitutes. Although the adulteration of herbs is a frequent phenomenon, there are few simple methods available for the screening of large numbers of commercial batches of product.

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Screening Herbs: Chemical Fingerprinting

The challenge to herb screening arises from the complexity and variability of genuine material combined with unrelenting conduct of adulteration. Herb and fruit variety, growing region, season, ripeness, and processing methods all contribute to the variability of the authentic product, making unambiguous characterization difficult.

Currently, one of the most reliable and applicable authentication methods is based on analytical chemical fingerprinting (non-targeted metabolomic) techniques. Gradient HPLC with coulometric electrochemical array detection is particularly suitable for generating information rich metabolite fingerprints of endogenous electroactive metabolites termed the "redoxome".

The analytes that form the redoxome are compounds that influence color, flavor, nutritional, value, stability, and aroma. Such fingerprints can be interrogated using pattern recognition and unsupervised statistical programs such as principal component analysis (PCA) to evaluate the authenticity (or possible adulteration) of an unknown sample by comparing its chromatogram with a compiled population of authenticated reference samples in the database. Poster Note 70534 illustrates the use of this approach to examine deliberate adulteration of herbs.

Authentic herbs and deliberate blends were extracted and analyzed using a gradient HPLC with coulometric electrochemical array detection method. The resulting chromatograms, containing patterns of both known and unknown analytes, were then used to form a database. An herb extract sample can then be analyzed and its metabolite fingerprint checked against the database to confirm authenticity or identify adulteration.

Oregano

Marjoram

Thyme

Thermo SCIENTIFIC

Download the Poster Note: Evaluation of Herb and Fruit Juice Adulteration and Authenticity by Coulometric Array Detection and Pattern

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Screening Herbs: Reference Samples

Column:	Acclaim 120, C18, 3 × 150 mm, 3 µm
Flow:	0.65 mL/min
Injection Volume:	10 or 20 µL
Mobile Phase:	A: 20 mM Monobasic sodium phosphate, 3% acetonitrile, 0.2% tetrahydrofuran, pH 3.35 B: 20 mM Monobasic sodium phosphate, 50% acetonitrile, 10% Tetrahydrofuran, pH 3.45 C: 90% Methanol
Gradient:	0–2 min: 2% B/3% C.; 30 min: 97% B/3% C; 45 min: 97% B/3% C Curve 7
EC Parameters:	16-channel array from 0 to +900 mV in +60 mV increments
Data Station:	Chromeleon CDS 6.8 SR9 and CoulArray software 3.1; Pirouette® software V4.5

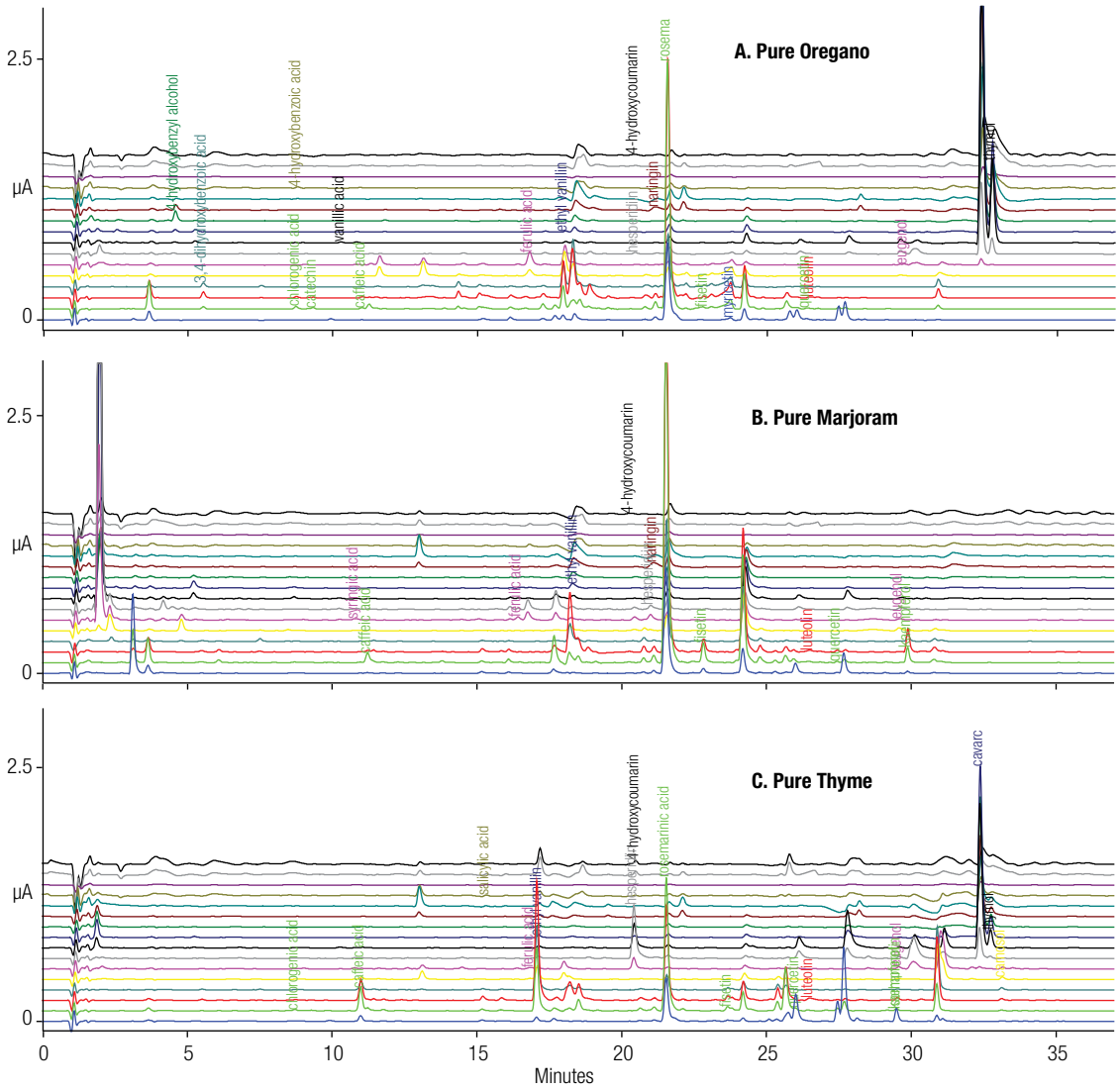

Figure 7-28. EC array chromatograms of A) dried pure oregano, B) dried pure marjoram and C) dried pure thyme herbs (low sensitivity presented for clarity).

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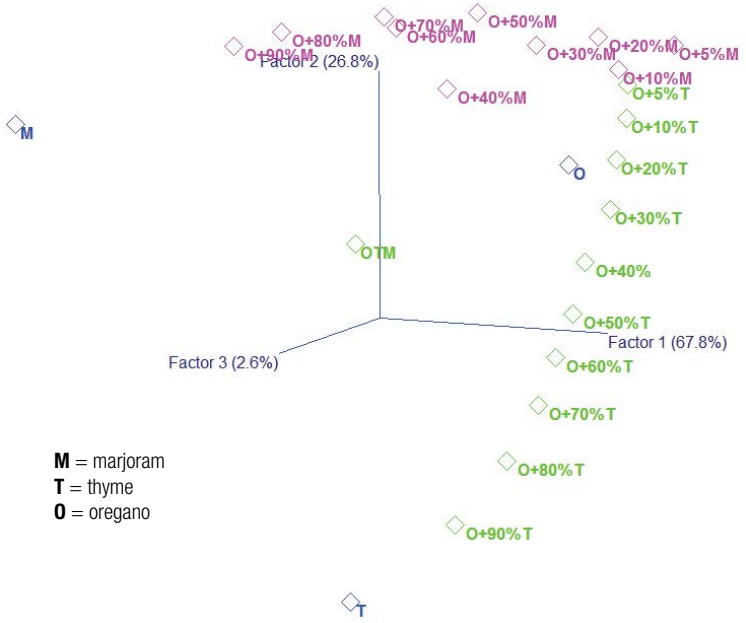
Screening Herbs: Principal Componenent Analysis

For easy visualization, metabolite patterns for each sample can be plotted in 3D space using principal component analysis. The more similar samples analytical fingerprints are, the closer they reside in the 3D plot.

In this way the location of an unknown sample can readily indicate authenticity (fingerprint similarity), level of adulteration, or whether a sample is totally different from samples in the database. As discussed in Application Note 1064 this approach can also be used to authenticate or access adulteration of many different samples such as botanicals, wine, beer, tea, coffee, honey, essential oils, olive oil, and juices.



M = marjoram
T = thyme
O = oregano

Figure 7-29. Principal component analysis of oregano-marjoram and oregano-thyme blends showing how the position in 3D space shifts according to the parent herb and the amount of blending taking place (shown as % in the figure).

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Screening Beer for Origin and Style

Beer is the most widely consumed alcoholic beverage in the world and the third most popular drink after water and tea. It is typically brewed from four basic ingredients: water, a starch source (e.g., malted barley), brewer's yeast, and a flavoring agent such as hops. Many varieties of beer result from differences in these ingredients, the additives used and the brewing process followed.

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Screening Beer: Principal Componenent Analysis

As discussed in Application Note 1065, gradient HPLC with coulometric electrochemical array detection could readily differentiate different beer styles, country of origin, and whether samples were normal or light.

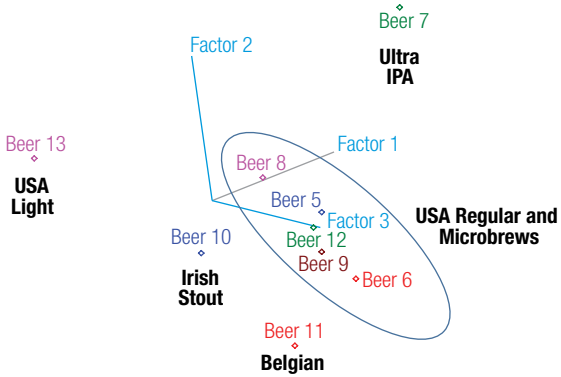

Figure 7-30. Various beer samples differentiated using principal component analysis.

Did You Know?

- Beer is the second most popular beverage in the world, coming in behind tea.
- After consuming a good quantity of a potent brew they called aul, or ale, the Vikings would head fearlessly into battle, often without armor or even shirts. In fact, "berserk" means "bare shirt" in Norse, and eventually took on the meaning of their wild battles.

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Adulteration and Authentication

Metabolomics

Screening for Fraudulent Wine

Wine fraud is a major problem dating back to Roman times. Fraud includes adulteration and packaging cheap, poor quality wine with labels of more expensive, better products.

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Wine: Adulteration and Mislabeling

Wine adulteration is usually done the addition of cheaper products such as water, juices, or cheaper wines, but can also include the addition of harmful chemicals and sweeteners (e.g., ethylene glycol and lead acetate) to compensate color or flavor.

Conformation of fraud is not easy, and many analytical approaches have to be used. As discussed in Application Note 1064, one approach that can be used to study differences between grape varietals, blending, vintage, mislabeling, and effects of growing region is Gradient HPLC with coulometric electrochemical array detection. This approach can target potential marker peaks and can differentiate samples based on their metabolic fingerprints.

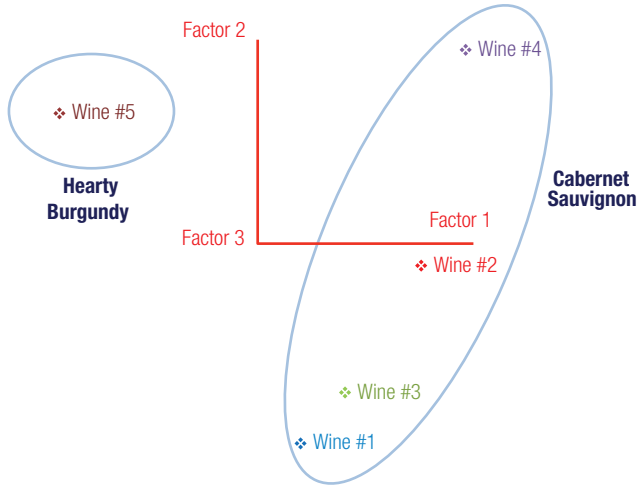

Figure 7-31. Initial study showing principal component analysis of wines and the ability to distinguish between wine types.

Table 7-2. Targeted analysis of some of the more abundant analytes measured in different wine samples. Wine 1: Cabernet Sauvignon, Argentina; Wine 2: Cabernet Sauvignon, South Africa; Wine 3: Cabernet Sauvignon, US; Wine 4: Cabernet Sauvignon, Chile; Wine 5: Hearty Burgundy, US.

Compound	Wine #1 Cabernet Sauvignon, Argentina (mg/L)	Wine #2 Cabernet Sauvignon, So. Africa (mg/L)	Wine #3 Cabernet Sauvignon, U.S. (mg/L)	Wine #4 Cabernet Sauvignon, Chile (mg/L)	Wine #5 Hearty Burgundy, U.S. (mg/L)
Apigenin	16	17.5	9.5	13	41
Caffeic Acid	8	13	5	17	3
Catechin Hydrate	37	26	26.5	24	22
Ellagic Acid Dihydrate	52	133	84	94	100
Epicatechin	19	15	16.5	11	4
Ferulic Acid	1	1	2	3	2
Gallic Acid	57	33.5	37	35	29.5
Isorhamnetin	6	5.5	2.5	6.5	2
Kaempferol	0.5	0.5	0.5	1	1
Myricetin	11	11	5	8	1.5
p-Coumaric Acid	8.5	16	2.5	14.5	3.5
Quercetin Dihydrate	13.5	15.5	3	14	4
cis-Resveratrol	1	1.5	0.5	2	0.5
trans-Resveratrol	2.5	2	1	2.5	1.5
Sinapic Acid	2	2	2	2	2
Syringic Acid	19	9.5	9	12	7
Vanillic Acid	6.5	4.5	2.5	8	4

Adulteration and Authentication

Metabolomics

Fruit Juice Adulteration

Fruit juice adulteration presents an economic and regulatory problem. The most common forms of adulteration include simple dilution and blending of inexpensive and synthetically produced juices into the more expensive ones. Adulteration can also include dilution followed by addition of peel and/or pulp wash.

Learn more about US Fruit Juice Adulteration Regulations

Learn more about the History of Fruit and Vegetable Juice US Regulations

Thermo SCIENTIFIC

Orange Juice Adulteration

The United States orange juice industry estimates that orange juice sales gross more than one billion dollars annually. While it is possible to identify orange juice adulteration through the measurement of some marker peak, or changes in levels of targeted analytes (see carbohydrate and organic ions earlier in the chapter), sometimes this is not possible.

As discussed in Application Note 1064 one approach that is showing promise to distinguish between orange juice varietals, juice dilution, the inclusion of peel or pulp wash or the effect of growing region is gradient HPLC with coulometric electrochemical array detection. A database of sample analyte fingerprints can be used to assess whether adulteration of pure orange juice has taken place.

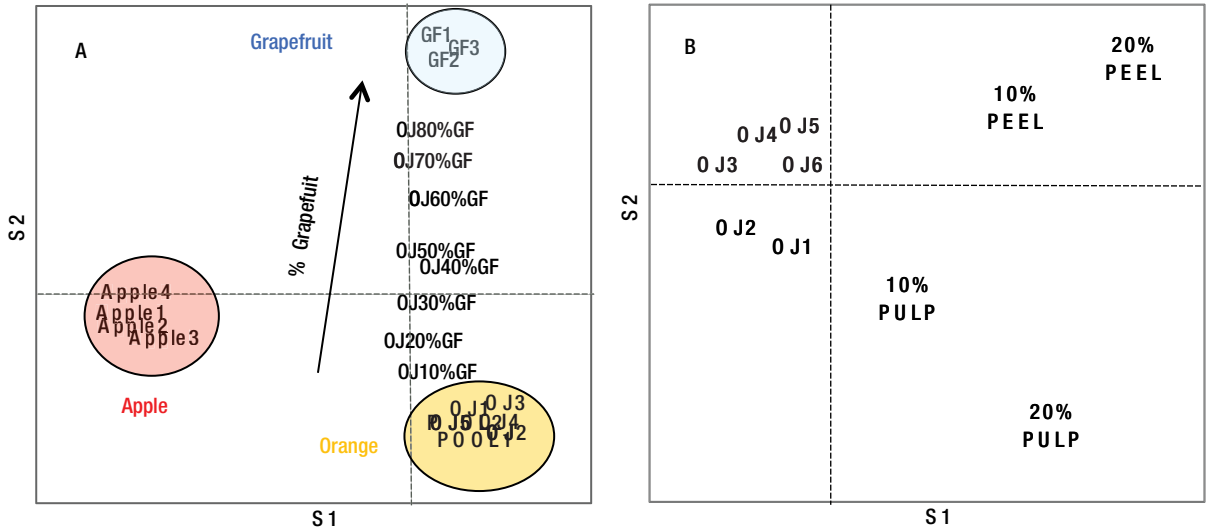

Figure 7-32. Measurement of orange juice adulteration by blending with other juices (A) or by addition of orange peel or pulp wash (B) using the Spectro-Electro Array and PCA. GF – grapefruit; OJ – orange juice; OJ10%GF – orange juice blended with 10% grapefruit juice. POOL – equal blend of several orange juice samples.

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Adulteration and Authentication

Various types of tea leaves

Tea Adulteration

Tea is another product that can readily be adulterated, including the addition of iron or leather flakes, kaolin, and turmeric. Spent tea leaves can be darkened with coal tar and added to normal tea leaves to bulk up the amount for sale. Non-tea leaves may be used. This is especially true for green tea where paddy husks and “lie tea” can be added, as the latter closely resembles tea leaves.

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Tea Adulteration

As discussed in Application Note 1064, one approach that is showing promise for authenticating tea, distinguishing between tea varietals, and with the potential to identify adulteration, is gradient HPLC with coulometric electrochemical array detection. A database of sample analyte fingerprints can be used to assess whether adulteration has taken place.

Table 7-3. Targeted analysis of some of the more abundant analytes measured in different types of tea.

Compound	Green Tea (mg/g)	Black Tea (mg/g)	White Tea (mg/g)
Catechin Hydrate	3.7	3.0	8.1
Epicatechin	50.8	9.3	39.8
Epicatechin Gallate	65.3	40.6	95.9
Epigallocatechin	49.2	2.5	32.3
Epigallocatechin Gallate	180	31.3	211
Gallocatechin	18.8	3.2	22.0
Gallocatechin Gallate	5.9	7.0	3.0

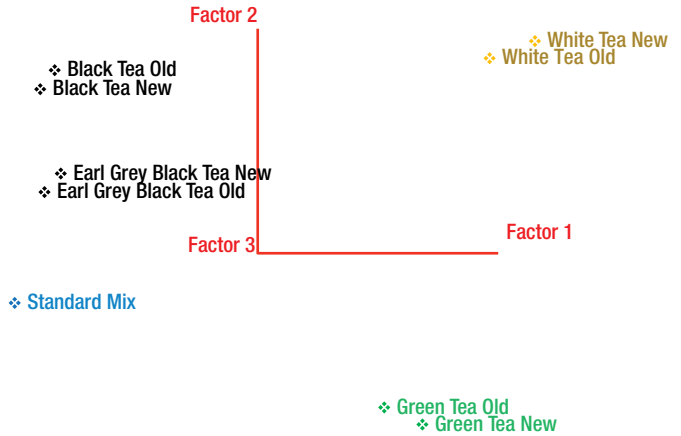

Figure 7-33. Initial study showing principal component analysis of tea and the ability to distinguish between tea types.

Did You Know?

All true tea (white, green, black, and oolong) comes from the same plant, the Camellia sinesis. It takes a minimum of three to five years for a tea bush to grow and be ready for harvesting.

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Adulteration of Botanical Supplements

It is estimated that there are more than 50,000 dietary supplements currently available to the consumer. More than half of the U.S. adult population (53%–55%) consume dietary supplements. Although the most common are multivitamins, a growing proportion are botanical supplements. Unfortunately, there is great opportunity for fraud to take place, including selling the wrong plant species or using the wrong part of the plant (bark instead of root, leaf instead of flower, etc).

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Adulteration of Botanical Supplements

Authentication and identification of botanical supplements is difficult due to the complexity of samples, the fact that they often lack marker peaks, differences in harvesting and drying processes, and the natural variations in metabolite levels due to geographical location and season.

As discussed in Poster Note 70540 one approach that is gaining acceptance for studying botanical supplements is gradient HPLC with coulometric electrochemical array detection. Hoodia supplement is used to illustrate the capabilities of this approach.

Table 7-4. Some examples of botanical supplements and their known adulterants.

Proposed Botanical	Adulterant
Ginkgo (*Ginkgo biloba*) leaf extract standardized to flavonol glycosides and terpenes	Ginkgo (*Ginkgo biloba*) leaf extract with added flavonol glycosides or aglycones (e.g., rutin, quercetin, etc.)
Asian species of *Akebia* and *Clematis* stem	*Aristolochia manshuriensis* stem (*guan mu tong*)
Skullcap herb (*Scutellaria lateriflora*)	Germander herb (*Teucrium chamaedrys*)
Plantain leaf (*Plantago lanceolata*)	*Digitalis lanata* leaf
Black cohosh root/rhizome (*Actaea racemosa*)	Chinese cimicifuga root/rhizome (*Actaea* spp.)

Did You Know?

The leading food categories with reported cases of food fraud include:

- Olive Oil
- Milk and milk-based products
- Honey, maple syrup, and other natural sweeteners
- Fruit juice
- Coffee and tea
- Spices
- Clouding agents

Download the Poster Note: Profiling Hoodia Extracts by HPLC with Charged Aerosol Detection, Electrochemical Array Detection, and Principal Component Analysis

Screening Botanical Supplements

Principal Component Analysis of Hoodia

Authentic Hoodia herb, capsules, and tablets were extracted and analyzed using a gradient HPLC with coulometric electrochemical array detection method. The resulting chromatograms, containing patterns of both known and unknown analytes were then used to form a database. This approach could distinguish between sample types and identify a sample of a common adulterant.

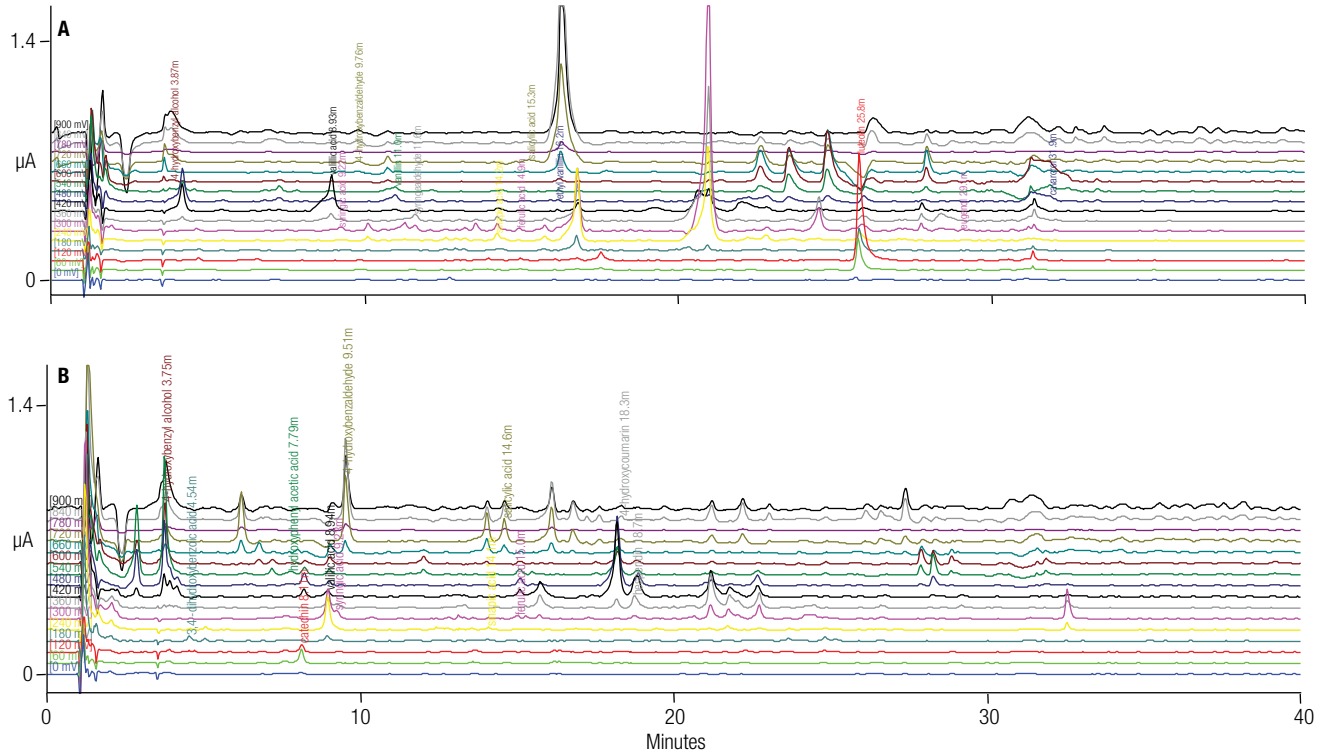

Figure 7-34. HPLC-ECD Array chromatograms of *Hoodia gordonii*: (A) mixed parts extract #3165 and (B) commercial product tablet extract HG6. Thanks to Drs. Ikhlas Khan and Bharathi Avula of the National Center for Natural Products Research at The University of Mississippi, Oxford, MS, for providing the plant materials and hoodigoside standards.

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Screening Botanical Supplements

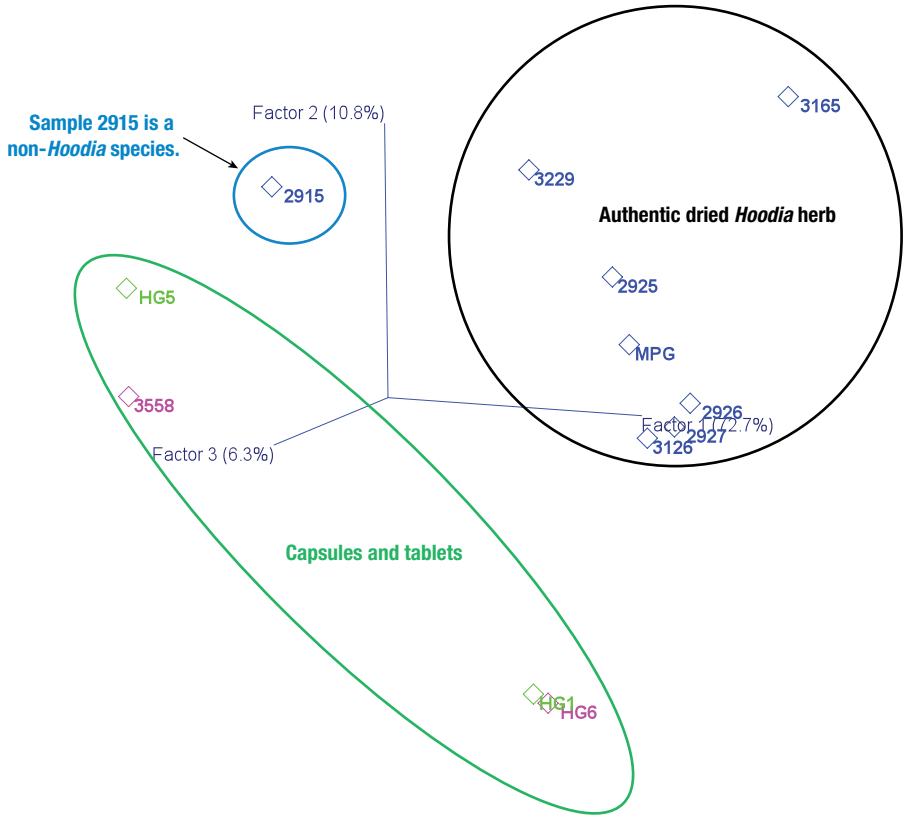

Figure 7-35. Principal component analysis of hoodia samples showing differentiation between authentic dried plant, capsules/tablets and adulterant.

Did You Know?

The Grocery Manufacturers Association (GMA) estimates that fraud may cost the global food industry between $10 billion and $15 billion per year, affecting approximately 10% of all commercially sold food products. Compared to the trillions of dollars spent on food and food ingredients globally each year, however, the prevalence of food fraud is ultimately very low.

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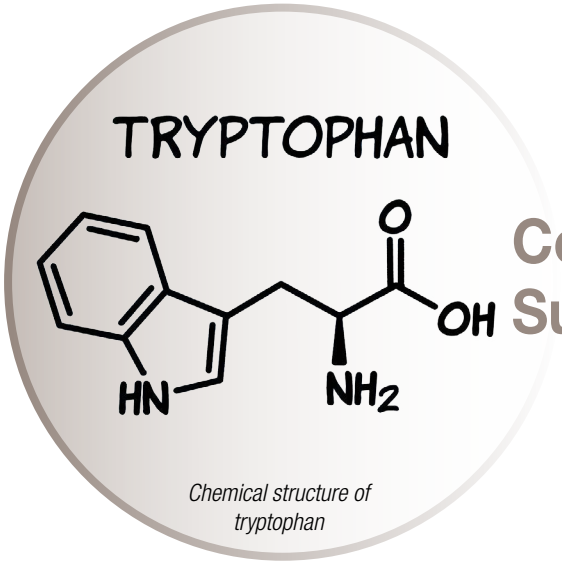

Chemical structure of tryptophan

Contamination of Tryptophan Supplements

Eosinophilia-Myalgia Syndrome (EMS) is an incurable and sometimes fatal flu-like neurological condition first recognized in 1989. EMS initially reported in New Mexico, was found to be related to use of L-tryptophan. Evidence suggested that the cause of the problem was improperly prepared tryptophan supplements, contaminated with potentially toxic metabolites. EMS resulted in 37 deaths and the banning of tryptophan supplements from 1991 to 2001

Screening Tryptophan Supplements

Analysis of tryptopthan supplements, commonly used for insomnia, depression, and PMS, using gradient HPLC with coulometric electrochemical array detection generated patterns of hundreds of electrochemically active analytes—both known and unknown compounds. Interrogation of these metabolomic profiles using chemometric software readily distinguished between control and contaminated supplement samples.

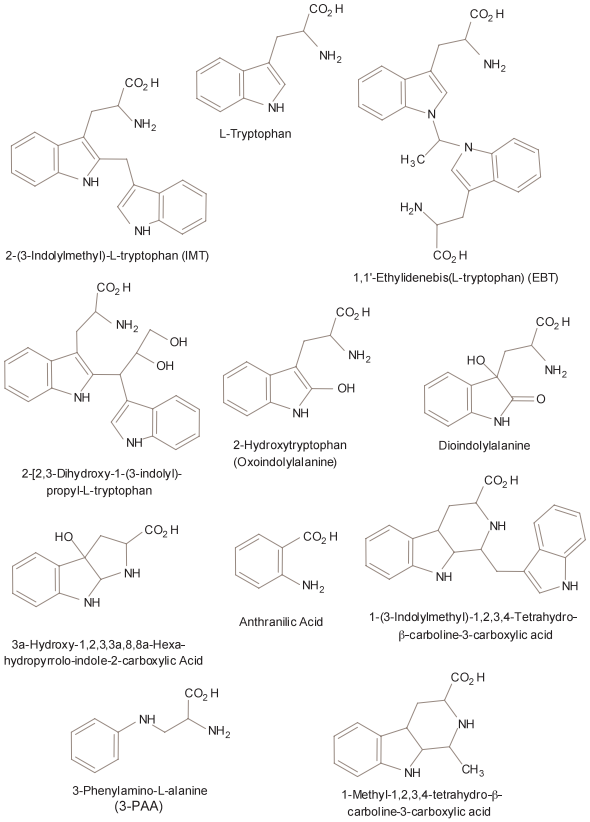

Figure 7-36. Structures of some possible tryptophan contaminants.

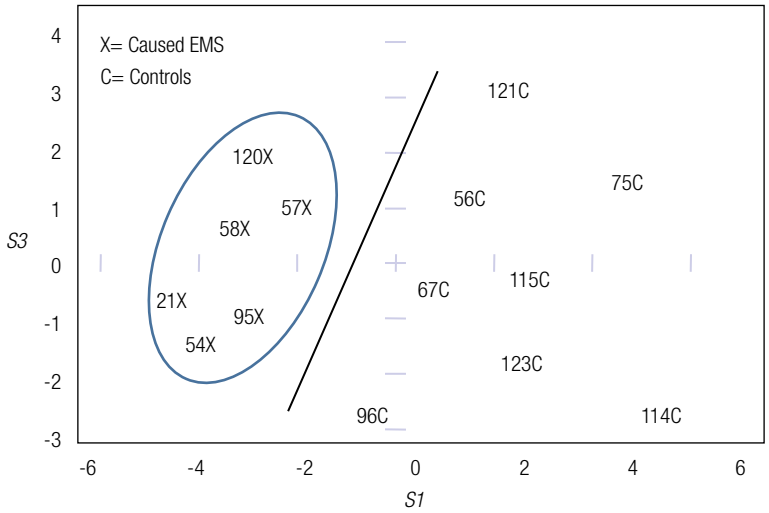

Figure 7-37. Principal component analysis of control and contaminated tryptophan supplements.

Download Application Brief 169: Metabolic Fingerprinting to Determine Tryptophan Contamination

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Adulteration of Olive Oil

Olive oil, high in monounsaturated fatty acids, is a key component of the heart-healthy Mediterranean diet recommended by doctors. But not all olive oils are nutritionally the same. There are a number of commercial grades of olive oil differing in quality, nutrient levels, antioxidant activity, and final chemical composition.

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Screening Olive Oil for Adulteration

"Virgin" olive oil is the juice of freshly harvested olive fruits; "extra virgin" is virgin olive oil of the highest quality based on both sensory and physical–chemical, characteristics. "Refined olive oil" is one that has been chemically purified; commercial "olive oil" is a blend of virgin and refined oils. Finally, "pomace" olive oil one that is obtained from the solid remains of olives already pressed for juice using solvent extraction.

Unfortunately, as consumer demand for extra virgin olive oil (EVOO) is set to outpace production, the inevitable increase in EVOO price makes it an ideal target for adulteration. Adulteration can take many forms. The ones that impact health the least are those that maintain the safety of the product such as passing off inferior olive oil as superior olive oil, or blending olive oil with cheaper oils such as sunflower, soybean, sesame, rapeseed, canola, corn, palm, and hazelnut. Blending with peanut oil is of concern for those with peanut allergies.

As discussed in Poster Note 70689, to help tackle the problem of olive oil adulteration a simple HPLC-based method using charged aerosol detection was used to generate patterns of fats (triglycerides) found in oil samples. These patterns can be used to determine pure EVOO samples from olive oil blended with corn oil, hazelnut oil, or pomace.

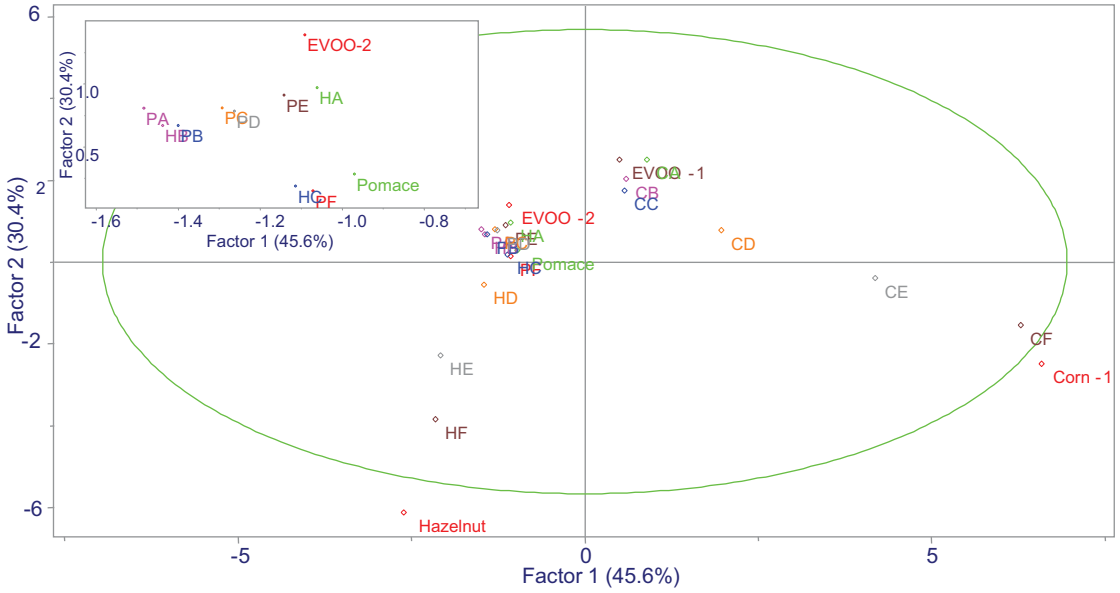

Figure 7-38. Principal component analysis of hydrolyzed EVOO with hazelnut (HA-HF), corn (CA–CF), and pomace oil (PA–PF) adulteration at six levels (1, 5, 10, 25, 50, and 75 mass-%, respectively) using hydrolyzed oil data. Inset showsresults for pomace oil analysis.

Download the Poster Note: Determination of Olive Oil Adulteration by Principal Component Analysis with HPLC-Charged Aerosol Detection Data

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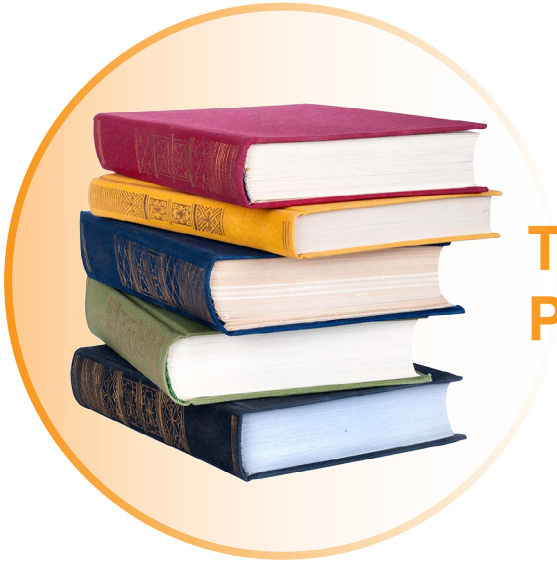

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RP-HPLC analysis of phenolic compounds and flavonoids in beverages and plant extracts using a CoulArray detector	Jandera, P.; Skeifíková, V.; Rehová, L.; Hájek, T.; Baldriánová, L.; Skopová, G.; Kellner, V.; Horna, A.	*J. Sep. Sci. 28 (9–10),* 1005–1022	2005 Jun
A new application of charged aerosol detection in liquid chromatography for the simultaneous determination of polar and less polar ginsenosides in ginseng products	Jia, S.; Li, J.; Yunusova, N.; Park, J. H.; Kwon, S. W.; Lee, J.	*Phytochem. Anal. 24 (4),* 374–380	2013 Jul–Aug
A combination of aspirin and γ-tocopherol is superior to that of aspirin and α-tocopherol in anti-inflammatory action and attenuation of aspirin-induced adverse effects	Jiang, Q.; Moreland, M.; Ames, B. N.; Yin, X.	*J. Nutr. Biochem. 20 (11),* 894–900	2009 Nov
HPLC analysis of rosmarinic acid in feed enriched with aerial parts of *Prunella vulgaris* and its metabolites in pig plasma using dual-channel coulometric detection	Jirovský, D.; Kosina, P.; Myslínová, M.; Stýskala, J.; Ulrichová, J.; Simánek V.	*J. Agric. Food Chem. 55 (19),* 7631–7637	2007 Sep 19
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Sensitive electrochemical detection method for alpha-acids, beta-acids and xanthohumol in hops (*Humulus lupulus L.*)	Kac, J.; Vovk, T.	*J. Chromatogr., B: Anal. Technol. Biomed. Life Sci. 850 (1–2),* 531–537	2007 May 1
Determination of phenolic compounds and hydroxymethylfurfural in meads using high performance liquid chromatography with coulometric-array and UV detection	Kahoun, D.; Rezková, S.; Veskrnová, K.; Královský, J.; Holcapek, M.	*J. Chromatogr., A 1202 (1),* 19–33	2008 Aug 15
Analysis of terpene lactones in a Ginkgo leaf extract by high-performance liquid chromatography using charged aerosol detection	Kakigi, Y.; Mochizuki, N.; Icho, T.; Hakamatsuka, T.; Goda, Y.	*Biosci., Biotechnol., Biochem. 74 (3),* 590–594	2010
Linear aglycones are the substrates for glycosyltransferase DesVII in methymycin biosynthesis: analysis and implications	Kao, C.; Borisova, S.; Kim, H.; Liu, H.	*J. Am. Chem. Soc. 128 (17),* 5606–5607	2006 May 3

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Title	Authors	Publication	Publication Date
Antioxidant-rich food intakes and their association with blood total antioxidant status and vitamin C and E levels in community-dwelling seniors from the Quebec longitudinal study NuAge	Khalil, A.; Gaudreau, P.; Cherki, M.; Wagner, R.; Tessier, D. M.; Fulop, T.; Shatenstein, B.	*Exp. Gerontol. 46 (6),* 475–481	2011 Jun
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Optimization of pressurized liquid extraction for spicatoside A in *Liriope platyphylla*	Kim, S. H.; Kim, H. K.; Yang, E. S.; Lee, K. Y.; Kim, S. D.; Kim, Y. C.; Sung, S. H.	*Sep. Purif. Technol. 71 (2),* 168–172	2010
Production of surfactin and iturin by *Bacillus licheniformis* N1 responsible for plant disease control activity	Kong, H. G.; Kim, J. C.; Choi, G. J.; Lee, K. Y.; Kim, H. J.; Hwang, E. C.; Moon, B. J.; Lee, S. W.	*Plant Pathol. J. 26 (2),* 170–177	2010
Transepithelial transport of microbial metabolites of quercetin in intestinal Caco-2 cell monolayers	Konishi, Y.	*J. Agric. Food Chem. 53 (3),* 601–607	2005 Feb 9
Absorption and bioavailability of artepillin C in rats after oral administration	Konishi, Y.; Hitomi, Y.; Yoshida, M.; Yoshioka, E.	*J. Agric. Food Chem. 53 (26),* 9928–9933	2005 Dec 28
Pharmacokinetic study of caffeic and rosmarinic acids in rats after oral administration	Konishi, Y.; Hitomi, Y.; Yoshida, M.; Yoshioka, E.	*J. Agric. Food Chem. 53 (12),* 4740–4746	2005 Jun 15
Intestinal absorption of *p*-coumaric and gallic acids in rats after oral administration	Konishi, Y.; Hitomi, Y.; Yoshioka, E.	*J. Agric. Food Chem. 52 (9),* 2527–2532	2004 May 5
Microbial metabolites of ingested caffeic acid are absorbed by the monocarboxylic acid transporter (MCT) in intestinal Caco-2 cell monolayers	Konishi, Y.; Kobayashi, S.	*J. Agric. Food Chem. 52 (21),* 6418–6424	2004 Oct 20
Transepithelial transport of rosmarinic acid in intestinal Caco-2 cell monolayers	Konishi, Y.; Kobayashi, S.	*Biosci., Biotechnol., Biochem. 69 (3),* 583–591	2005 Mar
Effects of various doses of selenite on stinging nettle (*Urtica dioica L.*)	Krystofova, O.; Adam, V.; Babula, P.; Zehnalek, J.; Beklova, M.; Havel, L.; Kizek, R.	*Int. J. Environ. Res. Public Health 7 (10),* 3804–3815	2010 Oct
Biofortified cassava increases β-carotene and vitamin A concentrations in the TAG-rich plasma layer of American women	La Frano, M. R.; Woodhouse, L. R.; Burnett, D. J.; Burri, B. J.	*Br. J. Nutr. 110 (2),* 310–320	2013 Jul 28
Chlorogenic acid is absorbed in its intact form in the stomach of rats	Lafay, S.; Gil-Izquierdo, A.; Manach, C.; Morand, C.; Besson, C.; Scalbert, A.	*J. Nutr. 136 (5),* 1192–1197	2006 May
Determination of 4-ethylcatechol in wine by high-performance liquid chromatography-coulometric electrochemical array detection	Larcher, R.; Nicolini, G.; Bertoldi, D.; Nardin, T.	*Anal. Chim. Acta 609 (2),* 235–240	2008 Feb 25
Determination of volatile phenols in wine using high-performance liquid chromatography with a coulometric array detector	Larcher, R.; Nicolini, G.; Puecher, C.; Bertoldi, D.; Moser, S.; Favaro, G.	*Anal. Chim. Acta 582 (1),* 55–60	2007 Jan 16

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Title	Authors	Publication	Publication Date
Acute, quercetin-induced reductions in blood pressure in hypertensive individuals are not secondary to lower plasma angiotensin-converting enzyme activity or endothelin-1: nitric oxide	Larson, A.; Witman, M. A. H.; Guo, Y.; Ives, S.; Richardson, R. S.; Bruno, R. S.; Jalili, T.; Symons, J. D.	*Nutr. Res. (N. Y., NY, U.S.) 32 (8),* 557–564	2012 Aug
High-performance liquid chromatography method for the determination of folic acid in fortified food products	Lebiedzińska, A.; Dąbrowska, M.; Szefer, P.; Marszałł M.	*Toxicol. Mech. Methods 18 (6),* 463–467	2008 Jul
Reversed-phase high-performance liquid chromatography method with coulometric electrochemical and ultraviolet detection for the quantification of vitamins B(1) (thiamine), B(6) (pyridoxamine, pyridoxal and pyridoxine) and B(12) in animal and plant foods	Lebiedzińska, A.; Marszałł, M. L.; Kuta, J.; Szefer, P.	*J. Chromatogr., A 1173 (1–2),* 71–80	2007 Nov 30
An improved method for the determination of green and black tea polyphenols in biomatrices by high-performance liquid chromatography with coulometric array detection	Lee, M. J.; Prabhu, S.; Meng, X.; Li, C.; Yang, C. S.	*Anal. Biochem. 279 (2),* 164–169	2000 Mar 15
Characterisation, extraction efficiency, stability and antioxidant activity of phytonutrients in *Angelica keiskei*	Li, L.; Aldini, G.; Carini, M.; Chen, C. Y. O.; Chun, H.; Cho, S.; Park, K.; Correa, C. R.; Russell, R. M.; Blumberg, J. B.; Yeum, K.	*Food Chem.* 115 (1), 227–232	2009 Jul
Vitamin A equivalence of the β-carotene in β-carotene-biofortified maize porridge consumed by women	Li, S.; Nugroho, A.; Rocheford, T.; White, W. S.	*Am. J. Clin. Nutr. 92 (5),* 1105–1112	2010 Nov
Phase IIa chemoprevention trial of green tea polyphenols in high-risk individuals of liver cancer: modulation of urinary excretion of green tea polyphenols and 8-hydroxydeoxyguanosine	Luo, H.; Tang, L.; Tang, M.; Billam, M.; Huang, T.; Yu, J.; Wei, Z.; Liang, Y.; Wang, K.; Zhang, Z. Q.; Zhang, L.; Wang, J. S.	*Carcinogenesis 27 (2),* 262–268	2006 Feb
Determination of four water-soluble compounds in *Salvia miltiorrhiza Bunge* by high-performance liquid chromatography with a coulometric electrode array system	Ma, L.; Zhang, X.; Guo, H.; Gan, Y.	*J. Chromatogr., B: Anal. Technol. Biomed. Life Sci. 833* (2), 260–263	2006 Apr 3
Effect of green tea powder (*Camellia sinensis L.* cv. Benifuuki) particle size on O-methylated EGCG absorption in rats, The Kakegawa Study	Maeda-Yamamoto, M.; Ema, K.; Tokuda, Y.; Monobe, M.; Tachibana, H.; Sameshima, Y.; Kuriyama, S.	*Cytotechnology 63 (2),* 171–179	2011 Mar
Supplementation of a γ-tocopherol-rich mixture of tocopherols in healthy men protects against vascular endothelial dysfunction induced by postprandial hyperglycemia	Mah, E.; Noh, S. K.; Ballard, K. D.; Park, H. J.; Volek, J. S.; Bruno, R. S.	*J. Nutr. Biochem. 24 (1),* 196–203	2013 Jan

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Mediterranean diet reduces endothelial damage and improves the regenerative capacity of endothelium	Marin, C.; Ramirez, R.; Delgado-Lista, J.; Yubero-Serrano, E. M.; Perez-Martinez, P.; Carracedo, J.; Garcia-Rios, A.; Rodriguez. F.; Gutierrez-Mariscal, F. M.; Gomez, P.; Perez-Jimenez, F.; Lopez-Miranda, J.	*Am. J. Clin. Nutr. 93 (2),* 267–274	2011 Feb
Photodiode array (PDA) and other detection methods in HPLC of plant metabolites	Markowski, W.; Waksmundzka-Hajnos, M.	Chapter 13 in *High Performance Liquid Chromatography in Phytochemical Analysis,* Chromatographic Science Series, Markowski, W., Sherma, J., Eds.; Taylor & Francis Group, LLC: Boca Raton, FL; 331–350	2010 Nov
Determination of water-soluble vitamins in infant milk and dietary supplement using a liquid chromatography on-line coupled to a corona-charged aerosol detector	Márquez-Sillero, I.; Cárdenas, S.; Valcárcel, M.	*J. Chromatogr., A. 1313C,* 253–258	2013 Oct 25
Sensitive high-performance liquid chromatographic method using coulometric electrode array detection for measurement of phytoestrogens in dried blood spots	Melby, M. K.; Watanabe, S.; Whitten, P. L.; Worthman, C. M.	*J. Chromatogr., B: Anal. Technol. Biomed. Life Sci. 826 (1–2),* 81–90	2005 Nov 5
Phenolic acids from beer are absorbed and extensively metabolized in humans	Nardini, M.; Natella, F.; Scaccini, C.; Ghiselli, A.	*J. Nutr. Biochem. 17 (1),* 14–22	2006 Jan
High-performance liquid chromatography analysis of plant saponins: An update 2005-2010	Negi, J. S.; Singh, P.; Pant, G. J.; Rawat, M. S.	*Pharmacogn. Rev. 5 (10),* 155–158	2011 Jul
Physicochemical effect of pH and antioxidants on mono- and triglutamate forms of 5-methyltetrahydrofolate, and evaluation of vitamin stability in human gastric juice: Implications for folate bioavailability	Ng, X.; Lucock, M.; Veysey, M.	*Food Chem. 106 (1),* 200–210	2008 Jan
Practical preparation of lacto-*N*-biose I, a candidate for the bifidus factor in human milk	Nishimoto, M.; Kitaoka, M.	*Biosci., Biotechnol., Biochem. 71 (8),* 2101-2104	2007 Aug
Hydrophilic interaction liquid chromatography—charged aerosol detection as a straightforward solution for simultaneous analysis of ascorbic acid and dehydroascorbic acid	Nováková, L.; Solichová, D.; Solich, P.	*J. Chromatogr., A. 1216 (21),* 4574–4581	2009 May 22

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No effect on adenoma formation in Min mice after moderate amount of flaxseed	Oikarinen, S.; Heinonen, S. M.; Nurmi, T.; Adlercreutz, H.; Mutanen, M.	*Eur. J. Nutr. 44 (5),* 273–280	2005 Aug
Measurement of isoflavones using liquid chromatography with multi-channel coulometric electrochemical detection	Ouchi, K.; Gamache, P.; Acworth, I.; Watanabe, S.	*BioFactors. 22 (1–4),* 353–356	2004
Quantitation of clovamide-type phenylpropenoic acid amides in cells and plasma using high-performance liquid chromatography with a coulometric electrochemical detector	Park, J. B.	*J. Agric. Food Chem. 53 (21),* 8135–8140	2005 Oct 19
Synthesis, HPLC measurement and bioavailability of the phenolic amide amkamide	Park, J. B.	*J. Chromatogr. Sci.* [Epub ahead of print]	2013 May 27
Synthesis of safflomide and its HPLC measurement in mouse plasma after oral administration	Park, J. B.; Chen, P.	*J. Chromatogr., B: Anal. Technol. Biomed. Life Sci.* 852 (1–2), 398–402	2007 Jun 1
Determination of lignans in human plasma by liquid chromatography with coulometric electrode array detection	Peñalvo, J. L.; Nurmi, T.; Haajanen, K.; Al-Maharik, N.; Botting, N.; Adlercreutz, H.	*Anal. Biochem. 332 (2),* 384–393	2004 Sep 15
Supercritical antisolvent fractionation of lignans from the ethanol extract of flaxseed	Perretti, G.; Virgili, C.; Troilo, A.; Marconi, O.; Regnicoli, G. F.; Fantozzi, P.	*J. Supercrit. Fluids 75,* 94–100	2013 Mar
Analysis of flavonoids in honey by HPLC coupled with coulometric electrode array detection and electrospray ionization mass spectrometry	Petrus, K.; Schwartz, H.; Sontag, G.	*Anal. Bioanal. Chem. 400 (8),* 2555–2563	2011 Jun
High-dose supplementation with natural α-tocopherol does neither alter the pharmacodynamics of atorvastatin nor its phase I metabolism in guinea pigs	Podszun, M. C.; Grebenstein, N.; Hofmann, U.; Frank, J.	*Toxicol. Appl. Pharmacol. 266 (3),* 452–458	2013 Feb 1
Application of high-performance liquid chromatography with charged aerosol detection for universal quantitation of undeclared phosphodiesterase-5 inhibitors in herbal dietary supplements	Poplawska, M.; Blazewicz, A.; Bukowinska, K.; Fijalek, Z.	*J. Pharm. Biomed. Anal. 84,* 232–243	2013 Oct
Isolation and analysis of ginseng: advances and challenges	Qi, L.; Wang, C.; Yuan, C.	*Nat. Prod. Rep. 28 (3),* 467–495	2011 Mar
Folate analysis in complex food matrices: Use of a recombinant Arabidopsis γ-glutamyl hydrolase for folate deglutamylation	Ramos-Parra, P. A.; Urrea-López, R.; Díaz de la Garza, R. I.	*Food Res. Int. 54 (1),* 177–185	2013 Nov
Optimisation of gradient HPLC analysis of phenolic compounds and flavonoids in beer using a coularray detector	Rehová, L.; Skeríková, V.; Jandera, P.	*J. Sep. Sci. 27 (15–16),* 1345–1359	2004 Nov
Chiral separation of (+)/(-)-catechin from sulfated and glucuronidated metabolites in human plasma after cocoa consumption	Ritter, C.; Zimmermann, B. F.; Galensa, R.	*Anal. Bioanal. Chem. 397 (2),* 723–730	2010 May

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Analysis of alkylresorcinols in cereal grains and products using ultrahigh-pressure liquid chromatography with fluorescence, ultraviolet, and CoulArray electrochemical detection	Ross, A. B.	*J. Agric. Food Chem. 60 (36),* 8954–8962	2012 Sep 12
Rapid and sensitive analysis of alkylresorcinols from cereal grains and products using HPLC-CoulArray-based electrochemical detection	Ross, A. B.; Kochhar, S.	*J. Agric. Food Chem. 57 (12),* 5187–5193	2009 Jun 24
Analysis of soy isoflavone plasma levels using HPLC with coulometric detection in postmenopausal women	Saracino, M. A.; Raggi, M. A.	*J. Pharm. Biomed. Anal. 53 (3),* 682–687	2010 Nov 2
A biosynthetic pathway for BE-7585A, a 2-thiosugar-containing angucycline-type natural product	Sasaki, E.; Ogasawara, Y.; Liu, H. W.	*J. Am. Chem. Soc. 132 (21),* 7405–7417	2010 Jun 2
The senescence-accelerated mouse-prone 8 is not a suitable model for the investigation of cardiac inflammation and oxidative stress and their modulation by dietary phytochemicals	Schiborr, C.; Schwamm, D.; Kocher, A.; Rimbach, G.; Eckert, G. P.; Frank, J.	*Pharmacol. Res. 74,* 113–120	2013 Aug
Comprehensive impurity profiling of nutritional infusion solutions by multidimensional off-line reversed-phase liquid chromatography × hydrophilic interaction chromatography-ion trap mass-spectrometry and charged aerosol detection with universal calibration	Schiesel, S.; Lämmerhofer, M.; Lindner, W.	*J. Chromatogr., A. 1259,* 100–10	2012 Oct 12
The effect of α-tocopherol supplementation on training-induced elevation of S100B protein in sera of basketball players	Schulpis, K. H.; Moukas, M.; Parthimos, T.; Tsakiris, T.; Parthimos, N.; Tsakiris, S.	*Clin. Biochem. 40 (12),* 900–906	2007 Aug
Determination of secoisolariciresinol, lariciresinol and isolariciresinol in plant foods by high performance liquid chromatography coupled with coulometric electrode array detection	Schwartz, H.; Sontag, G.	*J. Chromatogr., B: Anal. Technol. Biomed. Life Sci.* 838 (2), 78–85	2006 Jul 11
Assessment of probiotic strains ability to reduce the bioaccessibility of aflatoxin M 1 in artificially contaminated milk using an in vitro digestive model	Serrano-Niño, J. C.; Cavazos-Garduño, A.; Hernandez-Mendoza, A.; Applegate, B.; Ferruzzi, M. G.; San Martin-González, M. F.; García, H. S.	*Food Control 31 (1),* 202–207	2013 May
Intestinal uptake of quercetin-3-glucoside in rats involves hydrolysis by lactase phlorizin hydrolase	Sesink, A. L.; Arts, I. C.; Faassen-Peters, M.; Hollman, P. C.	*J. Nutr. 133 (3),* 773–776	2003 Mar
Quercetin glucuronides but not glucosides are present in human plasma after consumption of quercetin-3-glucoside or quercetin-4'-glucoside	Sesink, A. L.; O'Leary, K. A.; Hollman, P. C.	*J. Nutr. 131 (7),* 1938–1941	2001 Jul
Co-administration of quercetin and catechin in rats alters their absorption but not their metabolism	Silberberg, M.; Morand, C.; Manach, C.; Scalbert, A.; Remesy, C.	*Life Sci. 77 (25),* 3156–3167	2005 Nov 4
Nutritional status is altered in the self-neglecting elderly	Smith, S. M.; Mathews Oliver, S. A.; Zwart, S. R.; Kala, G.; Kelly, P. A.; Goodwin, J. S.; Dyer, C. B.	*J. Nutr. 136 (10),* 2534–2541	2006 Oct

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Binding of heterocyclic aromatic amines by lactic acid bacteria: results of a comprehensive screening trial	Stidl, R.; Sontag, G.; Koller, V.; Knasmüller, S.	*Mol. Nutr. Food Res. 52 (3),* 322–329	2008 Mar
Direct separation and detection of biogenic amines by ion-pair liquid chromatography with chemiluminescent nitrogen detector	Sun, J.; Guo, H. X.; Semin, D.; Cheetham, J.	*J. Chromatogr., A. 1218 (29),* 4689–4697	2011 Jul 22
Rapid purification method for fumonisin B1 using centrifugal partition chromatography	Szekeres, A.; Lorántfy, L.; Bencsik, O.; Kecskeméti, A.; Szécsi, Á.; Mesterházy, Á.; Vágvölgyi, C.	*Food Addit. Contam. 30 (1),* 147–155	2013
Determination of coenzyme Q10 in over-the-counter dietary supplements by high-performance liquid chromatography with coulometric detection	Tang, P. H.	*J. AOAC Int. 89 (1),* 35–39	2006 Jan–Feb
α-Tocopherol supplementation restores the reduction of erythrocyte glucose-6-phosphate dehydrogenase activity induced by forced training	Tsakiris, S.; Reclos, G. J.; Parthimos, T.; Tsakiris, T.; Parthimos, N.; Schulpis, K. H.	*Pharmacol. Res. 54 (5),* 373–379	2006 Nov
Tissue distribution of isoflavones in ewes after consumption of red clover silage	Urpi-Sarda, M.; Morand, C.; Besson, C.; Kraft, G.; Viala, D.; Scalbert, A.; Besle, J. M.; Manach, C.	*Arch. Biochem. Biophys. 476 (2),* 205–210	2008 Aug 15
Performance evaluation of charged aerosol and evaporative light scattering detection for the determination of ginsenosides by LC	Wang, L.; He, W. S.; Yan, H. X.; Jiang, Y.; Bi, K. S.; Tu, P. F.	*Chromatographia 70 (3–4),* 603–608	2009 Aug
Catechins are bioavailable in men and women drinking black tea throughout the day	Warden, B. A.; Smith, L. S.; Beecher, G. R.; Balentine, D. A.; Clevidence, B. A.	*J. Nutr. 131 (6),* 1731–1737	2001 Jun
Identification and quantification of polyphenol phytoestrogens in foods and human biological fluids	Wilkinson, A. P.; Wähälä, K.; Williamson, G.	*J. Chromatogr., B: Anal. Technol. Biomed. Life Sci. 777 (1–2),* 93–109	2002 Sep 25
Bioavailability and pharmacokinetics of caffeoylquinic acids and flavonoids after oral administration of Artichoke leaf extracts in humans	Wittemer, S. M.; Ploch, M.; Windeck, T.; Müller, S. C.; Drewelow, B.; Derendorf, H.; Veit, M.	*Phytomedicine 12 (1–2),* 28–38	2005 Jan
Validated method for the determination of six metabolites derived from artichoke leaf extract in human plasma by high-performance liquid chromatography-coulometric-array detection	Wittemer, S. M.; Veit, M.	*J. Chromatogr., B: Anal. Technol. Biomed. Life Sci. 793 (2),* 367–375	2003 Aug 15
HPLC in natural product analysis: The detection issue	Wolfender, J. L.	*Planta Med. 75 (07),* 719–734	2009 Jun
Simultaneous determination of isoflavones and bisphenol A in rat serum by high-performance liquid chromatography coupled with coulometric array detection	Yasuda, S.; Wu, P. S.; Hattori, E.; Tachibana, H.; Yamada, K.	*Biosci., Biotechnol., Biochem. 68 (1),* 51–58	2004 Jan
Impurities from polypropylene microcentrifuge tubes as a potential source of interference in simultaneous analysis of multiple lipid-soluble antioxidants by HPLC with electrochemical detection	Yen, H. C.; Hsu, Y. T.	*Clin. Chem. Lab. Med. 42 (4),* 390–395	2004 Apr

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Title	Authors	Publication	Publication Date
Simultaneous determination of triterpenoid saponins from *pulsatilla koreana* using high performance liquid chromatography coupled with a charged aerosol detector (HPLC-CAD)	Yeom, H.; Suh, J. H.; Youm, J. R.; Han, S. B.	*Bull. Korean Chem. Soc. 31 (5),* 1159–1164	2010
DPPH radical scavenging activities of 31 flavonoids and phenolic acids and 10 extracts of Chinese materia medica	Yuan, Y.; Chen, C.; Yang, B.; Kusu, F.; Kotani, A.	*Zhongguo Zhongyao Zazhi 34 (13),* 1695–1700	2009 Jul
Determination of residual clenbuterol in pork meat and liver by HPLC with electrochemical detection	Zhang, X. Z.; Gan, Y. R.; Zhao, F. N.	*Yaoxue Xuebao 39 (4),* 276–280	2004 Apr
Identification of equol producers in a Japanese population by high-performance liquid chromatography with coulometric array for determining serum isoflavones	Zhao, J. H.; Sun, S. J.; Arao, Y.; Oguma, E.; Yamada, K.; Horiguchi, H.; Kayama, F.	*Phytomedicine 13 (5),* 304–309	2006 May
Simultaneous sampling of volatile and non-volatile analytes in beer for fast fingerprinting by extractive electrospray ionization mass spectrometry	Zhu, L.; Hu, Z.; Gamez, G.; Law, W. S.; Chen, H.; Yang, S.; Chingin, K.; Balabin, R. M.; Wang, R.; Zhang, T.; Zenobi, R.	*Anal. Bioanal. Chem. 398 (1),* 405–413	2010 Sep
Comparison of various easy-to-use procedures for extraction of phenols from apricot fruits	Zitka, O.; Sochor, J.; Rop, O.; Skalickova, S.; Sobrova, P.; Zehnalek, J.; Beklova, M.; Krska, B.; Adam, V.; Kizek, R.	*Molecules 16 (4),* 2914–2936	2011 Apr 4

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Title	Authors	Publication	Publication Date
Development of analytical procedures to study changes in the composition of meat phospholipids caused by induced oxidation	Cascone, A.; Eerola, S.; Ritieni, A.; Rizzo, A.	*J. Chromatogr., A 1120 (1–2),* 211–220	2006 Jul 7
Evaporative light scattering and charged aerosol detector.	Chaminade, P.	Chapter 5. In *Hyphenated and Alternative Methods of Detection in Chromatography,* Chromatographic Science Series; Shalliker, A., Ed.; Taylor & Francis Group, LLC: Boca Raton, FL.; 145–160	2012
Simple and efficient profiling of phospholipids in phospholipase D-modified soy lecithin by HPLC with charged aerosol detection	Damnjanovic´, J.; Nakano, H.; Iwasaki, Y.	*J. Am. Oil Chem. Soc. 90 (7),* 951–957	2013 Jul
Discriminating olive and non-olive oils using HPLC-CAD and chemometrics	de la Mata-Espinosa, P.; Bosque-Sendra, J. M.; Bro, R.; Cuadros-Rodríguez, L.	*Anal. Bioanal. Chem. 399 (6),* 2083–2092	2011 Feb
Olive oil quantification of edible vegetable oil blends using triacylglycerols chromatographic fingerprints and chemometric tools	de la Mata-Espinosa, P.; Bosque-Sendra, J. M.; Bro, R.; Cuadros-Rodríguez, L.	*Talanta 85 (1),* 177–182	2011 Jul 15
Quantification of triacylglycerols in olive oils using HPLC-CAD	de la Mata-Espinosa, P.; Bosque-Sendra, J.; Cuadros-Rodriguez, L.	*Food Analytical Methods 4 (4), 574–581*	2011 Dec
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Squalene emulsions for parenteral vaccine and drug delivery	Fox, C. B.	*Molecules 14 (9),* 3286–3312	2009 Sep 1
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Composition analysis of positional isomers of phosphatidylinositol by high-performance liquid chromatography	Iwasaki, Y.; Masayama, A.; Mori, A.; Ikeda, C.; Nakano, H.	*J. Chromatogr., A 1216 (32),* 6077–6080	2009 Aug 7
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Title	Authors	Publication	Publication Date
Rapid quantification of yeast lipid using microwave-assisted total lipid extraction and HPLC-CAD	Khoomrung, S.; Chumnanpuen, P.; Jansa-Ard, S.; Ståhlman, M.; Nookaew, I.; Borén, J.; Nielsen, J.	*Anal. Chem. 85 (10),* 4912–4919	2013 May 21
A new liquid chromatography method with charge aerosol detector (CAD) for the determination of phospholipid classes. Application to milk phospholipids	Kieibowicz, G.; Micek, P.; Wawrzenczyk, C.	*Talanta 105,* 28–33	2013 Feb 15
An LC method for the analysis of phosphatidylcholine hydrolysis products and its application to the monitoring of the acyl migration process	Kiełbowicz, G.; Smuga, D.; Gładkowski, W.; Chojnacka, A.; Wawrzeńczyk, C.	*Talanta 94,* 22–29	2012 May 30
Separation of acylglycerols, FAME and FFA in biodiesel by size exclusion chromatography	Kittirattanapiboon, K.; Krisnangkurá, K.	*Eur. J. Lipid Sci. Technol. 110 (5),* 422–427	2008 Mar 17
Quantitation of triacylglycerols from plant oils using charged aerosol detection with gradient compensation	Lísa, M.; Lynen, F.; Holčapek, M.; Sandra, P.	*J. Chromatogr., A. 1176 (1–2),* 135–142	2007 Dec 28
Quantitative study of the stratum corneum lipid classes by normal phase liquid chromatography: comparison between two universal detectors	Merle, C.; Laugel, C.; Chaminade, P.; Baillet-Guffroy, A.	*J. Liq. Chromatogr. Relat. Technol. 33,* 629–644	2010 Mar
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Determination of intralumenal individual bile acids by HPLC with charged aerosol detection	Vertzoni, M.; Archontaki, H.; Reppas, C.	*J. Lipid Res. 49 (12),* 2690–2695	2008 Dec
Neurolipids and the use of a charged aerosol detector	Waraska, J.; Acworth, I.	*Am. Biotechnol. Lab. 26 (1),* 12–13	2008

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AB 119	UV	Rapid Separation of Paclitaxel and Related Compounds in Paclitaxel Injection
AB 134	MS	LC-MS Analysis of Anthocyanins in Bilberry Extract
AB 139	UV	Separation of Schizandrin, Schizandrin A, and Schizandrin B in a Tablet Sample
AB 153	UV	Save the Flavor – Robust Iso-α-Acids Assaying in Beer within Ten Minutes
AB 155	UV	Monitor the Brewing Process with LC-Transformation of Hop alpha-Acids into Beer Iso-alpha-Acids
AN 109	FLD	Determination of Glyphosate by Cation-Exchange Chromatography with Postcolumn Derivatization
AN 156	UV	The Everlasting Paradigm-Keep Beer Tradition or Prevent Beer from a Skunky Off-Flavor?
AN 196	FLD	Determination of Polycyclic Aromatic Hydrocarbons (PAHs) in Edible Oils by Donor-Acceptor Complex Chromatography (DACC)-HPLC with Fluorescent Detection
AN 207	UV	Chromatographic Fingerprinting of *Flos Chrysanthema indici* Using HPLC
AN 213	UV/FLD	Determination of Polycyclic Aromatic Hydrocarbons (PAHs) in Tap Water Using on-Line Solid-Phase Extraction Followed by HPLC with UV and Fluorescence Detections
AN 216	UV	Determination of Water- and Fat-Soluble Vitamins in Functional Waters by HPLC with UV-PDA Detection
AN 224	UV	Determination of Melamine in Milk Powder by Reversed-Phase HPLC with UV Detection
AN 232	UV	Determination of Anthraquinones and Stilbenes in Giant Knotweed Rhizome by HPLC with UV Detection
AN 236	UV	Determination of Iodide and Iodate in Seawater and Iodized Table Salt by HPLC-UV Detection
AN 245	UV	Fast Analysis of Dyes in Foods and Beverages
AN 251	UV	Determination of Water- and Fat-Soluble Vitamins in Nutritional Supplements by HPLC with UV Detection
AN 252	UV	HPLC Assay of Water-Soluble Vitamins, Fat-Soluble Vitamins, and a Preservative in Dry Syrup Multivitamin Formulation
AN 261	UV	Sensitive Determination of Microcystins in Drinking and Environmental Waters
AN 264	UV	Fast Determination of Anthocyanins in Pomegranate Juice
AN 266	FLD	Determination of Sialic Acids Using UHPLC with Fluorescence Detection
AN 272	FLD	Faster Yet Sensitive Determination of N-Methylcarbamates in Rice, Potato, and Corn by HPLC
AN 275	UV	Sensitive Determination of Catechins in Tea by HPLC
AN 287	UV	Two-Dimensional HPLC Combined with On-Line SPE for Determination of Sudan Dyes I–IV in Chili Oil
AN 292	UV	Determination of Aniline and Nitroanilines in Environmental and Drinking Waters by On-Line SPE
AN 293	CAD and UV	Steviol Glycoside Determination by HPLC with Charged Aerosol and UV Detections Using the Acclaim Trinity P1 Column
AN 299	UV	HPLC Analysis of Six Active Components of *Caulis Ionicerae* Using a Phenyl-1 Column
AN 1008	UV	Determination of Nitidine Chloride, Toddalolactone, and Chelerythrine Chloride by HPLC

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AN 1020	EC, UV	Chalcinoids and Bitter Acids in Beer by HPLC with UV and ECD
AN 1023	UV	Determination of Sudan Dyes I–IV in Curry Paste
AN 1026	CAD	Fatty Acid Esters at Low Nanogram Levels
AN 1027	CAD	Ginseng
AN 1028	CAD	Ginkgo biloba
AN 1029	CAD	Black Cohosh
AN 1030	CAD	Soy Saponins
AN 1032	CAD	Unsaturated Fatty Acid: Arachidonic, Linoleic, Linolenic and Oleic Acids
AN 1033	CAD	Corn Syrup
AN 1034	CAD	Honey Sugars
AN 1035	CAD	Phenolic Acids
AN 1036	CAD	Water-Soluble Antioxidants: Ascorbic Acid, Glutathione and Uric Acid
AN 1037	CAD	Artificial Sweeteners-Global Method
AN 1039	CAD	Simultaneous Measurement of Glycerides (Mono-, Di- and Triglycerides) and Free Fatty Acids in Palm Oil
AN 1040	CAD	Analysis of Commercially Available Products Containing Stevia
AN 1041	CAD	Phytosterols
AN 1042	UV	Rapid Separation of Anthocyanins in Cranberry and Bilberry Extracts Using a Core-Shell Particle Column
AN 1045	UV	Determination of Phthalates in Drinking Water by UHPLC with UV Detection
AN 1046	UV	Determination of Phenylurea Compounds in Tap Water and Bottled Green Tea
AN 1055	CAD	Determination of Virginiamycin, Erythromycin, and Penicillin in Dried Distillers Grains with Solubles
AN 1063	ECD	Targeted Analyses of Secondary Metabolites in Herbs, Spices, and Beverages Using a Novel Spectro-Electro Array Platform
AN 1064	ECD	Product Authentication and Adulteration Determination Using a Novel Spectro-Electro Array Platform
AN 1067	UV	Determination of Carbendazim in Orange Juice
AN 1069	UV	Two-Dimensional HPLC Determination of Water-Soluble Vitamins in a Nutritional Drink
AN 1070	UV	Determination of Inositol Phosphates in Dried Distillers Grains and Solubles
AN 20583	UV	Determination of Catechins and Phenolic Acids in Red Wine by Solid Phase Extraction and HPLC
AN 20610	UV	Fast Analysis of Coffee Bean Extracts Using a Solid Core HPLC Column
AN 20663	CAD	Comparative Analysis of Cooking Oils Using a Solid Core HPLC Column
AN 20847	CAD	Analysis of a Sports Beverage for Electrolytes and Sugars Using Multi-Mode Chromatography with Charged Aerosol Detection

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Product Number	Technique	Title
AN 70158	CAD	Novel Universal Approach for the Measurement of Natural Products in a Variety of Botanicals and Supplements
AN 70277	CAD	Simultaneous Analysis of Glycerides and Fatty Acids in Palm Oil
AU 144	UV	Determination of Hexavalent Chromium in Drinking Water Using Ion Chromatography
AU 170	UV	Fast Determination of Vanillin and its Synthesis Precursor by HPLC
AU 182	CAD	Measuring Lactose in Milk: A Validated Method
AU 184	CAD, UV	Mogroside V Determination by HPLC with Charged Aerosol and UV Detections
CAN 106	UV	Determination of the Punicalagins Found in Pomegranate by High Performance Liquid Chromatography
CAN 111	CAD	Determination of Triterpenes in *Centella asiatica* (Gotu Kola) by HPLC-CAD
CAN 112	CAD	Determination of Ginsenosides in Panax ginseng by HPLC-CAD
CAN 115	FLD	Clean-Up and Analysis of Aflatoxins and Ochratoxin A in Herbs and Spices
LPN 2062	MS	Profiling Analysis of 15 Prominent Naturally Occurring Phenolic Acids by LC-MS
LPN 2069	FLD	Fast and Effective Determination of Aflatoxins in Grains or Food Using Accelerated Solvent Extraction followed by HPLC
LPN 2421	UV	Achieving Maximum Productivity by Combining UHPLC with Advanced Chromatographic Techniques
LPN 2818	CAD	Analysis of Fat-Soluble Vitamins and Antioxidants in Supplements by RP-HPLC
LPN 2870	FLD	Benefits of High-Speed Wavelength Switching in UHPLC Methods Using Fluorescence Detection
LPN 2930	CAD	Determination of the Composition of Natural Products by HPLC with Charged Aerosol Detection
LPN 2923	CAD	Simple and Direct Analysis of Falcarinol and Other Polyacetylenic Oxylipins in Carrots by Reversed-Phase HPLC and Charged Aerosol Detection
LPN 2931	CAD	Quantification of Underivatized Omega-3 and Omega-6 Fatty Acids in Foods by HPLC CAD
LPN 2932	ECD	A Versatile Detector for the Sensitive and Selective Measurement of Numerous Fat-Soluble Vitamins and Antioxidants in Human Plasma and Plant Extracts
LPN 2934	CAD	Sensitive Analysis of Commonly Used Artificial and Natural Sweeteners Including Stevia and Their Impurities and Degradation Products
LPN 2991	CAD	Evaluation of Methods for the Characterization and Quantification of Polysorbates and Impurities Along with Other Surfactants and Emulsifiers Used in the Food and Pharmaceutical Industries
PN 70026	CAD	Carbohydrate Analysis Using PAD, FLD, CAD and MS Detectors
PN 70037	CAD	Sensitive HPLC Method for Triterpenoid Analysis Using Charged Aerosol Detection with Improved Resolution
PN 70055	CAD	Direct Analysis of Surfactants using HPLC with Charged Aerosol Detection
PN 70138	UV	Rapid Determination of Polyphenol Antioxidants in Green Tea and Cranberry Extract Using Core Shell Columns
PN 70538	CAD	Analysis of Silicone Oils by HPLC-CAD
PN 70540	CAD, ECD	Profiling *Hoodia* Extracts by HPLC with CAD, ECD, Principal Component Analysis

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Product Number	Technique	Title
AB 127	IC-PAD	Determination of Carbohydrates in Fruit Juice Using Capillary High-Performance Anion-Exchange Chromatography
AB 135	IC-SC	Determination of Anions and Organic Acids in Brewed Coffee Samples Using Capillary IC
AB 137	IC-SC	Determination of Inorganic and Organic Acids in Apple and Orange Juice Samples Using Capillary IC
AN 25	IC-SC	Determination of Inorganic Ions and Organic Acids in Non-Alcoholic Carbonated Beverages
AN 37	IC-PAD	Determination of Iodide and Iodate in Soy- and Mil-Based Infant Formulas
AN 46	IC-PAD	Ion Chromatography: A Versatile Technique for the Analysis of Beer
AN 54	IC-PAD	Determination of Total and Free Sulfite in Foods and Beverages
AN 67	IC-PAD	Determination of Plant-Derived Neutral Oligo- and Polysaccharides
AN 81	IC-SC	Ion Chromatographic Determination of Oxyhalides and Bromide at Trace Level Concentrations in Drinking Water Using direct Injection
AN 82	IC-PAD	Analysis of Fruit Juice Adulterated with Medium Invert Sugar from Beets
AN 87	IC-PAD	Determination of Sugar Alcohols in Confections and Fruit Juices by High-Performance Anion-Exchange Chromatography with Pulsed Amperometric Detection
AN 101	IC-SC	Trace Level Determination of Bromate in Ozonated Drinking Water Using Ion Chromatography
AN 112	IC-UV	Determination of Nitrate and Nitrite in Meat Using High-Performance Anion-Exchange Chromatography
AN 121	IC-SC	Analysis of Low Concentrations of Perchlorate in Drinking Water and Ground Water by Ion Chromatography
AN 123	IC-SC	Determination of Inorganic Anions and Organic Acids in Fermentation Broths
AN 133	IC-SC	Determination of Inorganic Anions in Drinking Water by Ion Chromatography
AN 136	IC-SC and IC-UV	Determination of Inorganic Oxyhalide Disinfection Byproduct Anions and Bromide in Drinking Water Using Ion Chromatography with the Addition of a Postcolumn Reagent for Trace Bromate Analysis
AN 140	IC-SC	Fast Analysis of Anions in Drinking Water by Ion Chromatography
AN 143	IC-SC	Determination of Organic Acids in Fruit Juices
AN 149	IC-SC	Determination of Chlorite, Bromate, Bromide, and Chlorate in Drinking Water by Ion Chromatography with an On-Line-Generated Postcolumn Reagent for Sub-µg/L Bromate Analysis
AN 150	IC-PAD	Determination of Amino Acids in Cell Cultures and Fermentation Broths
AN 154	IC-SC	Determination of Inorganic Anions in Environmental Waters Using a Hydroxide-Selective Column
AN 155	IC-PAD	Determination of Trans-Galactooligosaccharides in Foods by AOAC Method 2001.02

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Product Number	Technique	Title
AN 165	IC-SC	Determination of Benzoate in Liquid Food Products by Reagent-Free Ion Chromatography
AN 167	IC-SC	Determination of Trace Concentrations of Oxyhalides and Bromide in Municipal and Bottled Waters Using a Hydroxide-Selective Column with a Reagent-Free Ion Chromatography System
AN 168	IC-UV	Determination of Trace Concentrations of Disinfection By-Product Anions and Bromide in Drinking Water Using Reagent-Free Ion Chromatography Followed by Postcolumn Addition of Iol-Dianisidine for Trace Bromate Analysis
AN 169	IC-SC	Rapid Determination of Phosphate and Citrate in Carbonated Soft Drinks Using a Reagent-Free Ion Chromatography System
AN 172	IC-SC	Determination of Azide in Aqueous Samples by Ion Chromatography with Suppressed Conductivity Detection
AN 173	IC-PAD	Direct Determination of Cyanide in Drinking Water by Ion Chromatography with Pulsed Amperometric Detection (PAD)
AN 178	IC-SC	Improved Determination of Trace Concentrations of Perchlorate in Drinking Water Using Preconcentration with Two-Dimensional Ion Chromatography and Suppressed Conductivity Detection
AN 182	IC-SC and IC-PAD	Determination of Biogenic Amines in Alcoholic Beverages by Ion Chromatography with Suppressed Conductivity and Integrated Pulsed Amperometric Detections
AN 183	IC-SC and IC-PAD	Determination of Biogenic Amines in Fermented and Non-Fermented Foods Using Ion Chromatography with Suppressed Conductivity and Integrated Pulsed Amperometric Detections
AN 187	IC-SC	Determination of sub-µg/L Bromate in Municipal Waters Using Preconcentration with Two-Dimensional Ion Chromatography and Suppressed Conductivity Detection
AN1 88	IC-PAD	Determination of Glycols and Alcohols in Fermentation Broths Using Ion-Exclusion Chromatography and Pulsed Amperometric Detection
AN 197	IC-PAD	Determination of Glucosamine in Dietary Supplements Using HPAE-PAD
AN 227	ICE-PAD	Determination of Total Cyanide in Municipal Wastewater and Drinking Water Using Ion-Exclusion Chromatography with Pulsed Amperometric Detection (ICE-PAD)
AN 248	IC-PAD	Determination of Lactose in Lactose-Free Milk Products by High-Performance Anion-Exchange Chromatography with Pulsed Amperometric Detection
AN 253	IC-PAD	HPAE-PAD Determination of Infant Formula Sialic Acids
AN 270	IC-PAD	Determination of Hydroxymethylfurfural in Honey and Biomass
AN 273	IC-SC	Determination of Organic Acids in Fruit Juices and Wines by High-Pressure IC
AN 279	IC-SC	Time Savings and Improved Reproducibility of Nitrate and Nitrite Ion Chromatography Determination in Milk Samples
AN 280	IC-PAD	Carbohydrates in Coffee: AOAC Method 995.13 vs a New Fast Ion Chromatography Method
AN 295	IC-SC	Determination of Phytic Acid in Soybeans and Black Sesame Seeds
AN 1007	IC-SC	Determination of Mono-, Di-, and Triphosphates and Citrate in Shrimp by Ion Chromatography

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Product Number	Technique	Title
AN 1044	IC-SC	Determination of Anions in Dried Distillers Grains with Solubles
AN 1068	IC-SC	Determination of Organic Acids in Fruit Juices and Wines by High-Pressure IC
AU 132	IC-UV	Determination of Nitrite and Nitrate in drinking Water by Ion Chromatography with Direct UV Detection
AU 144	IC-UV	Determination of Hexavalent Chromium in Drinking Water Using Ion Chromatography
AU 148	IC-SC	Determination of Perchlorate in Drinking Water Using Reagent-Free Ion Chromatography
AU 150	IC-PAD	Determination of Plant-Derived Neutral Oligo- and Polysaccharides Using the CarboPac PA200
AU 151	IC-PAD	Determination of Sucralose in Reduced- Carbohydrate Colas using High-Performance Anion-Exchange Chromatography with Pulsed Amperometric Detection
AU 189	IC-SC	Determination of Choline in Infant Formula and Other Food Samples by IC
LPN 2982	IC-SC	Determination of Inorganic Anions and Organic Acids in Beverages Using a Capillary IC on a Monolith Anion-Exchange Column
PN 70743	IC-SC	Determination of Perchlorate Levels in Food and Soil Samples Using Accelerated Solvent Extraction and Ion Chromatography
TN 20	IC-PAD	Analysis of Carbohydrates by High-Performance Anion-Exchange Chromatography with Pulsed Amperometric Detection (HPAE-PAD)
TN 126	IC-SC	Determination of Organic Acids in Beer Samples Using a High-Pressure Ion Chromatography System
TN 135	IC-PAD	Determinations of Monosaccharides and Disaccharides in Beverages by Capillary HPAE-PAD

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Methods for extraction and determination of phenolic acids in medicinal plants: a review	Arceusz, A.; Wesolowski, M.; Konieczynski, P.	*Nat. Prod. Commun. 8 (12),* 1821–9	2013 Dec
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Pressurized liquid extraction of medicinal plants	Benthin, B.; Danz, H.; Hamburger, M.	*J. Chromatogr., A. 837 (1-2),* 211–9	1999 Apr
Comparison of the chemical composition of extracts from *Scutellaria lateriflora* using accelerated solvent extraction and supercritical fluid extraction versus standard hot water or 70% ethanol extraction	Bergeron, C.; Gafner, S.; Clausen, E.; Carrier, D. J.	*J. Agric. Food Chem. 53 (8),* 3076–80	2005 Apr
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Extraction of bitter acids from hops and hop products using pressurized solvent extraction (PSE)	Culík, J;. Jurková, M.; Horák, T.; Cejka, P; Kellner, V.; Dvorák, J.; Karásek, P.; Roth, M.	*J. Inst. Brew. 115 (3),* 220–225	2009
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Pressurized liquid extraction prior to liquid chromatography with electrochemical detection for the analysis of vitamin E isomers in seeds and nuts	Delgado-Zamarreño, M. M.; Bustamante-Rangel, M.; Sánchez-Pérez, A.; Carabias-Martínez, R.	*J. Chromatogr., A. 12; 1056 (1-2),* 249–52	2004 Nov

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Pressurized fluid extraction of carotenoids from *Haematococcus pluvialis* and *Dunaliella salina* and kavalactones from *Piper methysticum*	Denery, J. R.; Dragull, K.:Tang, C. S.; Li, Q. X.	*Anal. Chim. Acta. 501 (2),* 175–181	2004 Jan
Development and comparison of two multiresidue methods for the analysis of 17 mycotoxins in cereals by liquid chromatography electrospray ionization tandem mass spectrometry	Desmarchelier, A.; Oberson, J. M.; Tella, P.; Gremaud, E.; Seefelder, W.; Mottier, P.	*J. Agric. Food Chem. 58 (13),* 7510–9	2010 Jul
Identification, extraction and quantification of the synthetic cannabinoid JWH-018 from commercially available herbal marijuana alternatives	Dunham, S. J.; Hooker, P. D.; Hyde, R. M.	*Forensic Sci. Int. 223 (1-3),* 241–4	2012 Nov
Evaluation of polyphenol contents in differently processed apricots using accelerated solvent extraction followed by high-performance liquid chromatography-diode array detector	Erdogan, S.; Erdemoglu, S.	*Int. J. Food Sci. Nutr. 62 (7),* 729–39	2011 Nov
Determination of 2,4,6-trichloroanisole and guaiacol in cork stoppers by pressurised fluid extraction and gas chromatography–mass spectrometry	Ezquerro, Ó.; Garrido-López, Á.; Tena, M. T.	*J. Chromatogr., A. 1102 (12),* 18–24	2006 Jan
Multiwalled carbon nanotubes as matrix solid-phase dispersion extraction absorbents to determine 31 pesticides in agriculture samples by gas chromatography-mass spectrometry	Fang, G.; Min, G.; He, J.; Zhang, C.; Qian, K.; Wang, S.	*J. Agric. Food Chem. 57 (8),* 3040–5	2009 Apr
High-anthocyanin strawberries through cultivar selection	Fredericks, C. H.; Fanning, K. J.; Gidley, M. J.; Netzel, G.; Zabaras, D.; Herrington, M.; Netzel, M.	*J. Sci. Food Agric. 93 (4),* 846–52	2013 Mar
Optimal extraction and fingerprint analysis of *Cnidii fructus* by accelerated solvent extraction and high performance liquid chromatographic analysis with photodiode array and mass spectrometry detections	Gao, F.; Hu, Y.; Ye, X.; Li, J.; Chen, Z.; Fan, G.	*Food Chem. 1 141 (3),* 1962–71	2013 Dec
Simultaneous analysis of seven alkaloids in *Coptis-evodia* herb couple and Zuojin pill by UPLC with accelerated solvent extraction	Gao, X.; Yang, X. W.; Marriott, P. J.	*J. Sep. Sci. 33 (17-18),* 2714–22	2010 Sep
Determination of chromones in *Dysophylla stellata* by HPLC: method development, validation and comparison of different extraction methods	Gautam, R.; Srivastava, A.; Jachak, S. M.	*Nat. Prod. Commun. 5 (4),* 555–8	2010 Apr
Comparison of different extraction techniques for the determination of chlorinated pesticides in animal feed	Gfrerer, M.; Chen, S.; Lankmayr, E.; Xie, Q.; Yang, F.	*Anal. Bioanal. Chem. 378 (7),* 1861–1867	2004
Speciation analysis of selenium compounds in yeasts using pressurised liquid extraction and liquid chromatography–microwave-assisted digestion–hydride generation–atomic fluorescence spectrometry	Gómez-Ariza, J. L.; Caro de la Torre, M. A.; Giráldez, I.; Morales, E.	*Anal. Chim. Acta. 524, (1–2),* 305–314	2004 Oct

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Multianalysis of 35 mycotoxins in traditional Chinese medicines by ultra-high-performance liquid chromatography-tandem mass spectrometry coupled with accelerated solvent extraction	Han, Z.; Ren Y.; Zhu, J.; Cai, Z.; Chen, Y.; Luan, L.; Wu, Y.	*J. Agric. Food Chem. 60 (33),* 8233-47.	2012 Aug
Pressurized liquid extraction-capillary electrophoresis-mass spectrometry for the analysis of polar antioxidants in rosemary extracts	Herrero, M.; Arráez-Román, D.; Segura A.; Kenndler, E.; Gius, B.; Raggid, M. A.; Ibáñez, E.; Cifuentes, A.	*J. Chromatogr., A.1084 (1-2),* 54–62.	2005 Aug
Accelerated solvent extraction of alkylresorcinols in food products containing uncooked and cooked wheat	Holt, M D.; Moreau, R A,.;DerMarderosian, A.; McKeown, N.; Jacques, P. F.	*J. Agric. Food Chem. 60 (19),* 4799–802	2012 May
Application of response surface methodology to optimize pressurized liquid extraction of antioxidant compounds from sage (*Salvia officinalis* L.), basil (*Ocimum basilicum* L.) and thyme (*Thymus vulgaris* L.)	Hossain, M. B.; Brunton, N. P.; Martin-Diana, A. B.; Barry-Ryan, C.	*Food Funct. 1(3),* 269–77	2010 Dec
A review of modern sample-preparation techniques for the extraction and analysis of medicinal plants	Huie, C. W.	*Anal. Bioanal. Chem. 373 (1-2),* 23–30.	2002 May
Polychlorinated dioxins, furans, and biphenyls, and polybrominated diphenyl ethers in a U.S. meat market basket and estimates of dietary intake	Huwe, J. K.; Larsen, G. L.	*Environ. Sci. Technol. 39 (15),* 5606–5611	2005
Study of the effect of sample preparation and cooking on the selenium speciation of selenized potatoes by HPLC with ICP-MS and electrospray ionization MS/MS	Infante, H. G.; Borrego, A. A.; Peachey, E.; Hearn, R.; O'Connor, G.; Barrera, T G.; Ariza, J. L.	*J. Agric. Food Chem. 57(1),* 38–45.	2009 Jan
Pentacyclic triterpene distribution in various plants – rich sources for a new group of multi-potent plant extracts	Jäger, S.; Trojan, H.; Kopp, T.; Laszczyk, M. N.; Scheffler, A.	*Molecules. 14 (6),* 2016–31.	2009 Jun
Comprehensive multiresidue method for the simultaneous determination of 74 pesticides and metabolites in traditional Chinese herbal medicines by accelerated solvent extraction with high-performance liquid chromatography/tandem mass spectrometry	Jia, Z.; Mao, X.; Chen, K.; Wang, K.; Ji S.	*J. AOAC Int.; 93(5),* 1570–88.	2010 Sep-Oct
Gas chromatography-mass spectrometry (GC-MS) method for the determination of 16 European priority polycyclic aromatic hydrocarbons in smoked meat products and edible oils	Jira, W.; Ziegenhals, K.; Speer, K.	*Food Addit. Contam. Part A Chem. Anal. Control Expo. Risk Assess. 25 (6),* 704–13.	2008 Jun
Assessing pressurized liquid extraction for the high-throughput extraction of marine-sponge-derived natural products	Johnson, T. A.; Morgan, M. V.; Aratow, N. A.; Estee, S. A.; Sashidhara, K. V.; Loveridge, S. T.; Segraves, N L.; Crews, P.	*J. Nat. Prod. 73 (3),* 359–64.	2010 Mar
Lipophilic stinging nettle extracts possess potent anti-inflammatory activity, are not cytotoxic and may be superior to traditional tinctures for treating inflammatory disorders	Johnson, T. A.; Sohn, J.; Inman, W. D.; Bjeldanes, L. F.; Rayburn, K.	*Phytomedicine 20(2),* 143–7.	2013 Jan
Effects of solvent and temperature on pressurized liquid extraction of anthocyanins and total phenolics from dried red grape skin	Ju Z. Y.; Howard, L. R.	*J. Agric. Food Chem. 51 (18),* 5207–13.	2003 Aug

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Accelerated solvent extraction of ochratoxin A from rice samples	Juan, C.; González, L.; Soriano, J. M.; Moltó, J. C.; Mañes, J.	*J. Agric. Food Chem. 53 (24),* 9348–9351	2005
Accelerated solvent extraction of paclitaxel and related compounds from the bark of *Taxus cuspidate*	Kawamura, F.; Kikuchi, Y.; Ohira, T.; Yatagai, M.	*J. Nat. Prod. 62 (2),* 244–7.	1999 Feb
Determination of polybromodiphenyl ethers (PBDEs) in milk cream by gas chromatography-mass spectrometry	Kinani, S.; Bouchonnet, S.; Abjean, J.; Campargue, C.	*Food Addit. Contam. Part A Chem. Anal. Control Expo. Risk Assess. 25 (8),* 1007–14	2008 Aug
Determination of isoflavones in soy bits by fast column high-performance liquid chromatography coupled with UV–visible diode-array detection	Klejdus, B.; Mikelová, R.; Petrlová, J.; Potešil, D.; Adam, V.; Stiborová, J.; Hodek, P.; Vacek, J.; Kizek, R.; Kubán, V.	*J. Chromatogr., A. 1084 (1–2), 19,* 71–79	2005 Aug
Accelerated solvent extraction of lignin from *Aleurites moluccana* (candlenut) nutshells	Klein, A. P.; Beach, E. S.; Emerson, J. W.; Zimmerman, J. B.	*J. Agric. Food Chem. 58 (18),* 10045–8	2010 Sep
Application of TLC method with video scanning in estimation of daily dietary intake of specific flavonoids – preliminary studies	Koch, W.; Kukuła-Koch, W.; Marzec, Z.; Marc, D.	*Acta Pol. Pharm. 70 (4),* 611–20	2013 Jul-Aug
Evaluation of a fibrous cellulose drying agent in supercritical fluid extraction and pressurized liquid extraction of diverse pesticides	Lehotay, S. J.; Lee, C. H.	*J. Chromatogr., A.785 (1-2),* 313–27	1997 Oct
Application of accelerated solvent extraction to the investigation of saikosaponins from the roots of *Bupleurum falcatum*	Li, W.; Liu, Z.; Wang, Z.; Chen, L.; Sun, Y.; Hou, J.; Zheng, Y.	*J. Sep. Sci. 33 (12),* 1870–6	2010 Jun
Applicability of accelerated solvent extraction for synthetic colorants analysis in meat products with ultrahigh performance liquid chromatography-photodiode array detection	Liao, Q. G.; Li ,W. H.; Luo, LG.	*Anal. Chim. Acta. 716,* 128–32	2012 Feb
Extraction, isolation, and purification of analytes from samples of marine origin – a multivariate task	Liguori, L.; Bjørsvik, H. R.	*J. Chromatogr. B Analyt. Technol. Biomed. Life Sci. 910,* 46–53	2012 Dec
Investigation on levels of polybrominated diphenyl ethers in retail fish and egg products in Shenzhen	Liu, B.; Zhang, L. S.; Zhang, J. Q.; Jiang, Y. S.; Zhou, J.; Huang, H. Y.	*Zhonghua Yu Fang Yi Xue Za Zhi. 45 (12),* 1068–72	2011 Dec
Characterization of secondary volatile profiles in *Nigella sativa* seeds from two different origins using accelerated solvent extraction and gas chromatography-mass spectrometry	Liu, X.; Abd El-Aty, A. M.; Cho, S. K.; Yang, A.; Park, J. H.; Shim, J. H.	*Biomed. Chromatogr. 26 (10),* 1157–62	2012 Oct
Accelerated solvent extraction of monacolin K from red yeast rice and purification by high-speed counter-current chromatography	Liu, Y.; Guo, X.; Duan, W.; Wang, X.; Du, J.	*J. Chromatogr. B Analyt. Technol. Biomed. Life Sci. 878 (28),* 2881–5	2010 Oct
Multiresidue determination of organophosphorus pesticides in ginkgo leaves by accelerated solvent extraction and gas chromatography with flame photometric detection	Lu, Y.; Yi, X.	*J. AOAC Int. 88 (3),* 729–735	2005

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Influence of sample preparation on assay of phenolic acids from eggplant	Luthria, DL.; Mukhopadhyay, S.	*J. Agric. Food Chem. 54 (1),* 41–47	2006
Pressurised solvent extraction for organotin speciation in vegetable matrices	Marcic, C.; Lespe,s G.; Potin-Gautier, M.	*Anal. Bioanal. Chem. 382 (7),* 1574–83	2005 Aug
Comparison of different methods for the determination of the oil content in oilseeds	Matthäus, B.; Brühl, L.	*J. AOCS 78* 95–102.	2001 Jan
A comparison of automated and traditional methods for the extraction of arsenicals from fish	McKiernan, J. W.; Creed, J. T.; Brockhoff, C. A.; Caruso, J. A.; Lorenzana, R. M.	*J. Anal. At. Spectrom. 14,* 607–613	1999
Subcritical solvent extraction of anthocyanins from dried red grape pomace	Monrad, J. K.; Howard, L. R.; King, J.;, Srinivas, K.; Mauromoustakos, A.	*J. Agric. Food Chem. 58 (5),* 2862–8	2010 Mar
Subcritical solvent extraction of procyanidins from dried red grape pomace	Monrad, J. K.; Howard, L. R.; King, J. W.; Srinivas, K.; Mauromoustakos, A.	*J. Agric. Food Chem.58 (7),* 4014–21	2010 Apr
Pressurized liquid extraction of polar and nonpolar lipids in corn and oats with hexane, methylene chloride, isopropanol, and ethanol	Moreau, R. A.; Powell, M. J.; Singh, V.	*J. Oil Fat Industr. 80 (11),* 1063–1067	2003 Jan
Accelerated solvent extraction for natural products isolation	Mottaleb, M. A.; Sarker, S. D.	*Methods Mol. Biol. 864,* 75–87	2012
Optimization of extraction process for phenolic acids from black cohosh (*Cimicifuga racemosa*) by pressurized liquid extraction	Mukhopadhyay, S,.;Luthria, D. L,.;Robbins, R. J.	*J. Sci. Food Agric. 86 (1),* 156–162, 15	2006 Jan
Anxiolytic activity of a supercritical carbon dioxide extract of *Souroubea sympetala* (Marcgraviaceae)	Mullally, M.; Kramp, K.; Cayer, C.; Saleem, A.; Ahmed, F, McRae, C.; Baker, J.; Goulah, A.; Otorola, M.; Sanchez, P.; Garcia, M.; Poveda, L.; Merali, Z.; Durst, T.; Trudeau, V. L.; Arnason, J. T.	*Phytother. Res. 25 (2),* 264–70	2011 Feb
On-line clean-up of pressurized liquid extracts for the determination of polychlorinated biphenyls in feedingstuffs and food matrices using gas chromatography–mass spectrometry	Müller, A.; Björklund, E.; von Holst, C.	*J. Chromatogr., A. 925 (1–2),* 197–205	2001 Aug
Analysis of multiple herbicides in soybeans using pressurized liquid extraction and capillary electrophoresis	Nemoto, S.; Lehotay, S. J.	*J. Agric. Food Chem.; 46 (6),* 2190–2199	1998
Comparison of sample preparation methods, validation of an UPLC-MS/MS procedure for the quantification of tetrodotoxin present in marine gastropods and analysis of pufferfish	Nzoughet, J. K.; Campbell, K.; Barnes, P.; Cooper, K. M.; Chevallier, O. P, Elliott, C. T.	*Food Chem. 15; 136 (3-4),* 1584–9	2013 Feb
Multiresidue analysis of pesticides in vegetables and fruits using two-layered column with graphitized carbon and water absorbent polymer	Obana, H.; Akutsu, K.; Okihashi, M.; Hori, S.	*The Analyst 123,* 711–714	1998
Analysis of 2-alkylcyclobutanones with accelerated solvent extraction to detect irradiated meat and fish	Obana, H.; Furuta, M.; Tanaka, Y.	*J. Agric. Food Chem. 53 (17),* 6603–8	2005 Aug

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Determination of organophosphorus pesticides in foods using an accelerated solvent extraction system	Obana, H.; Kikuchi, K.; Okihashi, M.; Hori, S.	*Analyst 122 (3),* 217–20	1997 Mar
Pressurized hot water extraction of berberine, baicalein and glycyrrhizin in medicinal plants	Ong, E. S.; Shea Mei, L.	*Anal. Chim. Acta. 482 (1),* 81–89	2003 Apr
Pressurized liquid extraction of berberine and aristolochic acids in medicinal plants	Ong E. S.; Woo S. O.; Yong, Y. K.	*J. Chromatogr., A. 904 (1),* 57–6422	2000 Dec
Rapid determination of pesticide multiresidues in vegetables and fruits by accelerated solvent extraction coupled with online gel permeation chromatography-gas chromatography-mass spectrometry	Ouyang, Y.; Tang, H.; Wu, Y.; Li, G.	*Se Pu. 30(7),* 654–9	2012 Jul
Determination of zearalenone from wheat and corn by pressurized liquid extraction and liquid chromatography-electrospray mass spectrometry	Pallaroni, L.; von Holst, C.	*J. Chromatogr., A. 993,* 39–45	2003
Development of an extraction method for the determination of zearalenone in corn using less organic solvents	Pallaroni, L.; von Holst, C.	*J. Chromatogr., A. 5 1055 (1-2),* 247–9	2004 Nov
Stability of phenolic compounds during extraction with superheated solvents	Palma, M.; Piñeiro, Z.; Barroso, C. G.	*J. Chromatogr., A. 6 921 (2),* 169–74	2001 Jul
Extraction and analysis of trace amounts of cyclonite (RDX) and its nitroso-metabolites in animal liver tissue using gas chromatography with electron capture detection (GC-ECD)	Pan, X.; Zhang, B.; Cobb, G. P.	*Talanta 67 (4),* 816–23	2005 Oct
Simultaneous determination of 405 pesticide residues in grain by accelerated solvent extraction then gas chromatography-mass spectrometry or liquid chromatography-tandem mass spectrometry	Pang, G.; Liu, Y.; Fan, C.; Zhang, J.; Cao, Y.; Li, X.; Li, Z.; Wu, Y.; Guo, T.	*Anal. Bioanal. Chem. 384,* 1366–1408	2006 Mar
Automated sample preparation by pressurized liquid extraction-solid-phase extraction for the liquid chromatographic-mass spectrometric investigation of polyphenols in the brewing process	Papagiannopoulos, M.; Mellenthin, A.	*J. Chromatogr., A. 8 976 (1-2),* 345–8	2002 Nov
Online coupling of pressurized liquid extraction, solid-phase extraction and high-performance liquid chromatography for automated analysis of proanthocyanidins in malt	Papagiannopoulos, M.; Zimmermann, B.; Mellenthin, A.; Krappe, M.; Maio, G.; Galensa, R.	*J. Chromatogr., A. 7 958 (1-2),* 9–16	2002 Jun
Simultaneous determination of 13 quinolones from feeds using accelerated solvent extraction and liquid chromatography	Pecorelli, I.; Galarini, R.; Bibi, R.; Floridi, A. I.; Casciarri, E.; Floridi, A.	*Anal. Chim. Acta. 483 (1-2),* 81–89	2003 April
Comparison of soxhlet, ultrasound-assisted and pressurized liquid extraction of terpenes, fatty acids and Vitamin E from *Piper gaudichaudianum* Kunth	Péres, V. F.; Saffi, J.; Melecchi, M. I.; Abad, F. C.; de Assis Jacques, R.; Martinez, M. M.; Oliveira, E. C.; Caramão, E. B.	*J. Chromatogr., A. 1105 (1-2),* 115–8	2006 Feb
Pressurised fluid extraction (PFE) as an alternative general method for the determination of pesticide residues in rape seed	Pihlström, T.; Isaac, G.; Waldebäck, M.; Osterdahl, B. G.; Markides, K. E.	*Analyst 127 (4),* 554–9	2002 Apr
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An improved clean-up strategy for simultaneous analysis of polychlorinated diben-zo-p-dioxins (PCDD), polychlorinated dibenzofurans (PCDF), and polychlorinated biphenyls (PCB) in fatty food samples	Pirard, C.; Focant, J. F.; De, P. E.	*Anal. Bioanal. Chem. 372 (2),* 373–81.	2002 Jan
Extraction of polar and hydrophobic pollutants using accelerated solvent extraction (ASE)	Pörschmann, J., Plugge, J.	*Fresen. J. Anal. Chem. 364 (7),* 643–645	1999
Quantification of the total amount of artemisinin in leaf samples by thin layer chromatography	Quennoz, M.; Bastian, C.; Simonnet, X.; Grogg, A. F.	*Chimia (Aarau) 64 (10),* 755–7.	2010
Determination of fat in dairy products using pressurized solvent extraction	Richardson, R. K.	*J. AOAC Int. 84 (5),* 1522–1533	2001
Influence of altitudinal variation on the content of phenolic compounds in wild populations of *Calluna vulgaris,* Sambucus nigra, and *Vaccinium myrtillus*	Rieger, G.; Müller, M.; Guttenberger, H.; Bucar, F.	*J. Agric. Food Chem. 56 (19),* 9080–6.	2008 Oct
Pressurized liquid extraction of isoflavones from soybeans	Rostagno, M. A.; Palma, M.; Barroso, C. G.	*Anal. Chim. Acta. 522 (2),* 169–177.	2004 Sep
A multi-residue method for the analysis of organophosphorus residues in cooked and polished rice using accelerated solvent extraction and dispersive-solid phase extraction (D-SPE) technique and uncertainty measurement	Sanyal, D.; Rani, A.; Alam, S.	*J. Environ. Sci. Health, B 44 (7),* 706–16.	2009 Sep
Accelerated solvent extraction of lipids for determining the fatty acid composition of biological material	Schäfer, K.	*Anal. Chim. Acta. 358 (1),* 69–77	1998 Jan
HPLC analysis of kaempherol and quercetin derivatives isolated by different extraction techniques from plant matrix	Skalicka-Wozniak, K.; Szypowski, J.; Głowniak, K.	*J. AOAC Int. 94 (1),* 17–21.	Jan-Feb 2011
Statistical evaluation of fatty acid profile and cholesterol content in fish (common carp) lipids obtained by different sample preparation procedures	Spiric, A.; Trbovic, D.; Vranic, D.; Djinovic, J.; Petronijevic, R.; Matekalo-Sverak, V.	*Anal. Chim. Acta. 672 (1-2), 66–71.*	2010 Jul
Application of accelerated solvent extraction in the analysis of organic contaminants, bioactive and nutritional compounds in food and feed	Sun, H.; Ge, X.; Lv, Y.; Wang, A.	*J. Chromatogr., A. 1237,* 1–23.	2012 May
Development of an accelerated solvent extraction, ultrasonic derivatisation LC-MS/MS method for the determination of the marker residues of nitrofurans in freshwater fish	Tao, Y.; Chen, D.; Wei, H.; Yuanhu, P.; Liu, Z.; Huang, L.; Wang, Y.; Xie, S.; Yuan, Z.	*Food Addit. Contam. Part A Chem. Anal. Control Expo. Risk Assess. 29 (5),* 736–45.	2012
Simultaneous determination of lincomycin and spectinomycin residues in animal tissues by gas chromatography-nitrogen phosphorus detection and gas chroma-tography-mass spectrometry with accelerated solvent extraction	Tao, Y.; Chen, D.; Yu, G,.;Yu, H.; Pan, Y.; Wang, Y.; Huang, L.; Yuan, Z.	*Food Addit. Contam. Part A Chem. Anal. Control Expo. Risk Assess. 28 (2),* 145–54.	2011 Feb
Determination of 17 macrolide antibiotics and avermectins residues in meat with accelerated solvent extraction by liquid chromatography-tandem mass spectrometry	Tao, Y.; Yu, G.; Chen, D.; Pan, Y.; Liu, Z.; Wei, H.; Peng, D.; Huang, L.; Wang, Y.; Yuan, Z.	*J. Chromatogr. B Analyt. Technol. Biomed. Life Sci. 897,* 64–71.	2012 May
Determination of seven toxaphene congeners in ginseng and milkvetch root by gas chromatography tandem mass spectrometry	Tian, S.; Mao, X.; Miao, S.; Jia, Z.; Wang, K.; Ji, S.	*Se Pu. 30 (1),* 14–20.	2012 Jan

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A consecutive preparation method based upon accelerated solvent extraction and high-speed counter-current chromatography for isolation of aesculin from *Cortex fraxinus*	Tong, X.; Zhou, T, Xiao, X.; Li, G.	*J. Sep. Sci. 35 (24),* 3609–14	2012 Dec
Characterization of anthocyanins and anthocyanidins in purple-fleshed sweetpotatoes by HPLC-DAD/ESI-MS/MS	Truong, V. D.; Deighton, N.; Thompson, R. T.; McFeeters, R. F.; Dean, L. O.; Pecota, K. V.; Yencho, G. C.	*J. Agric. Food Chem. 58 (1),* 404–10	2010 Jan
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Analysis of zearalenone in cereal and swine feed samples using an automated flow-through immunosensor	Urraca, J. L.; Benito-Peña, E.; Pérez-Conde, C.; Moreno-Bondi, M. C.; Pestka, J. J.	*J. Agric. Food Chem. 53 (9),* 3338–3344	2005
Accelerated solvent extraction and gas chromatography/mass spectrometry for determination of polycyclic aromatic hydrocarbons in smoked food samples	Wang, G.; Lee, A. S.; Lewis, M.; Kamath, B.; Archer, R. K.	*J. Agric. Food Chem. 47 (3),* 1062–6.	1999 Mar
Subcritical water extraction of alkaloids in *Sophora flavescens* Ait. and determination by capillary electrophoresis with field-amplified sample stacking	Wang, H.; Lu, Y.; Chen, J.; Li, J.; Liu, S.	*J. Pharm. Biomed .Anal. 58,* 146–51.	2012 Jan
Evaluation of Soxhlet extraction, accelerated solvent extraction and microwave-assisted extraction for the determination of polychlorinated biphenyls and polybrominated diphenyl ethers in soil and fish samples	Wang, P.; Zhang, Q.; Wang, Y.; Wang, T.; Li X.; Ding, L.; Jiang, G.	*Anal. Chim. Acta. 663 (1),* 43–8.	2010 Mar
Determination of ten pesticides of pyrazoles and pyrroles in tea by accelerated solvent extraction coupled with gas chromatography-tandem mass spectrometry	Xu, D.; Lu, S.; Chen, D.; Lan, J.; Zhang, Z.; Yang, F.; Zhou, Y.	*Se Pu.; 31 (3),* 218–22.	2013 Mar
Online cleanup of accelerated solvent extractions for determination of adenosine 5'-triphosphate (ATP), adenosine 5'-diphosphate (ADP), and adenosine 5'-monophosphate (AMP) in royal jelly using high-performance liquid chromatography	Xue, X.; Wang, F.; Zhou, J.; Chen, F.; Li, Y.; Zhao, J.	*J. Agric. Food Chem. 57 (11),* 4500–5.	2009 Jun
Identification and quantitation of eleven sesquiterpenes in three species of *Curcuma* rhizomes by pressurized liquid extraction and gas chromatography–mass spectrometry	Yang, F. Q.; Li ,S.; Chen, Y.; Lao, S. C.; Wang, YT.; Dong, T. T. X.; Tsim, K. W. K.	*J. Pharm. Biomed. Anal. 39 (3/4),* 552–558	2005 Sep
Dispersive solid-phase extraction cleanup combined with accelerated solvent extraction for the determination of carbamate pesticide residues in *Radix glycyrrhizae* samples by UPLC-MS-MS	Yang, R. Z.; Wang, J. H.; Wang, M. L.; Zhang, R.; Lu, X. Y.; Liu, W. H.	*J. Chromatogr. Sci. 49 (9),* 702–8.	2011 Oct
Simultaneous determination of amitraz and its metabolite residue in food animal tissues by gas chromatography-electron capture detector and gas chromatography-mass spectrometry with accelerated solvent extraction	Yu, H.; Tao, Y.; Le, T.; Chen, D.; Ishsan, A.; Liu, Y, Wang, Y.; Yuan, Z.	*J. Chromatogr. B Analyt. Technol. Biomed. Life Sci. 878 (21),* 1746–52.	2010 Jul
Simultaneous determination of fluoroquinolones in foods of animal origin by a high performance liquid chromatography and a liquid chromatography tandem mass spectrometry with accelerated solvent extraction	Yu, H.; Tao, Y.; Chen, D.; Pan, Y.; Liu, Z.; Wang, Y.; Huang, L.; Dai, M.; Peng, D.; Wang, X.; Yuan, Z.	*J. Chromatogr. B Analyt. Technol. Biomed. Life Sci. 885-886,* 150–9.	2012 Feb

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