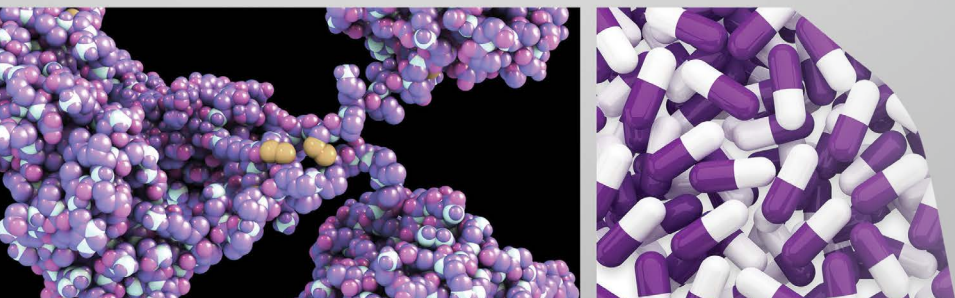


Productivity enhancement with liquid chromatography solutions

Application compendium



Pharma/BioPharma

- Peptide mapping analyses
- Acetaminophen impurities
- Quantification of paclitaxel
- Aggregate analysis of monoclonal antibodies
- Simultaneous analysis of monoclonal antibodies
- Doubling throughput
- Multi-detector platform
- Therapeutic protein mixtures

Food and Beverage

- Water- and fat-soluble vitamins in tablets
- Water-soluble vitamins in a nutritional drink
- Sudan dyes I-IV

Environmental

- Pesticide residues and toxins in drinking water
- Explosive compounds in water
- Polycyclic aromatic hydrocarbons in tap water
- Microcystins in water

Industrial

- Linear alkylbenzene sulfonate

Resources

The collective power of chromatography LC that takes your productivity to new heights

Modern laboratories across all industries are constantly being asked to achieve more with less without compromising data quality. To do this, innovations are needed in scientific instrumentation that will increase throughput, ease quantitative and qualitative sample characterization, and simultaneously improve return on investment.

Thermo Scientific Liquid Chromatography solutions help scientists overcome laboratory resource limitations and increase productivity. The key is the application of dual pump technology for smarter, more efficient workflows without the need for additional bench space.

Two systems that have such dual pump technology are the Thermo Scientific™ Vanquish™ Duo UHPLC System and the Thermo Scientific™ UltiMate™ 3000 Dual Gradient HPLC System. They are designed to help laboratories develop and establish workflows with more efficient use of resources.

Look through this compendium to find application-specific examples showing how to set up fluidic workflows for enhanced productivity that will

- Reduce cost per sample
- Benefit from the robustness built into the instrument platform
- Free up valuable bench space
- Increase sample throughput
- Enhance qualitative and quantitative sample knowledge
- Streamline workflows using intelligent software tools



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Resources

Tandem UHPLC operation for high-throughput LC-MS peptide mapping analyses

Martin Samonig, Sabrina Patzelt, Carsten Paul, Martin Rühl, and Remco Swart
Thermo Fisher Scientific, Germering, Germany

Overview

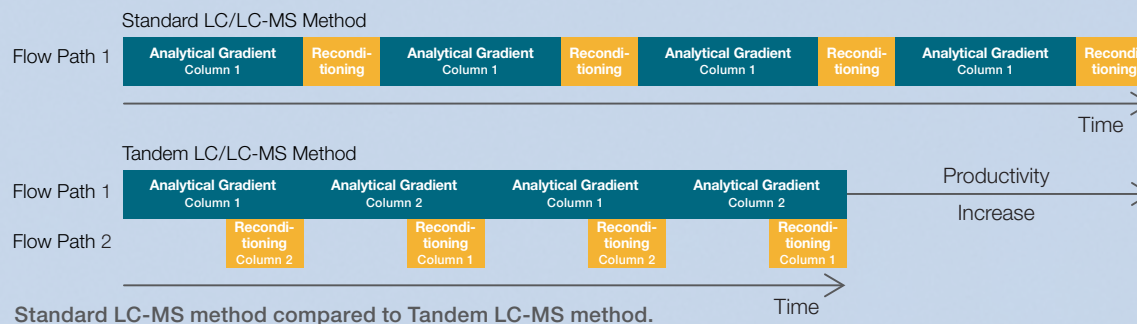
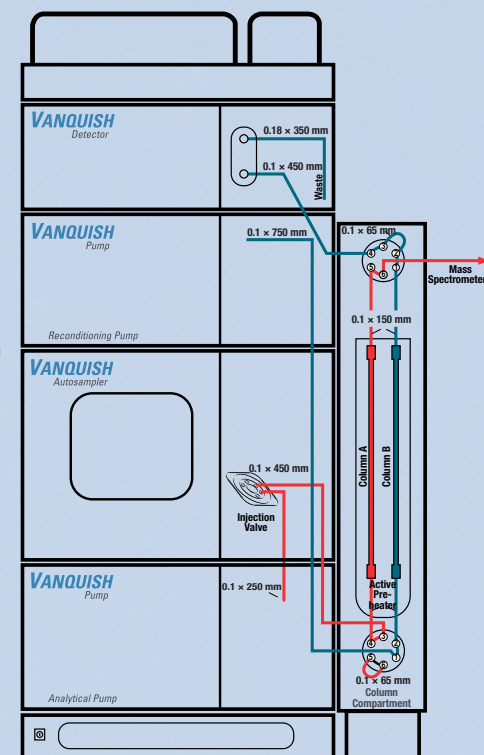
Many UHPLC peptide mapping methods require lengthy periods of column washing and equilibration between injections. To increase throughput and mitigate these delays without changing the chromatographic gradient, a Tandem LC approach with a dual pump setup and column switching capabilities can be implemented. In this setup one column is used for the ongoing separation, while the second column is diverted from the mass spectrometer and simultaneously washed and conditioned for the next injection.

This application note demonstrates how Tandem LC or LC-MS workflows enabled a throughput increase up to 40% without changing the actual gradient of an existing peptide mapping method. The retention time relative standard deviation (RSD) values were below 0.11% for the tandem and single column operation. The Tandem LC setup can be applied to other methods and samples as well.

Instrumentation

The Thermo Scientific Vanquish Horizon Duo UHPLC System for Tandem LC was configured with two binary high-pressure gradient pumps (used as an analytical pump and a reconditioning pump) to run the chromatography workflows in tandem for maximizing the MS detector utilization.

Vanquish Horizon Duo UHPLC System for Tandem LC workflows with 2-position/6-port (2p6p) valve configurations and required fluidic connections. The recommended capillary to connect the LC to individual mass spectrometer depends on the setup and is defined in the Vanquish MS Connection Kit.



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Industrial

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Resources

Simultaneous high-performance and ultra-high-performance liquid chromatographic analysis of acetaminophen impurities using a single instrument

Maria Grübner, Carsten Paul, and Frank Steiner
Thermo Fisher Scientific, Germering, Germany

Overview

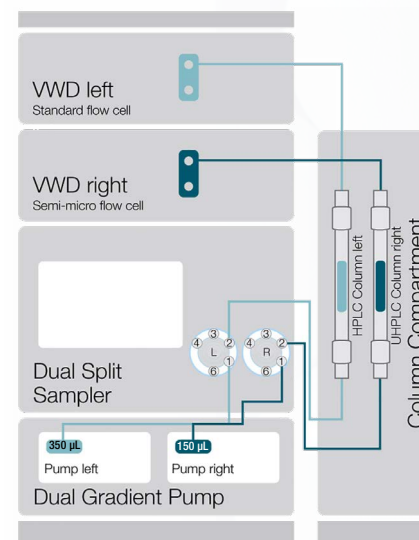
To increase throughput and generate more results in analytical laboratories, there is a growing need for faster methods and new analytical instrumentation. UHPLC-compatible instruments and spatial constraints play an increasing role in equipping such labs. This means LC systems that house two independent LC channels with two separate, individually configurable flow paths in the footprint of a single instrument are beneficial in multiple ways. Such a setup can be utilized for parallel implementation of completely independent HPLC and UHPLC methods and to speed up legacy HPLC methods on the same workstation.

This application note demonstrates how established HPLC methods and their UHPLC counterparts can be successfully run in parallel on the same instrument. In this study, a 2.5-fold throughput increase and savings of up to 80% mobile phase and 60% cycle time were achieved by speeding up an HPLC method for the analysis of acetaminophen as an active pharmaceutical ingredient and its impurities derived from a United States Pharmacopeia (USP) assay using UHPLC conditions.

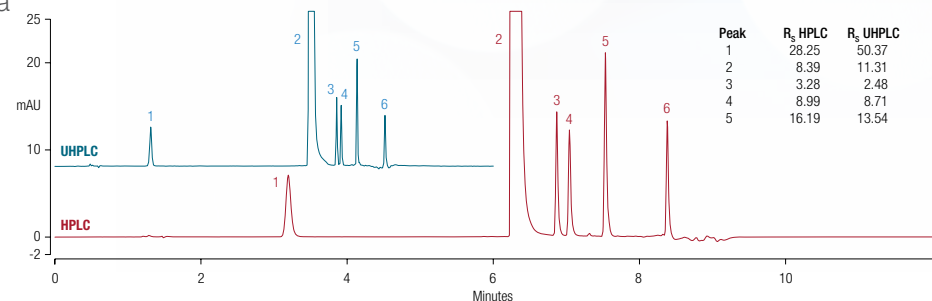
Comparison of HPLC (bottom, red) and UHPLC (top, blue) chromatograms presented at the same time, and signal scale. Differences in peak resolution are shown in the accompanying table.

Instrumentation

The Thermo Scientific™ Vanquish™ Flex Duo System for Dual LC was configured with one HPLC and one UHPLC flow path to run the two separate methods simultaneously on the same instrument.



Fluidic setup of Vanquish Flex Duo System for Dual LC with one HPLC (light blue) and one UHPLC (dark blue) flow path.



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Industrial

- Linear alkylbenzene sulfonate

Resources

Quantification of paclitaxel, its degradants, and related substances using UHPLC with charged aerosol detection

Michael Menz and Frank Steiner, Thermo Fisher Scientific, Germering, Germany
Ian Acworth, Thermo Fisher Scientific, Chelmsford, MA, USA

Overview

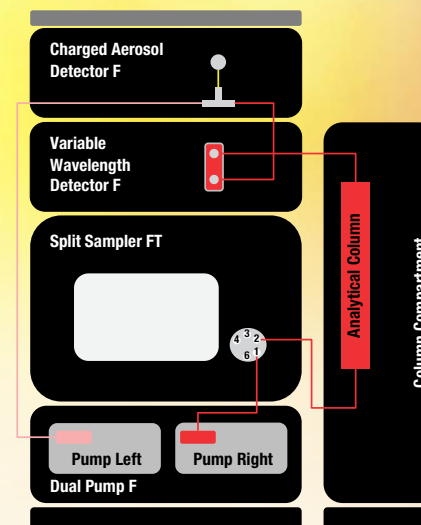
For any drug, unwanted impurities are a key concern during drug development and throughout the product's life cycle. Monitoring these impurities is mandatory to ensure a safe and effective product.

It can be particularly challenging to accurately classify impurities according to guidelines from the International Council on Harmonisation (ICH) with respect to the reporting, identification, or qualification threshold. Charged aerosol detection can be used to overcome these quantitation challenges (e.g., response or detectability issues).¹ The Charged Aerosol Detector (CAD) response is independent of the chemical structure of nonvolatile analytes, making it an ideal chromatographic approach when individual calibrants are unavailable. As CAD response is affected by mobile phase composition, it is necessary to ensure the universal response is applicable in gradient elution. A second gradient, the inverse of the analytical gradient, is applied post column so that the detector always experiences mobile phase of constant composition and response uniformity is maintained.

This application note shows how a successful thermal degradation study was performed using UHPLC-UV-CAD on the drug paclitaxel. The separation of the drug and its related compounds and impurities with single calibrant quantitation was successfully demonstrated. The uniform response of the CAD was shown to be applicable in gradient elution under UHPLC conditions.

Instrumentation

The separation was achieved using a Thermo Scientific[™] Accucore[™] Pentafluorophenyl (PFP) column. Detection was performed using the Vanquish Flex Variable Wavelength Detector followed by the Vanquish Flex Charged Aerosol Detector. The uniform response of the CAD was enabled by setting up the Vanquish Flex Duo UHPLC System for Inverse Gradient.



Fluidic scheme of the Vanquish Duo Inverse Gradient Workflow including a Charged Aerosol Detector F and Variable Wavelength Detector F.

Reference

1. <http://www.ich.org/products/guidelines>; accessed 11/20/2017

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Industrial

- Linear alkylbenzene sulfonate

Resources

High-throughput protein aggregate analysis of monoclonal antibodies using a novel dual-channel UHPLC instrument

Nicola McGillicuddy,¹ Amy Farrell,¹ Sara Carillo,¹ Martin Samonig,² and Jonathan Bones¹

¹National Institute for Bioprocessing Research and Training (NIBRT), Dublin, Ireland

²Thermo Fisher Scientific, Germering, Germany

Overview

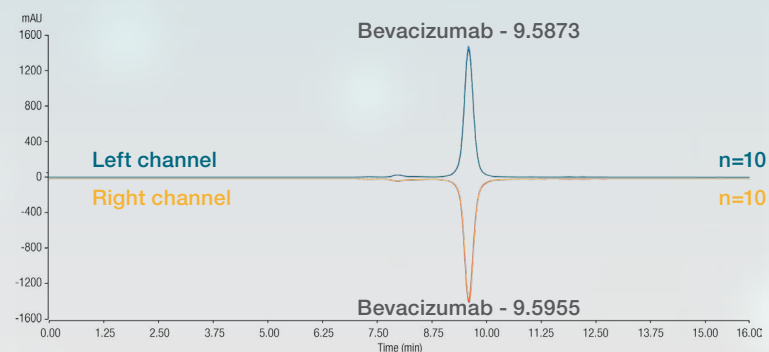
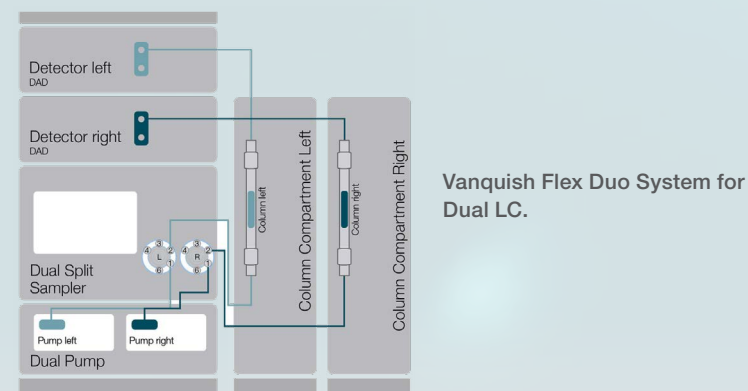
There is an increased need for rapid and robust high-throughput analysis of biotherapeutics due to the increased use of monoclonal antibodies (mAbs) to treat disease. In the biomanufacturing pipeline, handling and storage can cause unintentional size variants of the original product which are potentially harmful. Size-exclusion chromatography (SEC) is considered a standard workflow for monitoring the formation and level of mAb aggregates and fragments, and it is frequently performed in quality control (QC) laboratories.

Standardized chromatographic methods and excellent reproducibility are essential for sample analysis in QC laboratories. Although standard UHPLC systems can analyze samples simply and rapidly, they typically only allow the use of one stationary phase at any given time.

This application note demonstrates how Dual LC provides simple, rapid high-throughput analysis of biotherapeutics with high confidence in results. MAbs were analyzed on a Vanquish Duo System for Dual LC with excellent reproducibility; RSD values were below 1% for a number of analytical parameters, and data was comparable to that obtained on a standard UHPLC system.

Instrumentation

The Vanquish Flex Duo System for Dual LC was used for the high-throughput analysis of an mAb with UV detection. One hundred injections of bevacizumab were performed running the analysis on two identical Thermo Scientific™ MAbPac™ SEC-1 size exclusion columns at the same time on one instrument.



SEC-UV chromatograms mirror plot. Comparison of Vanquish Flex Duo System for Dual LC left and right channels

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Simultaneous analysis of monoclonal antibodies

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- Microcystins in water

Industrial

- Linear alkylbenzene sulfonate

Resources

Simultaneous analysis of monoclonal antibodies using a novel dual-channel UHPLC instrument and orthogonal chromatography

Nicola McGillicuddy,¹ Amy Farrell,¹ Sara Carillo,¹ Martin Samonig,² and Jonathan Bones¹

¹National Institute for Bioprocessing Research and Training (NIBRT), Dublin, Ireland

²Thermo Fisher Scientific, Germering, Germany

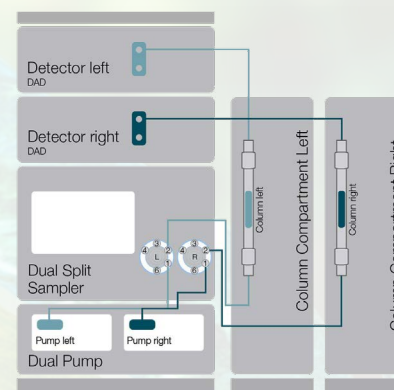
Overview

Standard UHPLC systems can analyze samples simply and rapidly, but typically only one stationary phase can be used at any given time. This is the case even though there are multiple features to monitor and each requires its own method, often using different elution solvents. As a result, analysts are limited in the number of injections that a chromatography system can perform, limiting the efficiency of sample analysis and adding costs.

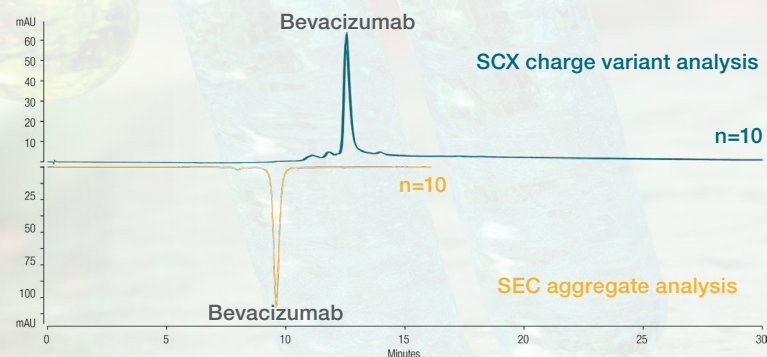
This application note demonstrates how the Vanquish Duo System for Dual LC provides simple and rapid high-throughput analysis of biotherapeutics using two simultaneous LC channels for the same sample. The simultaneous charge variant and aggregate analysis of monoclonal antibodies yielded excellent quality data on both chromatographic channels with high confidence in results. Excellent reproducibility with low % RSD values for a number of analytical parameters was obtained for both chromatographic channels.

Instrumentation

The Vanquish Flex Duo System for Dual LC was used to run two different chromatographic approaches, one on each channel from the same sample.



Vanquish Flex Duo System for Dual LC.



LC-UV chromatograms for the right (blue) and left (orange) LC channel. On the right channel, charge variant analysis of bevacizumab was performed, while on the left channel, a size exclusion intact analysis profile was obtained.

Pharma/BioPharma

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Industrial

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Resources

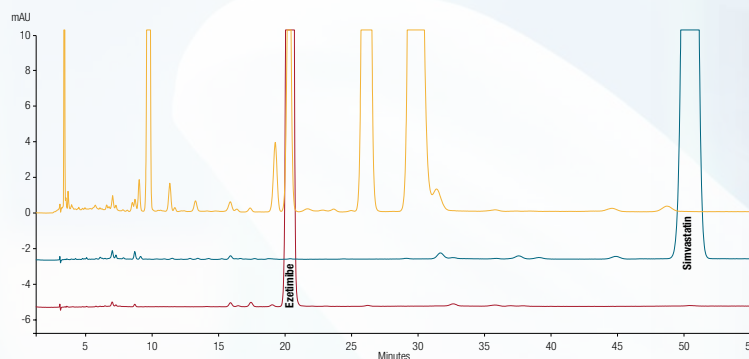
Doubling the throughput of long chromatographic methods by using a novel Dual LC workflow

Sylvia Grosse, Mauro De Pra, and Frank Steiner
Thermo Fisher Scientific, Germering, Germany

Overview

In the pharmaceutical industry, purity analyses of drugs are routinely run for purposes such as batch releases and stability studies. When many samples must be processed, for instance during stability studies, long isocratic methods will decrease the number of samples that can be processed per day, extending the length of studies with obvious cost consequences and tying up lab resources. Reducing the total run time for a method can significantly impact the productivity and success of a lab.

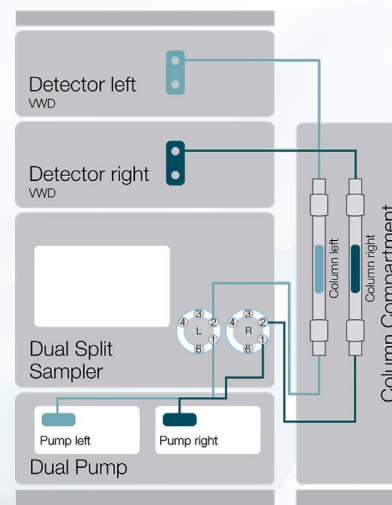
This application note introduces a Dual LC workflow using two separated flow paths on one system. It employs Dual LC for an isocratic stability-indicating method to profile the combined impurities of simvastatin (SMV) and ezetimibe (EZE), two drugs used to reduce cholesterol and triglycerides in blood. The Dual LC workflow enables the simultaneous analysis of two samples by the same instrument, in practice doubling the laboratory throughput within the footprint of one instrument.



Chromatogram overlays of stressed mixture of EZE and SMV (orange); untreated SMV (blue), and untreated EZE (red).

Instrumentation

The Vanquish Flex Duo System for Dual LC was used with a Thermo Scientific™ Hypersil™ GOLD PFP column for the stability indicating method.



Vanquish Flex Duo System for Dual LC.

Pharma/BioPharma

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- Therapeutic protein mixtures

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- Polycyclic aromatic hydrocarbons in tap water
- Microcystins in water

Industrial

- Linear alkylbenzene sulfonate

Resources

A multi-detector platform comprising UV/Vis, charged aerosol, and single quadrupole mass spectrometric detection for comprehensive sample analysis

Stephan Meding, Katherine Lovejoy, Remco Swart, Frank Steiner, and Martin Ruehl
Thermo Fisher Scientific, Germering, Germany

Overview

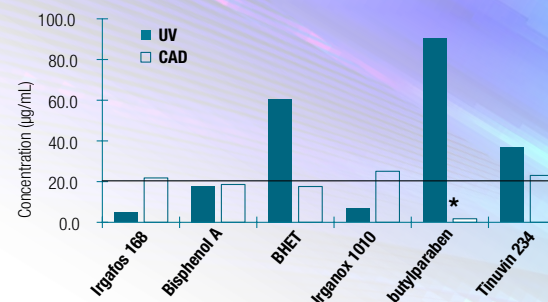
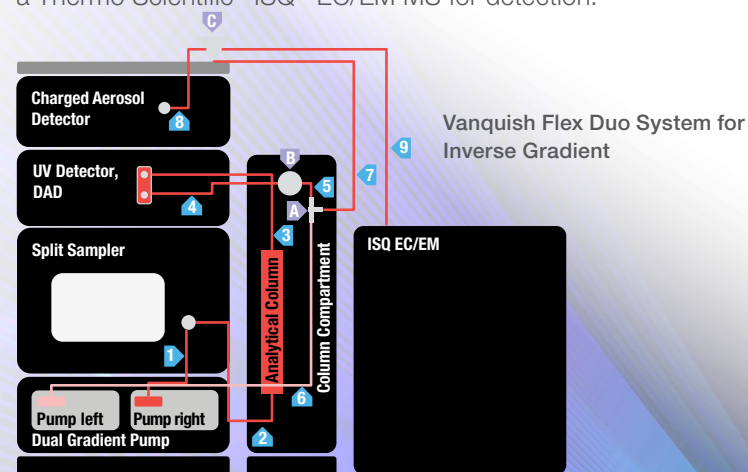
Comprehensive sample analysis with multiple complementary detectors is essential for determining the presence of unknown or unexpected compounds. Beyond mere detection, laboratories also frequently require identity confirmation and quantitation of these compounds to determine their nature and whether they are below acceptable concentration limits. A UV detector offers accurate quantification of chromophore-containing substances if reference standards are available. A CAD delivers universal detection of non- and semi-volatile compounds, making it an ideal complementary detector. Additionally, its near uniform response enables quantification without reference standards. Mass spectrometry offers identity confirmation of detected compounds. These three detection techniques provide a comprehensive sample analysis platform, which can be expanded further by applying two different ionization modes—heated electrospray ionization (HESI) and atmospheric pressure chemical ionization (APCI)—for MS detection.

In this application note, levels of 18 chemicals present in plastic packaging were determined in extracts of cell culture bags. They were analyzed with a UHPLC system in two different configurations for chromatographic analysis—a standard setup and an inverse gradient setup. The CAD and the diode array UV/Vis detector used to determine peak retention times were found to be complementary. The combined detection techniques allowed for determination

of the levels of all 18 standards. Use of a universal calibrant to determine levels of unknown substances was demonstrated with the inverse gradient setup.

Instrumentation

A Vanquish Flex UHPLC System was used in the default setup and extended to a Vanquish Duo System for Inverse Gradient setup for chromatographic analysis with a CAD, UV detector and a Thermo Scientific™ ISQ™ EC/EM MS for detection.



Comparison of quantification of a reinjected 20 µg/mL standard by UV and CAD using a universal calibrant (bisphenol A).

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Industrial

- Linear alkylbenzene sulfonate

Resources

A pre-concentration and online solid phase extraction setup for the LC-MS analysis of therapeutic protein mixtures

Martin Samonig, Sabrina Patzelt, Martin Rühl, and Remco Swart
Thermo Fisher Scientific, Germering, Germany

Overview

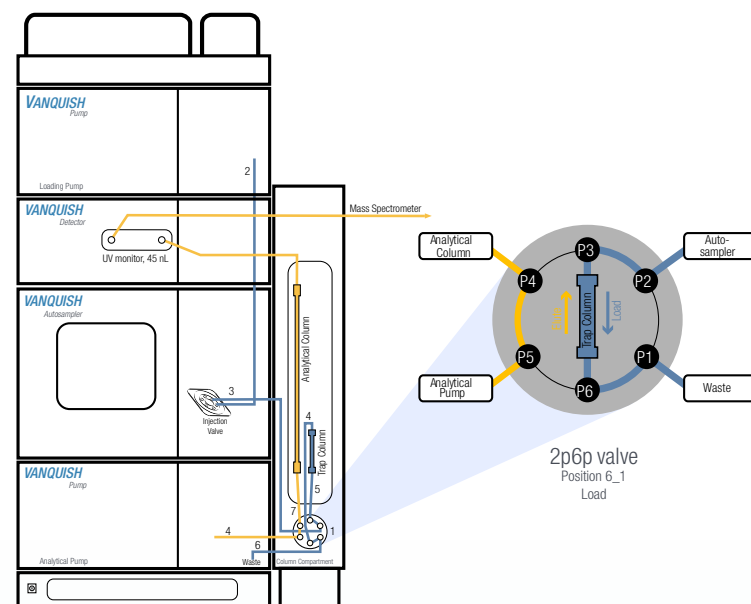
Solid-phase extraction (SPE) is a commonly used laboratory technique to isolate analytes of interest from complex matrices. Because this technique is typically performed manually, it may not satisfy productivity and automation requirements for all laboratories. When repeatability between different analysts shows too much variation or the sample is subject to contamination during the manual process, an automated sample cleanup is preferable.

In the online-SPE approach for the enrichment of low abundant compounds in LC, the sample is pre-concentrated on a trap column prior to chromatographic separation. This technique can be applied for the sample cleanup of protein mixtures often containing high amounts of nonvolatile salts, which are present in various biopharma formulation buffers. The presence of such buffers may interfere with the operation of electrospray ion sources by suppressing ionization.

This technical note shows a method for the separation of a five-protein mixture demonstrating an online SPE setup using a UHPLC system for biopharma samples of medium complexity. The setup can be used for fully automated sample cleanup and enables direct injection of untreated samples. An LC-MS system with single point chromatography data system (CDS) control fulfills GMP/GLP requirements and provides a turnkey solution for fully integrated and automated sample handling.

Instrumentation

A Vanquish Flex UHPLC System was set up for fully automated pre-concentration and sample cleanup, and a Thermo Scientific™ Q Exactive™ HF Mass Spectrometer was used in the detection of intact proteins. This setup used a quaternary pump for sample loading and a binary pump for the analytical gradient.



Vanquish Flex online SPE setup with the detailed 2-position/6-port (2p6p) valve configuration.

Pharma/BioPharma

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Industrial

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Resources

Simultaneous determination of water- and fat-soluble vitamins in tablets and energy drinks by using a novel Vanquish Flex Duo System for Dual LC

Sylvia Grosse, Mauro De Pra, and Frank Steiner
Thermo Fisher Scientific, Germering, Germany

Overview

Vitamins, essential nutrients in food and supplements, can be classified as water-soluble vitamins (WSV) or fat-soluble vitamins (FSV) based on their hydrophobicity.

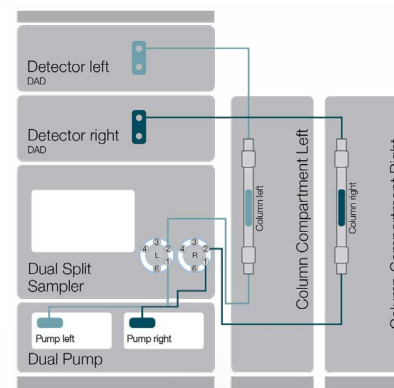
Reversed-phase HPLC is widely used to analyze vitamins in food, supplements, and beverages. Due to the dramatically different hydrophobicity of WSV and FSV, simultaneous LC analysis with the same method is difficult.

This application note introduces an efficient workflow for the simultaneous analysis of WSV and FSV. The workflow is based on the Vanquish Duo System for Dual LC which enables the independent and simultaneous use of two different columns and methods.

Two independent methods were developed and optimized for FSV and WSV and then run simultaneously on the Dual LC system. Compared to previous solutions, the Dual LC setup was remarkably simple to implement, and increased throughput due to the simultaneous use of two columns with two methods for faster analysis.

Instrumentation

The Vanquish Flex Duo System for Dual LC was used with two different chromatographic methods to analyze the two different classes of vitamins at the same time on two flow channels.



Vanquish Flex Duo System for Dual LC.





Pharma/BioPharma

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Sudan dyes I–IV

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Industrial

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Resources

Two-dimensional HPLC determination of water-soluble vitamins in a nutritional drink

Dai Zhenyu, Chen Jing, Xu Qun, and Jeffrey Rohrer
Thermo Fisher Scientific, Shanghai, People's Republic of China

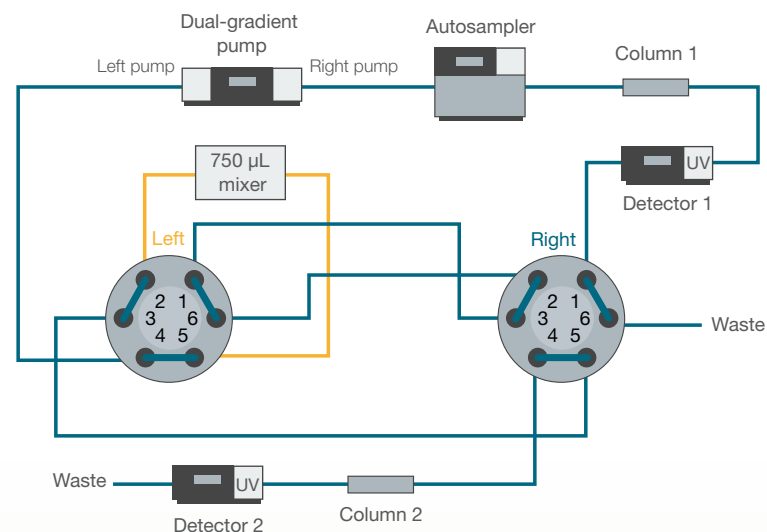
Overview

Poor diets often lack adequate levels of water-soluble vitamins (WSV). Furthermore, as WSV are not retained by the body, people often take WSV supplements in order to prevent illness. A number of reliable QC assays are available to ensure supplements contain the labeled amounts of vitamins. A routine HPLC method will satisfactorily quantify vitamins. However, some supplements, such as vitamin-enhanced milk and nontransparent multivitamin/mineral nutritional drinks, have too many additional components to allow a routine HPLC quantification method. These products may also contain other compounds such as amino acids, minerals, coenzyme Q10, and compounds from grape extracts, which can interfere with the separation of vitamins making quantification difficult.

This application note addresses a simple, sensitive two-dimensional HPLC (2D-HPLC) method to quantify vitamins in complex supplements. Analysis of a complex sample required only off-line filtration because the remainder of the sample preparation was automated by the LC instrument. The application was successful for determination of the B group vitamins in the presence of other nutritional additives. The method was also able to detect but not quantify Vitamin C.

Instrumentation

The Thermo Scientific™ UltiMate™ 3000 Dual-Gradient HPLC System and software automated much of the sample preparation and successfully determined B group vitamins in the presence of other nutritional additives.



UltiMate 3000 Dual Gradient HPLC System for online SPE with the detailed 2-position/6-port (2p6p) valve configuration.





Pharma/BioPharma

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Resources

Determination of Sudan dyes I-IV in curry paste

Supareerk Tukkeeree, Thermo Fisher Scientific, Bangkok, Thailand
 Jeffrey Rohrer, Thermo Fisher Scientific, Sunnyvale, CA, USA

Overview

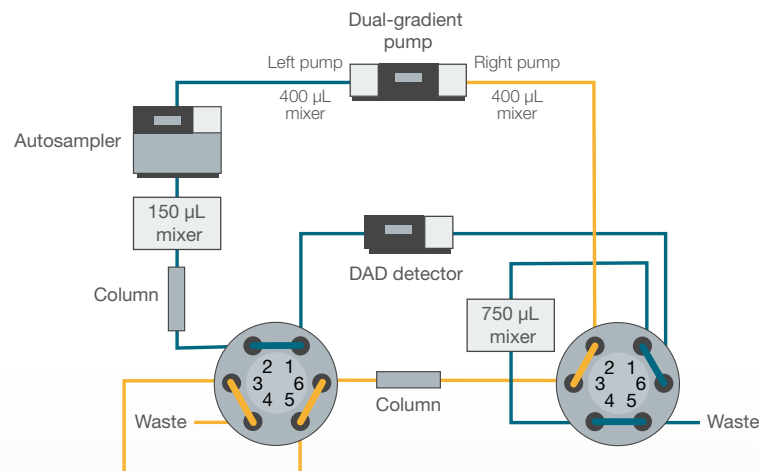
Synthetic Sudan dyes are banned as a food-coloring agent because they are classified as carcinogens. However, for economic reasons they are sometimes used illegally to color food for an improved appearance.

Methods are needed to determine whether food products have been adulterated with Sudan dyes. Typically, Sudan dyes are determined by reversed-phase chromatography with UV or MS detection, but complex food samples usually require rigorous offline sample preparation, such as solvent extraction, solid-phase extraction, sample evaporation, and/or sample reconstitution.

This application note shows a 2D-HPLC approach for determining Sudan dyes I, II, III, and IV in curry paste without the need for extensive offline SPE sample preparation as in other methods. This saves significant time and reduces the cost per analysis. The sample is partially separated in the first dimension, then the portions of the chromatogram containing peaks of interest are sent to the second dimension where they are further resolved. The second dimension uses a column with selectivity different from that used in the first dimension. The total runtime is 25 min. The offline sample preparation step is only a simple sample extraction using acetonitrile followed by filtration.

Instrumentation

This method requires neither an SPE column between the two dimensions nor the third pump to dilute the mobile phase from the first dimension. The same UV detector is used for both dimensions. To determine Sudan dyes in curry paste, only sample extraction and filtration are performed offline. The remaining steps are automated using an UltiMate 3000 Dual Gradient Rapid Separation LC System.



UltiMate 3000 Dual Gradient HPLC System with two-dimensional fluidic setup.

Pharma/BioPharma

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- Polycyclic aromatic hydrocarbons in tap water
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Industrial

- Linear alkylbenzene sulfonate

Resources

Determination of pesticide residues and toxins in drinking water by online SPE – high-performance liquid chromatography

Chinese Applications Team
Thermo Fisher Scientific

Overview

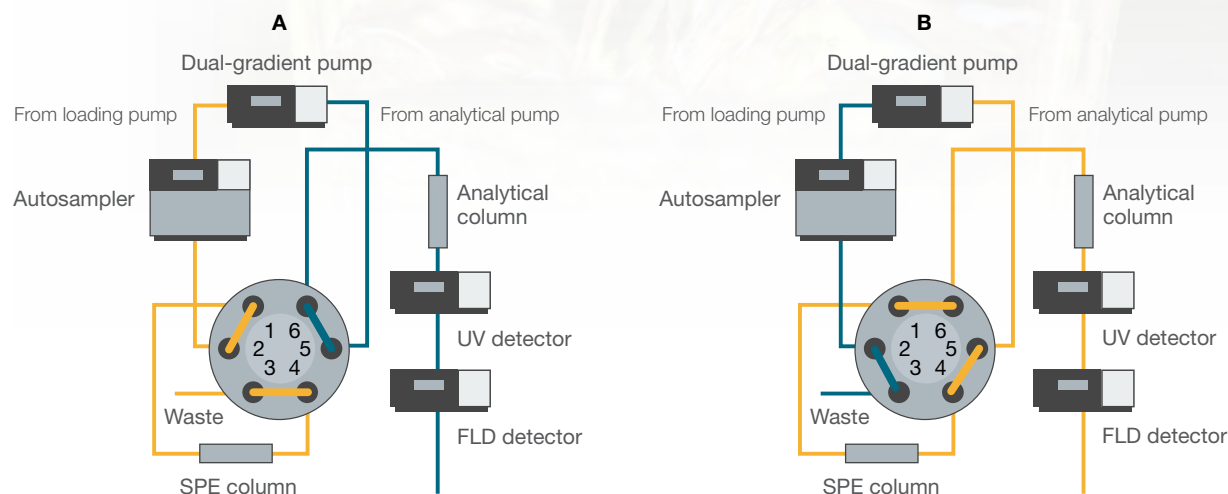
The analysis of drinking water requires the testing of several organic compounds such as those listed the Chinese standard GB/T 5479-20061 or the chemical contaminants list of the United States Environmental Protection Agency (US EPA).

Often, cost-effective optical detectors do not meet the sensitivity requirements for trace detection so extensive sample pretreatment methods are used to reach the required detection limit. The pretreatment is costly, requires time, ties up resources and is analytically less precise with limited repeatability.

This application note shows how online solid phase extraction combined with HPLC offers a simple, fast, and accurate sample pretreatment and analytical approach. The results include automated sample online enrichment, concentration, and matrix elimination up to a 2.5 mL sample volume in the default configuration. This option allows for high efficiency, high sensitivity, and lower analysis costs than traditional methods.

Instrumentation

The Thermo Scientific™ UltiMate™ 3000 Dual Gradient LC System with semi-preparative split-loop autosampler was used.



The UltiMate 3000 Dual-Gradient pump with autosampler and fluorescence detector connected to a six-port switching valve to load, clean, and extract the sample on the solid phase extraction column (A), followed by elution and separation on an analytical column (B)

Pharma/BioPharma

- Peptide mapping analyses
- Acetaminophen impurities
- Quantification of paclitaxel
- Aggregate analysis of monoclonal antibodies
- Simultaneous analysis of monoclonal antibodies
- Doubling throughput
- Multi-detector platform
- Therapeutic protein mixtures

Food and Beverage

- Water- and fat-soluble vitamins in tablets
- Water-soluble vitamins in a nutritional drink
- Sudan dyes I–IV

Environmental

- Pesticide residues and toxins in drinking water
- Explosive compounds in water
- Polycyclic aromatic hydrocarbons in tap water
- Microcystins in water

Industrial

- Linear alkylbenzene sulfonate

Resources

Sensitive determination of explosive compounds in water

Chen Jing, Xu Qun, and Jeffrey Rohrer
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Overview

Residual explosive materials and their degradation products are highly toxic to the environment and can persist in places such as groundwater. From there, the substances can enter tap water and present a risk to human health.

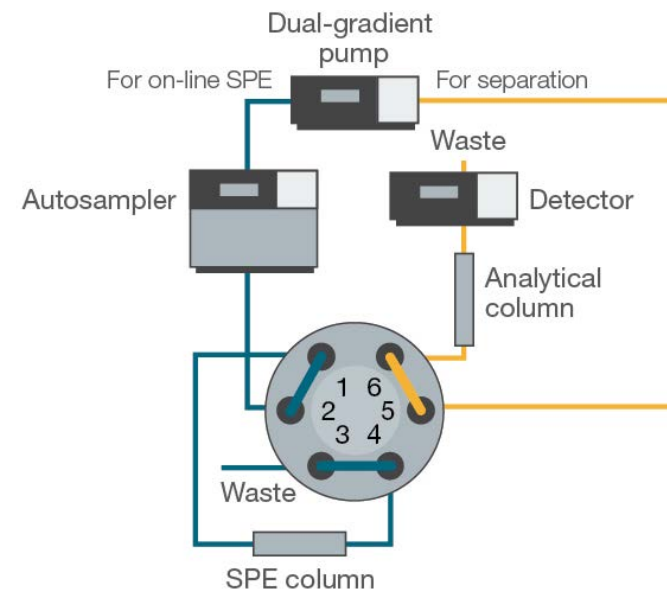
EPA Method 8330A describes an HPLC method with UV detection to determine 14 explosives and related substances in water. This method requires the same sample to be separated on two different columns. Additionally, the method requires the preparation of two groups of solutions containing 2,4-DNT and 2,6-DNT, respectively, when both are to be determined.

The sample preparation procedure for low concentrations of the target compounds in water, which is described in EPA Method 8830A, is a salting-out extraction. This approach is time-consuming, requires large amounts of reagents, and is deficient in terms of process control.

This application note presents an online method that eliminates the salting-out step for the determination of low-level concentrations of the target compounds.

Instrumentation

The analysis was performed on the UltiMate 3000 Dual gradient RSLC System. The use of online solid phase extraction with UV detection eliminated the labor associated with offline extraction and provided a convenient method that achieved reduced method detection limits (MDLs) when determining the residual materials and their byproducts in water.



Flow schematic of online SPE.

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Sensitive and rapid determination of polycyclic aromatic hydrocarbons in tap water

Chen Jing, Dai Zhenyu, Xu Qun, and Liang Lina
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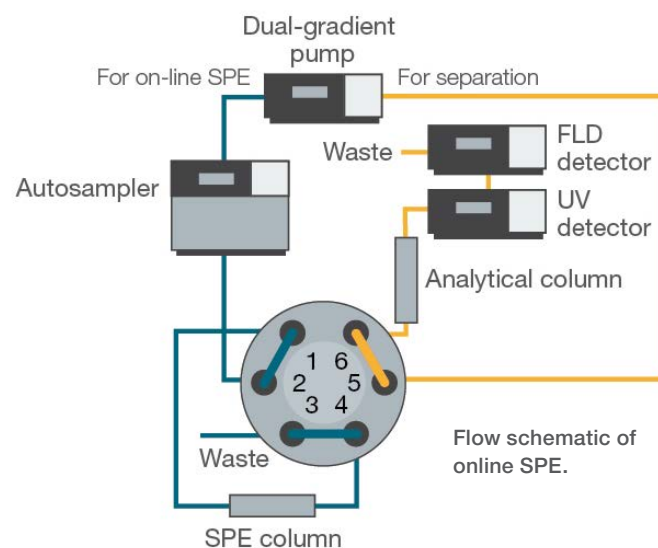
Overview

Polycyclic aromatic hydrocarbons (PAHs) are chemicals formed from the incomplete combustion of organic matter. Due to their potential carcinogenic and mutagenic properties, most countries have regulations limiting the concentrations of a variety of PAHs in drinking water, food additives, cosmetics, workplaces, and factory emissions.

This application note describes an online solid phase extraction HPLC method with UV absorbance and fluorescence detection for rapid and sensitive determination of 20 PAHs in tap water. The reduced MDLs for UV and fluorescence detection provide a convenient method for determining these compounds in drinking and environmental waters using HPLC.

Instrumentation

The determination was performed on an UltiMate 3000 Dual Gradient LC System combined with a Hypersil Green PAH analytical column. Analytes were determined by UV and fluorescence detection in series.



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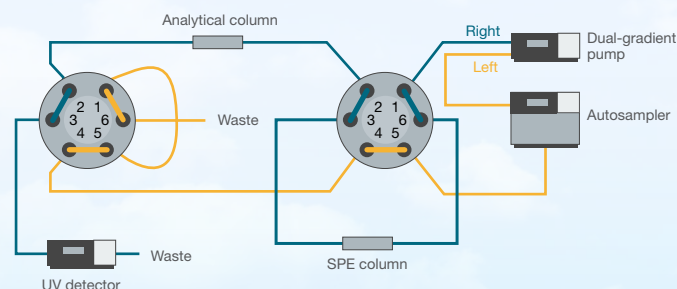
Sensitive determination of microcystins in drinking and environmental waters

Thermo Fisher Scientific

Overview

Microcystins (MCs) are toxins stemming from the waterblooms of cyanobacteria (blue-green algae) growing in lakes, ponds and rivers used as drinking water sources. MC contamination of drinking water is considered a risk factor for cancer, and MC-LR has been associated with most of the incidents of toxicity. As a result, the World Health Organization (WHO) has proposed a provisional guideline concentration of 1.0 µg/L for MC-LR in drinking water.

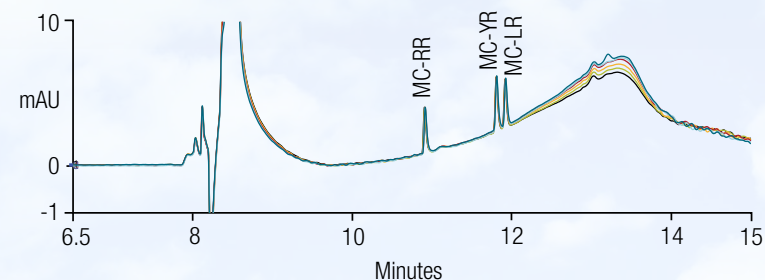
In this application note, an atypical target-cut online SPE method followed by HPLC with UV detection was applied to determine three MCs (-LR, -RR, and -YR) in drinking, tap and lake water. Sub-µg/L concentrations of the three MCs spiked in water samples were determined, which exceeds the WHO requirement.



Flow schematic for the target-cut online SPE method equipped with two 2p-6p valves.

Instrumentation

A target-cut online SPE method was employed followed by HPLC with UV detection on an UltiMate 3000 Dual Gradient Standard LC System.



Overlay of chromatograms of six consecutive injections of a drinking water sample spiked with 0.5 µg/L each of microcystin-RR (MC-RR) and microcystin-YR (MC-YR).



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Determination of linear alkylbenzene sulfonate in textile using online solid-phase extraction followed by HPLC with UV detection

Chinese Applications Team
Thermo Fisher Scientific

Overview

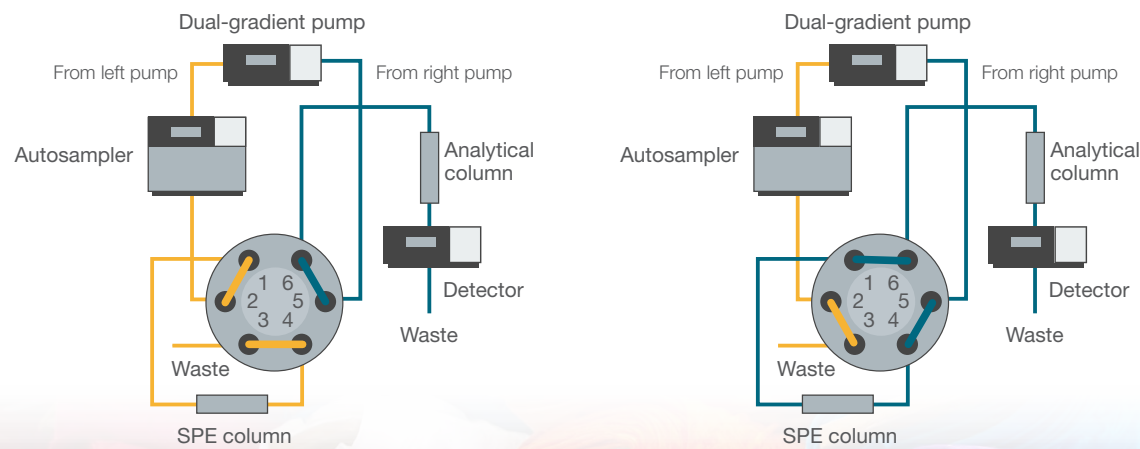
The textile industry regularly performs analysis to detect linear alkylbenzene sulfonate (LAS), which generally refers to a mixture of homologs possessing a linear alkyl carbon number from 10 to 13. The Chinese Standard GB/T 23325-2009 method for the detection of LAS in textiles requires an expensive LC-MS/MS system and the detection limit, 2 mg per kg of textile sample, is relatively high.

This application note demonstrates how the Chinese Standard GB/T 23325-2009 method can be successfully transferred to a lower cost LC-UV system. A lower detection limit than that required in the Chinese Standard can also be obtained

by increasing injection volume and using online SPE to enrich textile extracts. The reusability of the solid phase extraction column reduces sample analysis costs, which is of benefit for labs who routinely analyze LAS in textiles. The good recovery rates of between 101 and 114% and the low detection limit of 0.15 mg/kg, which is significantly lower than the method outlined under GB/T 23325-2009, allow this method to be recommended for general practice.

Instrumentation

The UltiMate 3000 Dual Gradient Standard LC System was used with a Thermo Scientific™ Acclaim™ Surfactant column.



Instrument configuration. When valve is in position 1-2, sample is loaded onto the SPE column (A). When the valve is in position 6-1, the sample is being transferred and separated on the analytical column (B).

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- [Vanquish UHPLC Systems web page](#)
- [Vanquish Duo UHPLC Systems brochure](#)
- [Vanquish Horizon UHPLC System brochure](#)
- [Vanquish Flex UHPLC Systems brochure](#)
- [UltiMate 3000 HPLC Systems web page](#)
- [ISQ EC & EM MS Systems brochure](#)
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- [Charged Aerosol Detector bibliography](#)
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