

Application Note Summaries for Pesticide Analysis

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Extraction of Organochlorine Pesticides from Oyster Tissue Using Accelerated Solvent Extraction.

Pranathi Perati and SM Rahmat Ullah Thermo Fisher Scientific, Sunnyvale, CA, USA

Overview:

Organochlorine pesticides (OCPs) are a class of chemicals that were used to control insect pests since the 1940s. The use of OCPs was banned in the later part of the last century due to their longevity, a trait that made them effective for long-term pest control, but also increased concerns of potential health outcomes such as cancer in humans and ecosystem disruption. Sample preparation is challenging for wet animal tissue such as an oyster sample.

Method:

This Application Brief discusses the use of Thermo Scientific™ Dionex™ ASE Prep MAP, a proprietary polymer designed to remove moisture. A comparison study of spiked oyster samples was extracted using two drying agents and using the conditions listed.

- 1 Thermo Scientific™ Dionex™ ASE Prep MAP and Dionex ASE Prep DE
2. Sodium sulfate

The extracts were analyzed by gas chromatography with an Electron-Capture Detector (GC-ECD).

Conclusions:

This Application Brief describes a simple and reliable method to extract OCPs from oyster tissue. This method also demonstrates the use of Dionex ASE Prep DE and Dionex ASE Prep MAP for in-cell extractions without any pre and post-extraction steps to remove moisture and increase extraction efficiencies in wet samples. The method is ideal for routine extractions of OCPs from wet samples.

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Accelerated Solvent Extraction of Pesticide Residues in Food Products

Oven Temperature: 100 °C

Oven Heat-up Time: 5 min

Static Time: 5 min

Static Cycles: 1-2

Flush Volume: 60% of extraction cell volume

Nitrogen Purge: 100 s

Solvent: Hexane with 10% Acetone

Total Extraction Time: 14–18 min per sample

Total Solvent Used: 15–45 mL per sample

Overview:

Many of the traditional procedures used to perform extractions are time-consuming and solvent-intensive. Accelerated solvent extraction is an extraction technique that speeds the extraction process and reduces the total amount of solvent used. The system uses conventional liquid solvents at elevated temperatures and pressures, which results in increased extraction kinetics. The technique is illustrated by analysis of organochlorine pesticides (OCPs) spiked into potato, wheat and banana.

Method:

Weigh dry samples (1–20 g) and add directly to extraction cells containing a cellulose extraction filter. Grind wet samples (1–10 g) and mix with 6 g of Dionex ASE™ Prep DE (diatomaceous earth) and extract under the conditions listed.

The extracts were analyzed by gas chromatography with an Electron-Capture Detector (GC-ECD).

Conclusions:

Using accelerated solvent extraction, pesticide residue analysis laboratories can increase sample throughput while reducing overall solvent usage. The simplicity of the accelerated solvent extraction technique, combined with results showing excellent correlation to existing methods, have resulted in the rapid acceptance of accelerated solvent extraction in food testing laboratories.

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Determination of Pesticides in Large-Volume Food Samples Using Accelerated Solvent Extraction (ASE)

Overview:

Food samples such as fruit and vegetables have very high water contents and must be mixed with desiccants such as sodium sulfate to achieve quantitative pesticide recovery. Accelerated Solvent Extraction (ASE[®]) has the capability to extract samples with volumes as large as 100 mL. This capability allows the direct extraction of food and vegetable samples with weights in the 30 to 50-g range. This application note reports on the use of the ASE for the determination of organophosphorus pesticides (OPPs) in fruits and vegetables.

Temperature: 100 °C

Solvent: Ethyl acetate/cyclohexane or MeCl₂/acetone (1:1, v/v)

Heat-up Time: 5 min

Static Time: 5 min

Flush Volume: 60%

Purge Time: 180 s

Static Cycles: 1–2

Method:

Baby foods and homogenised apples and carrots are spiked with Organophosphorus Pesticides (OPP), mixed with ASE[®] Prep DE (diatomaceous earth) and extracted using the conditions listed.

Analysis using gas chromatography with flame photometric detection.

Conclusions:

These results confirm that pesticide residues can be easily extracted from large-volume food samples using the ASE. Traditional extraction methods would take from one to several hours for each sample and several hundred millilitres of solvent would be used for each sample. With the ASE, these samples can be extracted in about 15 min each with about 160 mL of solvent for each sample.

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Rapid Determination of Organochlorine Pesticides in Animal Feed Using Accelerated Solvent Extraction (ASE)

Overview:

Animal feed contaminated with organochlorine pesticides (OCPs) has begun to attract worldwide attention. When ingested, the OCPs from animal feed tend to accumulate in certain animal products, especially those rich in fat, such as meat, milk, and butter. Accelerated Solvent Extraction (ASE[®]) offers a more effective approach to recovering pesticides for analysis than more conventional techniques. This Application Note examines the use of ASE[®] in the determination of OCPs from certified reference materials (CRM) BCR 115.

Solvent: Hexane: acetone (3:2)

Temperature: 100 °C

Static Time: 9 min

Static Cycles: 1

Flush: 60%

Purge: 60 s

Method:

Powdered samples are mixed with ASE[®] Prep DE (diatomaceous earth) and extracted under the conditions listed.

Following extractions, samples are cleaned using silica gel adsorption followed by gel permeation chromatography (GPC) and analysed by GC/MS.

Conclusions:

The extraction efficiency and reproducibility of ASE for extracting OCPs from animal feed was tested using an optimized method to extract a certified reference material (BCR 115). ASE provides a faster way to extract OCPs from animal feed than traditional techniques, such as Soxhlet. Additionally, ASE can accomplish these results using far less solvent.

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Fast and Ultrafast LC-MS/MS Methods for Robust and Reliable Analysis of Pesticides in Food Using the Vanquish UHPLC System

Giorgia Greco, Claudia P.B. Martins, Katerina Bousova and Remco Swart
Thermo Fisher Scientific

Overview:

Food safety is an increasing concern that has resulted in stringent pesticide regulation globally and in continuous recalls of food products. Here we present a comparison between fast and ultrafast LC-MS/MS methods in timed SRM mode for the analysis of 250+ pesticides in food extracts. The two methods were compared in terms of analysis time and data quality.

Method:

An already existing Thermo Scientific LC-MS method developed with the Thermo Scientific™ Dionex™ UltiMate™ 3000 RSLC system for the quantitative analysis of more than 250 pesticides in food extracts was transferred to the Vanquish UHPLC system.

Thermo Scientific™ TSQ Endura™ Triple Quadrupole Mass Spectrometer was used for detection with data acquisition and processing using Thermo Scientific™ Dionex™ Chromeleon™ Chromatography Data System (CDS) software, version 7.2 SR2.

Conclusions:

Fast UHPLC separations in combination with ultra-narrow timed SRM scan windows permitted the number of monitored transitions to be maintained without compromising data quality. Outstanding retention time stability achieved with the Vanquish UHPLC system is the key factor for fast timed SRM MS analysis with a high number of data points across the peak.

Ultrafast UHPLC separation resulted in savings of 67% in analysis time and an increase of the sample throughput of 300% without losing critical information.

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Use of UHPLC and High-Resolution MS for Quantitative Analysis of Pesticides in Onion Matrix

Charles Yang, Leo Wang, Dipankar Ghosh, Thermo Fisher Scientific, San Jose, CA

Overview:

Monitoring for pesticide and other chemical residues in produce is essential to maintaining a safe food supply. This application note demonstrates the ability of a high-resolution, accurate-mass UHPLC-MS system, combined with appropriate application-specific workflow software, to provide fast, confident and precise screening and quantitative analysis of pesticides in onion matrix.

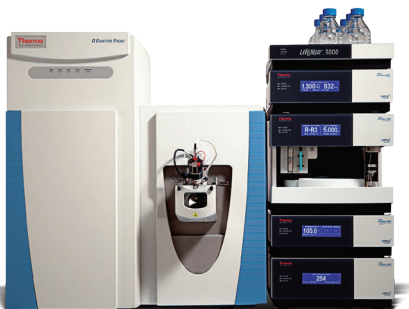
Method:

Onion was prepared for analysis by using a modified QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) method. A mixture of 120 pesticides was used as standards and spiked into onion matrix to determine if there was any ion suppression. Chromatographic analysis was performed using the Thermo Scientific™ Dionex™ UltiMate™ 3000 RSLC UHPLC system using a Thermo Scientific Hypersil GOLD aQ™ column (50 x 2.1 mm, 1.9 μm). All samples were analyzed on a Thermo Scientific Exactive™ Plus benchtop Orbitrap mass spectrometer. Data processing was carried out with Thermo Scientific™ TraceFinder™ software for quantitation and targeted-screening workflows.

Conclusions:

The quantification data showed good reproducibility and recovery rates. The results showed good linearity with excellent sensitivity at very low LOQs, which will assist in detecting pesticides. The Exactive Plus instrument's exceptionally high mass resolution helped resolve matrix compounds that would otherwise interfere with detection of low-level analytes.

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Quantitative and Qualitative Confirmation of Pesticides in Beet Extract Using a Hybrid Quadrupole-Orbitrap Mass Spectrometer

Charles Yang and Dipankar Ghosh, Thermo Fisher Scientific, San Jose, CA
Olaf Scheibner, Thermo Fisher Scientific, Bremen, Germany

Overview:

As world agricultural trade has expanded and concerns over food safety have grown, the enforcement of stricter pesticide regulations has become of utmost importance. A method is described for the analysis of pesticides, showing the utility of a full-scan data-dependent MS/MS workflow to achieve regulatory levels while providing a complete targeted and screening analysis using a high-resolution, accurate mass (HRAM) spectral library for identification and confirmation.

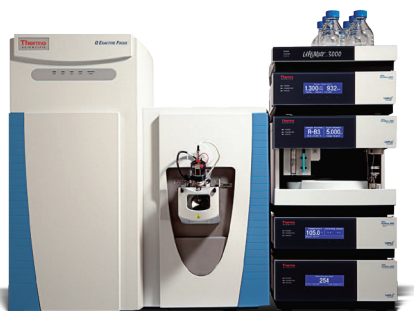
Method:

Beet samples are extracted using a modified QuEChERS method and spiked with a pesticide mix. Chromatographic analysis was performed using the Thermo Scientific™ Dionex™ UltiMate™ 3000 LC system with a Thermo Scientific Accucore™ aQ™ column (100 x 2.1 mm, 2.6 μm). A generic FS-ddMS2 method on a Thermo Scientific™ Q Exactive™ Focus hybrid quadrupole-Orbitrap™ mass spectrometer was used for all samples. Data processing was carried out with Thermo Scientific™ TraceFinder™ software.

Conclusions:

The benchtop Q Exactive Focus MS provided easy access to full quantitative, confirmation and screening data in a single injection. The high resolution and mass accuracy enabled quantification of the compounds over a wide dynamic range (0.05–200 ng/mL) with linear fit, correlation better than 0.99, and %RSD below 15%.

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Streamlined Analysis of 400+ Pesticides in a Single Run Using the TSQ Quantum Access MAX Mass Spectrometer and TraceFinder Software

Jia Wang, Charles T. Yang, Jonathan R. Beck, and Dipankar Ghosh;
Thermo Fisher Scientific, San Jose, CA

Overview:

Growing concerns over food safety and the expanding world agricultural trade have led to the enforcement of stricter pesticide regulations. In 2006, Japan introduced the Positive List System that established maximum residue levels (MRLs) for hundreds of agricultural chemicals in food. Including approximately 400 pesticides, and set a uniform limit of 10 µg/kg (ppb) for chemicals for which MRLs have not been determined.

Method:

Apple, orange and asparagus matrices were prepared for analysis by using a modified QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) method. Chromatographic analysis was performed using the Thermo Scientific Accela 1250 U-HPLC system, a Thermo Scientific™ Hypersil GOLD™ aQ column (100 x 2.1mm, 1.9 µm particle size) and an HTC-PAL autosampler. All samples were analyzed on the Thermo Scientific TSQ Quantum™ Access MAX triple stage quadrupole mass spectrometer. Data acquisition and data processing were performed with Thermo Scientific™ TraceFinder™ software.

Conclusions:

A multi-residue method was developed for the screening and determination of 437 pesticides in 45 minutes in a single run on a triple quadrupole mass spectrometer. The majority of the pesticides were detected in the spiked matrices at concentrations lower than the MRLs established by EU and Japan.

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Determination of Organochlorine Pesticides Using GC-MS with a Helium-conserving Injector

Andrea Caruso, Thermo Fisher Scientific, Milan, Italy



Overview:

Organochlorine pesticides are among the most toxic synthetic pesticides in the world. For this reason, their presence in the environment must be constantly and carefully monitored. Gas chromatography coupled with mass spectrometry for detection is one of the techniques of choice used to analyze these compounds. A recent innovation for sample introduction is now available that reduces helium carrier gas use, making considerable savings.

Method:

A wide range of organochlorine residues are analyzed using a Thermo Scientific™ TRACE™ 1310 GC portion of the Thermo Scientific™ ISQ™ LT GC-MS system, equipped with one Instant Connect Helium Saver injector with a splitless w/glass wool liner. A Thermo Scientific™ TraceGOLD™ TG-5MS 30 m, 0.25 mm 0.25 µm column was used.

Data collection and processing was carried out with Thermo Scientific Dionex™ Chromeleon™ 7.2 Chromatography Data System (CDS) software.

Conclusions:

The analytical performance of the Instant Connect Helium Saver injector is remarkable in terms of both area and retention time reproducibility. As with other TRACE 1300 Series injectors and detectors, the Helium Saver benefits from its modularity, providing freedom from helium supply shortages, while maintaining performance identical to that of traditional SSL injectors. These unique features of the Helium Saver reduce cost without the need to change analytical methods or routine.

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High Efficiency, Broad Scope Screening of Pesticides Using Gas Chromatography High Resolution Orbitrap Mass Spectrometry

Dominic Roberts,¹ Hans Mol,² Marc Tienstra,² Cristian Cojocariu,¹ and Paul Silcock¹

¹Thermo Fisher Scientific, Runcorn, UK

²RIKILT – Wageningen UR, Wageningen, The Netherlands

Overview:

Pesticides are used globally to improve the production and yields of agricultural crops and their use is essential to ensure a sufficient global food supply. To increase the scope of the analysis, chemical screening methods using high-resolution, full-scan mass spectrometry have received significant attention in recent years. These methods use non-targeted acquisition, in which a generic full-scan acquisition is run, followed by targeted data processing of a list of compounds within a database.

Method:

Food and feed samples were extracted following an acetate buffered QuEChERS-based approach. In all experiments, a Thermo Scientific™ Q Exactive™ GC hybrid quadrupole-Orbitrap mass spectrometer was used. Sample introduction was performed using a Thermo Scientific™ TriPlus™ RSH autosampler and chromatographic separation was obtained using a Thermo Scientific™ TRACE™ 1310 gas chromatograph and a Thermo Scientific™ TraceGOLD TG-5SilMS™ capillary column.

Conclusions:

This evaluation demonstrated that the Thermo Scientific Q Exactive GC hybrid quadrupole-Orbitrap mass spectrometer, in combination with TraceFinder™ software, is an extremely effective tool for the routine screening of pesticides in food and feed samples. The Orbitrap mass spectrometer delivers excellent resolving power, mass accuracy and sensitivity

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Fast Screening, Identification, and Quantification of Pesticide Residues in Baby Food Using GC Orbitrap MS Technology

Cristian Cojocariu¹, Dominic Roberts¹, Michael T. Hetmanski², Richard J. Fussell², and Paul Silcock¹

¹Thermo Fisher Scientific, Runcorn, UK. ²Food and Environment Research Agency (FERA), York, UK

Overview:

Pesticides are chemicals widely used to control a variety of pests. We demonstrate the use of GC coupled to Orbitrap™ MS technology for fast, high-throughput pesticide residues analysis in baby food samples, with an almost unlimited scope in the analysis through full-scan acquisition. Quantitative performance comparable to triple quadrupoles and compliance with SANCO guidelines will also be demonstrated.

Method:

Baby food samples were extracted using a citrate buffered QuEChERS protocol. In all experiments, a Thermo Scientific™ Q Exactive GC hybrid quadrupole-Orbitrap mass spectrometer was used. Chromatographic separation was obtained using a Thermo Scientific™ TRACE™1310 gas chromatograph and a Thermo Scientific™ TraceGOLD TG-5SilMS™ capillary column. Data was acquired and processed using Thermo Scientific™ TraceFinder™ software.

Conclusions:

The Q Exactive GC system provides high performance quantitative analysis in full-scan for broad-scope pesticide residue testing, even with fast GC separations.

Acquisition with a routine mass resolution of 60,000 FWHM at m/z 200 eliminates isobaric interferences, increasing confidence in results when screening pesticides in complex matrices. The consistent sub-ppm mass accuracy achieved for all compounds ensures confident compound identification.

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Analysis of Dithiocarbamate Pesticides by GC-MS

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¹National Research Center for Grapes, Pune, India,

²Thermo Fisher Scientific, Mumbai, India,

³Thermo Fisher Scientific, Singapore

Overview:

The class of dithiocarbamate fungicides (DTCs) is widely used in agriculture. They are non-systemic and both the formulation and their break-down products typically remain at the site of application. DTCs are not stable and cannot be extracted or analyzed directly.

Method:

Dithiocarbamates are quantitatively converted to carbon disulphide by reaction with tin(II)chloride in aqueous HCl (1:1) in a closed bottle at 80 °C. The CS₂ gas produced is absorbed into iso-octane solvent and measured by GC-MS. A Thermo Scientific™ TRACE GC Ultra™ gas chromatograph equipped with Thermo Scientific™ Triplus™ RSH liquid autosampler and coupled to a Thermo Scientific™ ITQ™ 900 ion trap mass spectrometer was used for analysis.

Conclusions:

The sensitivity of the method was evaluated in terms of the limit of detection (LOD) and limit of quantification (LOQ) which were respectively 0.005 and 0.04 µg/mL. This method has been developed initially for the ITQ ion trap mass spectrometer, but the same parameter setup is suitable for the Thermo Scientific™ ISQ™ series single quadrupole or Thermo Scientific™ TSQ™ Quantum XLS Ultra or Thermo Scientific™ TSQ 8000™ triple quadrupole mass spectrometers, as well.

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Analysis of Multi-Residue Pesticides Present in Ayurvedic Churna by GC-MS/MS

Manoj Surwade, Sunil T Kumar, Aarti Karkhanis, Manish Kumar, Soma Dasgupta, Hans-Joachim Huebschmann, Thermo Fisher Scientific

Overview:

Ayurveda is a Sanskrit term, made up of the words “ayus” and “veda” meaning life and science; together translating to ‘science of life’. A blend of several herbs and spices make up the powdered mixture known as “churna”. Depending on its intended use for medicinal, beauty or culinary purpose, the recipe varies. Due to the use of pesticides in the fresh herbs, the “churna” may contain residual pesticides.

Method:

QuEChERS extracts are analysed using the Thermo Scientific™ TriPlus™ RSH liquid auto sampler, a Thermo Scientific™ TRACETM 1300 Series gas chromatograph fitted with a Thermo Scientific TraceGOLDTM TG-5 SiIMS, 30 m x 0.25 mm x 0.25 µm column and the Thermo Scientific™ TSQ 8000™ triple quadrupole GC-MS/MS system. The Thermo Scientific™ TraceFinder™ software was used for method setup and data processing.

Conclusions:

Within 28 minutes, 200 pesticides were screened and quantitatively determined using the described pesticide analysis method. The QuEChERS sample preparation method provided high recoveries and good reproducibility. Linearity, specificity, recovery, and repeatability of the method were established with minimal sample preparation time. This method can be utilized for detection and confirmation of trace amounts of pesticides in difficult matrices such as herbal churnas.

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Three-Fold Increase in Productivity for Pesticide Residue Analysis in Baby Food Using Fast Triple Quadrupole GC-MS/MS

Cristian Cojocariu,¹ Michael T. Hetmanski,² Paul Silcock,¹ and Richard J. Fussell²

¹Thermo Fisher Scientific, Runcorn, UK

²Food and Environment Research Agency (FERA), York, UK

Overview:

Trace amounts of pesticide residues can be found in food products, including baby food. Babies are more sensitive to chemicals than adults, and their food needs to be analyzed as accurately as possible. Detecting pesticides in food requires sensitive instruments that must be able to analyze a large number of samples in a short turnaround.

Method:

Baby food is analyzed for pesticide residues using acetonitrile as the extraction solvent. Direct injection of low sample volume QuEChERS acetonitrile extracts is combined with fast temperature ramps to shorten gas chromatograph run times. The Thermo Scientific™ TSQ™ 8000 Evo triple quadrupole GC-MS/MS with EvoCell collision chamber technology and selected reaction monitoring (SRM) scheduling with timed-SRM software allows run time to be shortened significantly.

Conclusions:

Laboratory productivity was tripled as a result of direct analysis of acetonitrile extracts, fast data acquisition with EvoCell fast collision cell technology, and comprehensive detection of pesticides using simultaneous full scan and SRM data acquisition. Excellent sensitivity was achieved, with all pesticides detected at a concentration of 5–10 ng/g with IDL values from 0.2–3.7 ng/g.

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Multi-Residue Pesticide Analysis in Herbal Products Using Accelerated Solvent Extraction with a Triple Quadrupole GC-MS/MS System

Hans-Joachim Huebschmann, Joachim Gummersbach, Thermo Fisher Scientific, Dreieich, Germany

Nicole Rueckert, Johann Kirchner, Elmar Häfner, Phytolab GmbH & Co KG, Vestenbergsgreuth, Germany

Overview:

The residue analysis of pesticides has developed in recent years into a comprehensive methodology for the detection of many hundreds of potential contaminating compounds. A multi-residue method for herbal products and teas is faced with additional challenges from the worldwide origin of the products and the complex matrix of the dried materials.

Method:

Herbal and tea samples were extracted with an accelerated solvent extraction method using the Thermo Scientific™ Dionex™ ASE™ 350 Accelerated Solvent Extractor. The ASE method used is described in an official pesticide standard method and is also detailed in the full application note. The extracts were cleaned up via gel permeation chromatography and analysed using the Thermo Scientific TriPlus™ RSH liquid autosampler, TRACE™ GC 1310 gas chromatograph and the TSQ™ 8000 triple quadrupole GC-MS/MS detection system.

Conclusions:

Quantitative calibrations were performed in a standard matrix and showed excellent linearity and precision over the relevant concentration range to control the regulated MRL levels. The high matrix selectivity of the TSQ 8000 system allowed for reduced sample preparation, providing high recoveries for a wide range of chemically diverse pesticide compounds.

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Validation of the Method for Determination of Pesticide Residues by Gas Chromatography – Triple-Stage Quadrupole Mass Spectrometry

Laszlo Hollosi, Katerina Bousova, Michal Godula
Thermo Fisher Scientific, Food Safety Response Center, Dreieich, Germany

Overview:

Pesticide residue analysis in food is one of the most important and challenging tasks in routine laboratory practice. The objective of this validation study was to prove a complete workflow solution (delivered by Thermo Scientific chemicals, consumables and instrumentation) that can be implemented for routine multi-residue pesticide analysis.

Method:

Ready-to-use QuEChERS kit containing both extraction and clean-up tubes and associated protocol were used for sample preparation. Sample measurements were carried out using the Thermo Scientific™ TRACE™ 1310 Gas Chromatograph coupled to the TSQ™ 8000 Evo GC-MS/MS Pesticide Analyzer. For instrument control, analysis, data review and reporting Thermo Scientific™ TraceFinder™ software was used.

Conclusions:

The method performance parameters indicate that the performance for the majority of target compounds complies with current regulatory requirements. TSQ 8000 GC-MS system delivers the required system performance for the target compounds, especially regarding sensitivity, selectivity and recovery.

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Detection of Organochlorine Pesticides by GC-ECD Following U.S. EPA Method 8081

Andrea Caruso and Massimo Santoro
Thermo Fisher Scientific, Milan, Italy

Overview:

Organochlorine insecticides are among the oldest and most toxic synthetic pesticides. Organochlorines are neurotoxic and some organochlorine compounds are suspected carcinogens. For these reasons, the presence of these analytes in water, soils and other solids must be strictly controlled and various analytical methods have been developed to extract and purify them from various matrixes.

Method:

The experimental method follows the guidelines for U.S. EPA method 8081, with slight modifications to the GC ramp and use of a column with 0.25 μm film thickness to guarantee better separation. Analysis is performed with the Thermo Scientific™ TRACE™ 1310 Gas Chromatograph, an Instant Connect Split/Splitless (SSL) Injector and an Instant Connect Electron Capture Detector (ECD). All samples are acquired and processed using the Thermo Scientific™ Dionex™ Chromeleon™ 7.2 chromatography data system (CDS) software.

Conclusions:

The system shows excellent results in terms of linearity, sensitivity and reproducibility. These results indicate that this system is a sensitive, cheaper and simpler alternative to mass spectrometry for assessing the presence of organochlorine residues, as a result of the excellent selectivity of the ECD for chlorinated compounds.

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High Precision Pesticide Analysis in Produce using GC Triple Quadrupole and U-SRM Mode

Inge de Dobbeleer, Joachim Gummersbach, Hans-Joachim Huebschmann, Anton Mayer, Thermo Fisher Scientific, Dreieich, Germany

Overview:

Pesticides are widely used in agriculture to protect crops and to improve efficiency of production. Consequently, governments, food producers and food retailers have the duty to ensure that any residues occurring in foods for human consumption are at or below Statutory Maximum Residue Levels (MRLs). Many of these MRLs are set at a default value of 0.01 mg/kg, the typical limit of determination of routine analytical methods.

Method:

All samples were prepared using the QuEChERS technique. All sample analyses were carried out using the TSQ Quantum XLS Ultra™ GC-MS/MS system, equipped with a Thermo Scientific™ TRACE GC Ultra™ gas chromatograph. Sample introduction was performed using the Thermo Scientific™ TriPlus™ RSH autosampler. The capillary column was a 30m x 0.25mm Thermo Scientific™ TraceGOLD™ TG-5MS column (5% phenyl film) and 0.25 µm film thickness.

Conclusions:

Advances in HyperQuad technology offers increased analytical performance for routine applications such as pesticide analysis. A high level of accuracy and precision was reached during data evaluation. This resolution technology development allows for advanced GC-MS/MS operations to be performed, such as U-SRM to further increase selectivity in complex matrices. This not only improves quantitative measurements, but it is also amenable when using a reduced sample clean-up which is typical for QuEChERS methodologies.



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EPA Method 557 – Analysis of Haloacetic Acids, Dalapon and Bromate in Drinking Water by IC-MS/MS

Jonathan R. Beck, Terri Christison, and Hans Schweingruber,
Thermo Fisher Scientific, San Jose, CA

Overview:

Haloacetic acids (HAAs) are formed as disinfection byproducts when water is chlorinated to kill bacteria. According to the EPA, there is an increased risk of cancer associated with long-term consumption of water containing levels of HAAs that exceed 0.06 mg/L. This method allows for the analysis of all nine HAAs, plus bromate and the pesticide dalapon in the same IC-MS/MS run without sample preparation.

Method:

Samples needing no pretreatment were directly injected onto a Thermo Scientific™ Dionex™ ICS-5000+ Reagent-Free™ IC system with MS/MS detection. Mass spectrometry (MS) was performed with a Thermo Scientific™ TSQ Endura™ triple quadrupole mass spectrometer with a heated electrospray ionization (HESI-II) probe. Data acquisition and processing were carried out using the Thermo Scientific™ TraceFinder™ software.

Conclusions:

Reagent Free IC systems coupled with an MS/MS detector proved a powerful tool used in the quantitation of haloacetic acid samples. Excellent reproducibility and time savings were achieved when compared to the conventional EPA methods using GC with electron capture detection.

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