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Regulatory Acceptance for Accelerated Solvent Extraction: A Reference Summary

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Executive Summary

National level governing bodies and organizations which monitor global health risks, such as the United States Congress and the World Health Organization, authorize regulatory agencies to set maximum contaminant levels for toxic contaminants found in the environment and consumer goods. These regulatory agencies also advise on the appropriate analytical technique to use for the quantitative determinations of these compounds by providing scientific methods.

Sample preparation is critical for successfully performing the recommended scientific methods. Disregarding the importance of sample preparation can result in insufficient percent recoveries of the contaminants of interest.

For the specific extraction of solid and semisolid matrices, accelerated solvent extraction is a technique that increases sample extraction efficiency by utilizing elevated temperature and pressure conditions. This technique is recommended for use in various regulatory methods worldwide, a few which will be highlighted below.

Key Words

Automated Soxhlet, Soxhlet, Accelerated solvent extraction, Polychlorinated biphenyls, Toxaphene, Perchlorate, Polycyclic aromatic hydrocarbons, U.S. EPA Method 3545A, U.S. EPA Method 8276, U.S. EPA Method 6860, GB/T 19649-2006, SN/T 2593.1-2010, National Standard NMX-AA-146-SCFI-2008, L00.00-34, Pesticide residues

Protecting the Global Community

In order to protect the global community from risks associated with human health and environmental safety, laws and standards are developed to set legal limits on harmful substances which can be found on land, in ground and surface water or in commercial products (food, electronic devices, etc). The standards can be created by governing bodies at the national and state levels as well as by organizations that form policies on human health and environmental safety issues.

Regulatory agencies including the United States Environmental Protection Agency (U.S. EPA), are granted the power to develop and enforce regulations. The United States Congress specifically authorizes the U.S. EPA to write regulations based on environmental laws they have created. For example, Section 408 of the Federal Food, Drug and Cosmetic Act authorizes the U.S. EPA to regulate pesticide residues in foods by setting maximum residue limits.

Regulatory agencies also develop scientific methods which provide recommended techniques for extraction, separation and detection of regulated compounds which analytical laboratories utilize for compliance monitoring. High performance liquid chromatography (HPLC), gas chromatography (GC), ion chromatography (IC) and mass spectrometry (MS) are common examples of recommended techniques for the quantitative determinations of regulated compounds. The process of sample preparation in these scientific methods is very critical as any variation in extraction conditions can affect the percent recoveries of the analytes of interest.

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Accelerated Solvent Extraction

Accelerated solvent extraction is a sample preparation technique that was first introduced in 1995. It improves extraction efficiency for solid and semisolid sample matrices through the use of elevated temperature and pressure with organic and water based solvents. The technique reduces both extraction times and solvent usage in addition to providing more consistent and reliable data when compared to methods such as Soxhlet and automated Soxhlet.

Many of the organic solvents used boil at relatively low temperatures at atmospheric pressure. If sufficient pressure is exerted on the solvent, the boiling point of the solvent is elevated. As a result, temperatures above the

boiling point can be used for extraction. When extractions are achieved at elevated temperatures, several factors contribute to improved speed, efficiency, and reduced solvent use:

1. Solvating power is higher
2. Diffusion rates are faster
3. Solvent viscosity is decreased
4. Solute-matrix interactions (dipole attractions, Van der Waals forces, hydrogen bonding, etc.) are more easily disrupted allowing the analytes to be removed from the matrix.

Techniques such as Soxhlet or automated Soxhlet may only allow extractions to take place at the solvent's boiling point.

Acceptance of Accelerated Solvent Extraction by Regulatory Agencies

The below scientific methods recommend the use of the accelerated solvent extraction method for sample extraction from solid and semisolid matrices. The section under U.S. EPA Method 3545A has been expanded to show a specific example of time and solvent savings when using the accelerated solvent extraction method compared to Soxhlet or Automated Soxhlet.

United Stated of America

U.S. EPA Method 3545A

This method is for the extraction of water insoluble or slightly water soluble organic compounds (semivolatile organic compounds, organophosphorus pesticides, organochlorine pesticides, chlorinated herbicides, polychlorinated biphenyls (PCBs), polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans (PCDDs/PCDFs), and diesel range organics (DRO)) from soils, clays, sediments, sludges, and waste solids using accelerated solvent extraction method.[1] Table 1 demonstrates both the solvent and time savings experienced using the accelerated solvent extraction technique in comparison to other traditional extraction techniques; only 15–45 mL of solvent is required per sample with a total extraction time of 10-15 min.

Table 1. Solvent and time savings.

EPA Method	Technique	Solvent Used (mL)	Extraction Time
3545A	Accelerated Solvent Extraction	15–45	10-15 min
3540C[1]	Soxhlet	300–500	18 hours
3541[2]	Automated Soxhlet	50	2 hours

U.S. EPA Method 8276

Toxaphene is a toxic insecticide which does not degrade easily in the environment and can cause damage to the lungs, immune and nervous systems.[2] U.S. EPA Method 8276 is used to determine the concentrations of certain toxaphene congeners and technical toxaphene in extracts from solid and liquid matrices. For determining the concentration of technical toxaphene in fish tissue specifically, the accelerated solvent extraction technique can be used followed by gas chromatography with negative ion chemical ionization mass spectrometry (GC-NICI/MS).[3]

U.S. EPA Method 6860

Perchlorate can interfere with the human thyroid's ability to produce hormones which are required for growth and development.[4] U. S. EPA Method 6860/6850 is used for the determination of perchlorate in surface water, groundwater, salt water and soil using IC coupled with electrospray ionization (ESI) mass spectrometry (MS) or tandem mass spectrometry (MS/MS). Accelerated solvent extraction is a recommended technique for the extraction of perchlorate in selected soils and wastewater treatment sludges specifically. [5]

China

GB/T 19649-2006

Agricultural chemicals, which include pesticides, are used to protect the health of crops and livestock, boost crop yields, and manage weed growth. However plants and animals must be tested for pesticide residues in order to minimize health risks to consumers which can depend on factors including pesticide type and the length of exposure.[6] China has created a recommended method for regulating 475 pesticides and related chemicals residues in barley, wheat, oat, rice and corn. GB/T 19649-2006 is a recommended national standard which uses the accelerated solvent extraction technique followed by gas chromatography-mass spectroscopy (GC-MS). [7]

SN/T 2593.1-2010

Polycyclic aromatic hydrocarbons (PAHs) are a widespread environmental pollutant comprising specific isomers which are classified as carcinogens, mutagens, and teratogens.[8] The Chinese Method SN/T 2593.1-2010 is used for the determination of 16 polycyclic aromatic hydrocarbons (PAHs) specifically in electronics using the accelerated solvent extraction technique followed by HPLC as the analytical technique.[9]

Mexico

National Standard NMX-AA-146-SCFI-2008

This standard is for the determination of PAHs in soils and sediments using the accelerated solvent extraction technique followed by GC-MS and HPLC using an Ultraviolet Visible (UV-VIS) Fluorescence Detector. [10]

Germany

Lebensmittel Bedarfsgegenstände Gesetz (LMBG) L00.00-34

The official method Lebensmittel Bedarfsgegenstände Gesetz (LMBG) L00.00-34 is for the determination of pesticide residues in various foodstuffs. The accelerated solvent extraction technique is recommended for plant material and foodstuffs with a fat content below 2.5g/100g and water content below 20g/100g depending on the specific type of compounds. Analysis is done by GC with multiple detectors including a flame photometric detector (FPD), a mass spectrometric detector (MS), a nitrogen/phosphorous detector (NPD) and a electron capture detector (ECD) depending on the application.[11]

Conclusion

Compliance monitoring assures that the regulated community adheres to a set of policies created by regulatory agencies for the determination of compounds which pose global health risks. Sample preparation is an important part of this determination process and can require concerning amounts of time and solvent when using traditional extraction techniques. Use of the accelerated solvent extraction technique in regulatory methods including U.S. EPA 3545A provides examples of how this automated technique reduces both the solvent usage and extraction time per sample.

References

1. U.S. Environmental Protection Agency. Our Mission and What We Do. [Online] http://www2.epa.gov/aboutepa/our-mission-and-what-we-do (accessed May 19, 2014).
2. U.S. Environmental Protection Agency. Pesticide Registration Manual. [Online] http://www.epa.gov/pesticides/bluebook/chapter11.html (accessed May 19, 2014).
3. Thermo Fisher Scientific. Whitepaper 70652: Use of Accelerated Solvent Extraction to Improve Laboratory Workflow. Sunnyvale, CA, 2013. [Online] http://www.dionex.com/en-us/webdocs/114404-WP-ASE-Lab-Workflow-WP70652_E.pdf (accessed April 23, 2014).
4. Thermo Fisher Scientific. ASE Environmental Applications Notebook. Sunnyvale, CA. 2013. [Online] http://www.thermoscientific.com/envASEnotebook (accessed April 7, 2014)
5. SW- 846 Method 3545A: Pressurized Fluid Extraction (PFE). U.S. Environmental Protection Agency, February 2007.
6. SW-846 METHOD 3540C: Soxhlet Extraction. U.S. Environmental Protection Agency, Dec 1996.
7. SW-846 METHOD 3541: Automated Soxhlet Extraction. U.S. Environmental Protection Agency, Sept 1994.
8. Persistent Bioaccumlative and Toxic (PBT) Chemical Program, U.S. Environmental Protection Agency. [Online] http://www.epa.gov/pbt/pubs/toxaphene.htm (accessed April 17, 2014).
9. SW- 846 METHOD 8276: Toxaphene and Toxaphene Congeners by Gas Chromatography/Negative Ion Chemical Ionization Mass Spectrometry (GC-NICI/MS), U.S. Environmental Protection Agency, Sept 2012.
10. Perchlorate, U.S. Environmental Protection Agency. [Online] http://water.epa.gov/drink/contaminants/unregulated/perchlorate.cfm (accessed May 19, 2014)
11. SW-846 METHOD 6860: Perchlorate in Water, Soils and Solid Wates Using Ion Chromatography/Electrospray Ionization / Mass Spectrometry (IC/ESI/MS OR IC/ESI/MS/MS), U.S. Environmental Protection Agency, Jan 2007.
12. Thermo Fisher Scientific. Dionex Agricultural Chemical Contaminants. [Online] http://www.dionex.com/en-us/markets/food-beverage/food-safety/agricultural-chemical/lp-71654.html (Accessed May 19, 2014).
13. Guofang Peng et.al. GB/T 19649-2006: Determination of 475 Pesticides and Related Chemicals Residues in Grains Using Gas Chromatography-Mass Spectroscopy (GC-MS), 2006.
14. Thermo Fisher Scientific. Whitepaper 71064: Accelerated Solvent Extraction for Monitoring Persistent Organic Pollutants in Ambient Air. Sunnyvale, CA, 2014. [Online] http://www.thermoscientific.com/content/dam/tfs/ATG/CMD/CMD%20Documents/white-papers/WP-71064-Monitoring-Persistent-Organic-Pollutants-POPs-Ambient-Air-WP71064-EN.pdf (accessed April 7, 2014).
15. Minghui Zhou et.al. SN/T 2593.1-2010: Determination of Polycyclic Aromatic Hydrocarbons (PAHs) in Electrical and Electronic Equipment: Part 1High-Performance Liquid Chromatography (HPLC), 2010
16. National Standard NMX-AA-146-SCFI-2008: Soil – Polycyclic Aromatic Hydrocarbons by Gas Chromatography/Mass Spectrometry (GC/MS) or by High Resolution Liquid Chromatography using Fluorescence and Ultraviolet Visible Detectors (UV-VIS), General Standards Office of the Ministry of Economy, 2009.
17. Beuth Verlag, Berli. Collection of Official Methods under Article 35 of the German Federal Food Act. Analysis of foodstuffs: Modular multiple analytical method for the determination of pesticide residues in foodstuffs, 1999, L00.00-34. [Online] www.beuth.de (accessed July 22, 2014).

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