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Accelerated Solvent Extraction (ASE) in the Natural Products Industry.

The term natural products is a very broad classification and can be a part of Food and Beverage (Functional Foods) and Pharma/Life Science (Nutraceuticals) markets, and includes herbal health care and Traditional Chinese Medicine (TCM). Many scientists and organizations are working to understand what compounds are present in plant matrices that potentially cause the observed pharmacological effects. One of the challenges facing these scientists is the isolation of these compounds from the plant matrices. Often, traditional extraction methods produce extracts that are too complicated to analyze or characterize well. ASE[®] instruments increase the productivity of labs engaged in this field of research and produce extracts that are easier to analyze and characterize. Because water can be used as the extraction solvent, ASE instruments can also mimic one of the traditional modes of administration—seeping in hot water. Clearly, ASE technology is a good fit for any scientist or laboratory involved in isolating and studying natural products.

Features Unique to ASE	Benefits to Natural Product Extraction
True Walk-Away Automation	Overnight Extraction = Higher Productivity
Extract Same Sample with Different Solvents	Fractionation Extractions
Flow-Through Design	Removal of Interferences During Extraction
Acid/Base Extraction Capabilities	Hydrolyzation of Complex Matrices
Combined Static/Dynamic Extractions	Exhaustive Extraction = Increased Recoveries
Automatic Rinsing	System Rinsed with Any Solvent Automatically

ASE Features and Benefits

True Walk-Away Automation Details

This feature is very beneficial to labs extracting natural products because generally they have higher sample throughput, and preparation can be the bottleneck in the process. Using an ASE system, an entire rack of 24 can be set up at the beginning of the day and another rack of 24 samples can be set up to run overnight with extracts that are ready for analysis the first thing in the morning. With minimal user intervention, 48 samples, or more, per day can be extracted and ready for analysis. No other sample preparation process available offers this type of automation!

Fractionation Extraction Details

Being able to extract the same sample into different collection vials allows the operator to use different solvents to extract different analytes from the same sample. For example, a plant sample can be extracted using weakly polar solvent first, removing the less polar compounds of interest, then the same sample can be extracted using a more strongly polar solvent to remove the more polar compounds of interest, with each extract collected in a separate vial for easy analysis. This makes the extracts easier to analyze because there are fewer compounds in each extract. This feature is unique to ASE and cannot be duplicated by any other extraction method available!

Flow-Through Design Details

Because the ASE extraction cells are designed for the solvent to enter the top and exit the bottom of the cell with the analytes, the user can add various adsorbents to the bottom of the cell to remove unwanted co-extractables during the extraction process. This helps eliminate post extraction cleanup steps and accelerates the entire sample preparation process. This is very important to a laboratory performing extraction on natural products. Many times these samples are more difficult to extract and the extracts must be cleaned or filtered prior to analysis. With the ASE system, this labor-intensive step of the sample preparation process can be eliminated.

Acid/Base Extraction Capabilities Details

Many times, natural product samples can be difficult to extract due the complexity of the plant-based matrix. The acid- and base-resistant capabilities of the ASE system offer the customer some beneficial options. First, the samples can be hydrolyzed using a strong acid or base such as 8 M HCl or NaOH. This breaks down the plant matrix and allows the extraction solvent to reach the analytes of interest. The second option allows acidified or base-treated solvent to be pumped into the cell as the extraction is performed. Again, ASE is the only extraction method that offers this option!

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Dynamic and Static Extraction Details

ASE is the only extraction method that provides both a dynamic and static extraction in the same run! Dynamic extraction is defined as the ability to introduce fresh solvent during the extraction process. This is important because it ensures that the extraction solvent will not become oversaturated with the analyte, decreasing its ability to remove more analyte. Static extraction is defined as holding the extraction solvent and sample for a set period of time to maximize the solubility of the analytes. Performing both dynamic and static extractions is what defines ASE as an exhaustive extraction technique and provides maximum analyte recoveries. This is important to laboratories extracting natural products to determine the quantity of active ingredients present in each sample.

Automatic Rinsing Details

The automatic rinse function of the ASE system allows the user to set up different batches of samples for the same extraction run using different solvents for each batch. The system will automatically change solvents and rinse the entire system with the next solvent to be used, with no need for user intervention. This is ideal for laboratories extracting natural products, as many of the samples are different matrices and a run of 24 samples can contain any number of combinations of sample matrices and/or extraction methods. ASE is the only extraction method to offer automatic rinsing!

Application Notes

AB 102: Determination of Aucubin, Genipoide, and Pinoresinol Diglucoside in *Cortex Eucommiae* Using ASE and HPLC

Overview: This application brief (AB) describes an efficient method for the determination of aucubin, genipoide, and pinoresinol diglucoside in *Cortex eucommiae* using Accelerated Solvent Extraction (ASE) and an UltiMate[®] 3000 HPLC system. The ASE method for the extraction of *C. eucommiae* replaced the soxhlet extraction method in the Chinese Pharmacopeia Edition.

http://www.dionex.com/en-us/webdocs/77703-AB102-ASE-LC-Cortex-18Aug09-LPN2192-01.pdf

AN 192: Rapid Analysis of Ginseng Using Accelerated Solvent Extraction and High Performance Liquid Chromatography

Overview: Here, the authors describe a method that combines ASE extraction with a 25 min HPLC separation for the analysis of 15 ginsenosides. This method is suitable for analyzing Asian ginseng, American ginseng, and notoginseng.

http://www.dionex.com/en-us/webdocs/61830-AN192_ASE_HPLC_ Ginseng_29Aug07_LPN1965.pdf

AN 207: Chromatographic Fingerprinting of *Flos Chrysanthema* Indici Using HPLC

Overview: In this application note, the authors establish an HPLC chromatographic fingerprint of *F. chrysanthemi* based on discriminating the characteristic peaks of chlorogenic acid and flavonoids (luteolin-7-o-glucoside, linarin, luteolin and apigenin; structures shown in Figure 1). An ultrasonic extraction technique is also compared to Accelerated Solvent Extraction for isolation of the target components from the samples.

http://www.dionex.com/en-us/webdocs/71823-AN207-HPLC-Flos%20Chrysanthema-14April09-LPN2092.pdf

AN 232: Determination of Anthraquinones and Stilbenes in Giant Knotweed Rhizome by HPLC with UV Detection

Overview: An efficient HPLC method that determines the eight main active components of giant knotweed rhizome in a single injection (anthraglycoside A, anthraglycoside B, emodin, physcion, rhein, chrysophanol, resveratrol, and polydatin.) is described here. Samples were extracted using ultrasonic extraction and the ASE 200 Accelerated Solvent Extractor respectively, with satisfactory results obtained for both extraction methods.

http://www.dionex.com/en-us/webdocs/77633-AN232-LC-Anthraquinones-Knotweed-21Aug2009-LPN2280-01.pdf

AN 335: Accelerated Solvent Extraction (ASE) of Active Ingredients from Natural Products

Overview: This application note describes the use of ASE for extraction of commercially available nutritional supplements; specifically hypericin from *Hypericum perforatum* (St. John's Wort) and berberine from *Hydrastis canadenis* (Goldenseal root). The current methods used for extraction of this product are Soxhlet and sonication, both of which are time- and solvent-intensive. Here, ASE extracts were generated in 14 minutes per sample using 15 mL of solvent and were immediately ready for processing and analysis.

http://www.dionex.com/en-us/webdocs/4476-AN335.pdf

AN 346: Totally Automated Sample Preparation Using Accelerated Solvent Extraction (ASE) Coupled with the Gilson ASPEC®: The Determination of Dianthrones in St. John's Wort

Overview: Here, the authors report on the use of a coupled ASE-ASPEC system as an automated extraction and extract manipulation device for the determination of dianthrones in St. John's Wort (*Hypericum perforatum*). The results using the coupled system are compared to those obtained using a traditional sonication procedure followed by manual SPE cleanup.

http://www.dionex.com/en-us/webdocs/4521-AN346_V13.pdf



AN 357: Extraction of Phenolic Acids from Plant Tissue Using Accelerated Solvent Extraction (ASE)

Overview: The extraction of phenolic acids from two different plants (eggplants and black cohosh) using ASE is the focus of this method. ASE is able to extract phenolic acids from plant tissue more efficiently than traditional extraction techniques while saving time and solvent.

http://www.dionex.com/en-us/webdocs/40398-AN357_V31_releasedJC120806.pdf

AN 362: Extraction of Herbal Marker Compounds Using Accelerated Solvent Extraction (ASE) Compared to Traditional Pharmacopoeia Protocols

Overview: The use of the ASE system for solvent extraction of five marker compounds from herbal preparations followed by analysis by HPLC is reported here. The results are compared to traditional pharmacopoeia extraction methods.

http://www.dionex.com/en-us/webdocs/69956-AN362-ASE-Marker-Compounds-Supplements-14Jan09-LPN2144.pdf

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Names of scientists using ASE for natural products can be obtained by contacting the Salt Lake Technical Center directly or via email at asesupport@dionex.com.

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