

# Automated Sample Preparation Accelerated Solvent Extraction



# Advanced Extractions ASE 150 and ASE 350 Solvent Extractors Deliver Greater Capabilities

### **Unsurpassed Extraction Technology**

The Thermo Scientific Dionex ASE Accelerated Solvent Extractor for solid samples and the Thermo Scientific Dionex AutoTrace Solid-Phase Extraction instrument for liquid samples provide your lab with total sample preparation. The Dionex ASE™ system uses a combination of elevated temperature and pressure with common solvents to increase the efficiency of the extraction process. The result is faster extraction times and a significant reduction in solvent use. The Dionex ASE 150 and ASE 350 continue to deliver the proven ASE technology for solvent extraction of any solid sample. ASE methods are established and accepted in the environmental, pharmaceutical, foods, polymer, and consumer products industries. ASE technology is accepted by the U.S. EPA as Methods 3545A and 6860, and by the American Society for Testing and Materials as ASTM D 7210. Using solvents at elevated temperature and pressure, ASE combines the following features to deliver superior extractions:

- High analyte solubility
- Reduced matrix effects or weaker matrix and analyte interaction
- Faster analyte diffusion from matrix into solvent
- Decreased solvent viscosity for increased solvent penetration into the matrix
- Increased pressure to maintain solvent in a liquid phase during the extraction process

### The Value of ASE

Only ASE provides the user with unsurpassed flexibility:

- Wide range of sample sizes: 1–100 g
- Consistent extraction from sample to sample and batch to batch
- Reduced solvent consumption using standard ASE operation or solvent saver mode
- Flow-through technology with pH-hardened Thermo Scientific Dionium pathway to support acid or alkaline sample matrices and solvents

Sequential extractions and the Dionex ASE SmartRun system assist with method development. SmartRun<sup>™</sup> is a feature of the Dionex ASE 350 instrument that ensures the proper collection vial is matched with the proper size extraction cell. The pH-hardened pathway is ideal for in-cell cleanup using special resins or acidic or alkaline sorbents.

### The Dionium Pathway

The chemically-inert Dionium<sup>™</sup> pathway is ideal for samples pretreated with acids or bases. Dionium supports expanded choices of both pretreated matrices and post-treatment adsorbents for in-cell cleanup.

Dionex ASE 350 Feature	Benefit/Value
Accepts both 250- and 60-mL collection bottles and vials—28 60-mL vials, 19 250- mL bottles, or 28 of the 60-mL vials and five of the 250-mL bottles	Increased flexibility for different extractions
Dionium, chemically resistant pathway	Increased capability and different applications
Solvent saver mode	Increased solvent savings
New faster pump (70 mL/min)	Faster extractions
Larger front panel with new keyboard	Greater ease of operation
New needle assembly with two vent needles	Reliable operation
Integrated solvent controller	Smaller instrument footprint
Improved waste bottle interface	Easier access for dumping waste
Electronic calibration of pressure transducer and hydrocarbon sensor	Simpler calibration procedure
Two 250-mL bottles for collection of rinse solvent	Full capacity
USB port for firmware update and computer control with Chromeleon 7.1 CDS or higher	Downloading sample information

Dionex ASE 150 Feature	Benefit/Value
Accepts both 250- and 60-mL collection bottles and vials	Increased flexibility
Safety shield over bottles and vials	Added user protection
Dionium, chemical resistant pathway	Acid and base tolerance to support more applications
New, faster pump (70 mL/min)	Faster extractions
Accommodates all sizes of cells (1, 5, 10, 22, 34, 66, and 100 mL)	Increased flexibility to meet sample requirements for many applications
	Accepts both 250- and 60-mL collection bottles and vials Safety shield over bottles and vials Dionium, chemical resistant pathway New, faster pump (70 mL/min) Accommodates all sizes of cells

### High Capability, Flexibility, Productivity

Dionex ASE 150 and 350 extractors deliver many advantages to laboratories requiring automated solid and semisolid extraction. The Dionex ASE 350 extractor handles 1–100 g of sample, combining the abilities of the previous Dionex ASE 200 and ASE 300 into a single system. It also includes an integrated solvent controller, reducing bench space requirements. Both the Dionex ASE 150 and 350 systems include a faster pump (70 mL/min), which means greater productivity for your laboratory.

### **ASE Performs Extraction in Minutes**

Other extraction techniques can take up to 48 h. With ASE, extractions are typically performed in 12– 20 min. See the Time Savings table for examples.

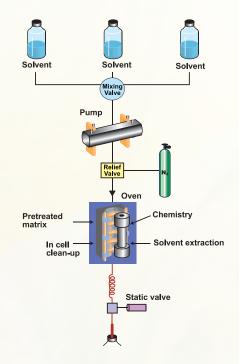
Time Savings						
Technique	Average Extraction Times*					
Soxhlet	4–48 h					
Automated Soxhlet	1–4 h					
Sonication	0.5–1 h					
SFE	0.5–2 h					
Microwave	0.5–1 h					
Dionex ASE 150/350 with in cell clean-up	0.2–0.3 h					

\*Extraction times are based on a per sample basis. This estimate does not include sample weighing, loading, or concentration, although it does include sample filtering, if necessary. In-cell cleanup is a technique used with ASE flow-through design, to add absorbent directly to the extraction cell that retains interferences. This technique further increases time savings.

Solvent Savings					
Technique	Solvent Usage*				
Soxhlet	150–500 mL				
Automated Soxhlet	50–100 mL				
Sonication	150–200 mL				
SFE	5–50 mL				
Microwave	25–50 mL				
Dionex ASE 150/350**	5–200 mL				

\*Solvent usage are based on a per sample basis. Additionally, ASE has many cells sizes to adapt to sample size requirements

\*\* Solvent saver mode provides further reduction in solvent consumption.



### **ASE Reduces Solvent Consumption**

Save 50–90% in solvent consumption when compared to other techniques. See the Solvent Savings table for examples.

### Performing ASE

Add solid or semisolid samples to the Dionex ASE Dionium extraction cell. Sample Pretreatment (optional) If necessary, pretreat samples by performing a hydrolysis step or adjusting pH of samples using acids or bases, so that net concentration of acid or base does not exceed 0.1 M. ASE automatically performs solvent extraction as follows:

- 1. Cell is filled with solvent (aqueous or organic).
- 2. Cell is heated and pressurized.
- 3. Sample is held at 1500 psi and desired temperature.
- 4. Fresh solvent is pumped through sample and entire pathway.
- 5. System is purged with nitrogen.

*In-Cell Cleanup (Optional)* Various resins can be layered in the extraction cell to retain interferences, providing a clean extract that is ready for analysis and requires no post-extraction cleanup step.

### **Benefits of Flow-Through Pathways**

- In-line filtration
- In-cell removal of interferences
- Sequential extractions with multiple solvents to remove interferences and then analytes
- Reproducible temperature control foreach extraction

# **Extraction Applications ASE Meets Your Application Requirements**

# **Environmental Analysis**

For environmental applications, ASE is proven to produce data equivalent to or better than traditional methods. ASE is accepted for use in EPA Method SW-846 3545 for the extraction of:

- Pesticides and herbicides.
- PAHs and semivolatile compounds
- PCBs

Trichloronat

Prothiofos

Merphos

Sulprofos

FPN

Fensulfothion

Azinphos-methyl

Coumaphos

Tetrachlorvinphos

- Dioxins and furans •
- TPH (DRO)
- Explosive compounds

ASE is also accepted for use in EPA Method SW-846 6860 for the determination of perchlorate and in CLP OLM 04.2 A for semivolatiles and pesticides. More recently, ASE has proven effective in extracting organic compounds from air using 3

Percent Recovery of Organophosphorus Pesticides from 30-g Apple Puree Sample Fortified at 50 ppb							
Pesticides	% Recovery	SD	RSD %				
Dichlorvos/Naled	87	10	12				
Nevinphos	100	12	12				
EPP	121	20	16				
)emeton-0	65	12	19				
Ethoprop)	95	11	12				
ulfotep	95	10	10				
horate	86	8	9				
emeton-S	59	11	18				
imethoate	128	19	15				
iazinon	93	9	10				
lisulfoton	63	11	18				
arathion-methyl	95	10	10				
enchlorphos	91	9	10				
lalathion	94	14	15				
enthion	86	7	8				
Chlorpyrifos	91	9	10				
arathion-ethyl	99	11	11				
		-					

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# **Food Analysis**

European and Asian government agencies have approved ASE for the determination of contaminants in food. ASE delivers optimal extractions for the following applications.

- · Determination of pesticide residues in a variety of sample types marketed for human or animal consumption
- Determination of lipids after acid hydrolysis
- Determination of fat and/or additive content of many food types in accordance with new and more stringent labeling requirements

• Determination of the flavor profile of consumer products With 100-mL extraction cells, ASE is capable of extracting large-gram weight samples with high moisture content, to support the large sample volumes required for low-detectionlimit food and beverage analyses. For agricultural products, ASE delivers automated extraction of residual pesticides (chlorinated and phosphorous), PCBs, and dioxins. and dioxins.

Analyte Class (EPA Analysis Method)	MDLa mg/kg	Accuracy (% Recovery of CRM)	Recovery as a % of Soxhlet	Precision (%RSD)
Organochlorine pesticides (8081) (average of 20 compounds)	0.5–3.2	66–84	75–105	3.2
PCBs (Aroclor 1254, 8082)	57–70	99	96.3	3.5
Total Petroleum Hydrocarbons (DRO, 8015)	5.1	104.1	NA	9.7
Organophosphorus Pesticides (8141) (average of 24 compounds)	18.9–171	56–72	90–111	16.3
Chlorinated Herbicides (8151) (average of 8 compounds)	22–261	36–69	101–118c	15.5
Semivolatiles (BNAs, 8270) (average of 56 compounds)	16–89	58–70	66–120	5.4
Dioxins (8280/8290)	Low ppt	73b	96b	4.24d

<sup>a</sup>Calculated as per SW-846 chapter 1. bAverage recovery of surrogates <sup>C</sup>Shaker method dAverage RSD of Congeners

n=12a

Extraction for Marker Compounds from Natural Products										
Dianthrones (St. John's Wort)		Deacylsaponins Silybin (Horse Chestnut) (Milk Thistle)		Curcumin (Tumeric Rhizome)		Thymol (Thyme)				
Method	Soxhlet	ASE	Soxhlet & Reflux	ASE	Soxhlet	ASE	Reflux	ASE	Steam Distillation	ASE
Percent wt. (RSD%)	0.028 (7.1) <sup>a</sup>	0.035 (2.9)	2.6 (12)	3.7 (5.4)	1.13 (3.5)	1.16 (3.4)	0.89 (2.2)	1.06 (0.94)	1.15 (7.0)	1.17 (3.4)
Solvents	DCM Acetone	DCM MeOH	DCM MeOH	DCM MeOH	Petrol MeOH	Hexane MeOH	MeOH	MeOH	Water	Hexane DCM
Volume	250 mL	<50 mL	170 mL	<50 mL	200 mL	<70 mL	50 mL	<20 mL	250 mL	<80 mL
Total Time	38 h	<25 min	7 h	<40 min	9 h	<25 min	1 h	<30 min	2 h	<25 min

### **Pharmaceuticals, Dietary Supplements**

For pharmaceuticals, natural products, and dietary supplements, ASE is used to:

- Extract natural products from plants.
- Verify that nutritional supplements such as St. John's Wort, echinacea, and ginkgo biloba meet industry guidelines for standardized marker compound levels.
- Monitor the level of pharmaceutical agents and their metabolites in animal tissues, and test stability.
- Verify that levels of active compounds in products such as transdermal patches are within specification.

For these types of applications, ASE saves time and increases efficiency, as shown in the table above. Automation and improved reproducibility result in better control of manufacturing processes and faster identification of potential therapeutic agents. The ability to extract samples with a variety of different polarity solvents results in the generation of selective extract profiles, which then can be screened more easily for activity.

Total Extractables from Styrene-Butadiene Rubber (SBR)									
Sample	Target Value	ASE Value (%)	ASE RSD (n = 3)	ASE Recovery (% vs. Target value)					
1	1 32.59 32.66		0.52%	100.2					
2	32.60	32.77	0.12%	100.5					
3	3 33.86 33		0.56%	100.1					
4	34.83	34.44	0.91%	98.9					

Extraction of Additives from Low Density Polyethylene (LDPE)								
Procedure	Additive Concentration (ppm)							
	I-3114	I-1010	I-1330	I-168	I-1076			
Chloroform dissolution	nd	95	599	659	205			
ASE	nd	95	598	694	154			

### **Chemical, Petrochemical, Polymers**

In the polymer industry, ASE is used to characterize polymer structures using ASTM method D-7210:

- Extraction of plasticizers from PVC
- Extraction of polypropylene and polyethylene for additives

such as UV stabilizers and antioxidants and slip agents

- Extraction of oils and organic acids from SBR samples
- Extraction of residual sugars in plant materials

ASE replaces 12–24-h solvent reflux extractions, significantly reducing extraction time and solvent exposure. ASE can also be used for competitive products analysis and routine product quality testing.

Weight Percent of Each Plasticizer in Poly Vinyl Chloride (PVC)									
Plasticizer	ASE Recovery (n=3)	Soxhlet Recovery (ASTM D2124) (n=2)	ASE Recovery (% vs. Target value)						
DOA	9.81	9.56	102.6						
TOP	9.50	9.28	102.4						
DOP	9.42	9.35	100.7						
TOTM	9.17	9.05	101.3						

Extraction of Additives	from High Density	Polyethylene (HDPE)
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Procedure	Additive Concentration (ppm)						
	I-1330	I-168	I-1076				
ASTM D-6953-11	353	132	nd	nd	240		
ASE	335	138	nd	nd	281		

# Sample Preparation ASE Goes Beyond Extraction to Deliver a Comprehensive Solution

Sample preparation is a critical link in the overall analytical process. It often requires many steps and multiple hours to prepare samples for Chromatography. To improve productivity and minimize turnaround time, sample preparation requires more than just speed of the solvent extraction step. The ability to integrate pretreatment, solvent extraction, and post-extraction cleanup are critical for total productivity. Integration of these steps improves overall productivity, reduces sample handling, and minimizes preparation time.

*Pretreatment:* Change in chemical or physical features of the sample prior to solvent extraction. This includes treatment of solid material with acids and bases.

Solvent extraction: Separating analytes from the solid matrix and placing them in a compatible solvent.

*Post-extraction treatment:* Includes sample cleanup to remove interferences using sorbent. Unlike other techniques, ASE integrates pretreatment, extraction, and post-extraction treatment, as shown below.

### **Applications with pH-Hardened Pathway**

The Dionium pathway supports a variety of applications that require sample pretreatment, such as:

- Determination of fats and lipids using acid hydrolysis
- Determination of fats and total lipids using alkaline saponification
- Determination of phenolic compounds in complex matrices
- Determination of chlorophenoxy acid herbicides in soils
- Determination of residual sugars in waste vegetation

Comparison of Extraction Techniques for Lipid (FAME) Determination Using GC/MS (n = 3)									
Mayonaise	Average	RSD	%RSD						
Mojonnier	75.1	0.89	1.18						
ASE	74.2	74.2 0.43							
Corn Chips	Average	RSD	%RSD						
Mojonnier	30.41	0.37	1.21						
ASE	29.85	0.33	1.10						
Parmesean Cheese	Average	RSD	%RSD						
Mojonnier	26.41	0.284	1.08						
ASE	26.27	0.220	0.839						
Baked Shortbread	Average	RSD	%RSD						
Mojonnier	13.95	0.033	0.238						
ASE	14.07	0.451	3.20						
Bologna Sample	Average	RSD	%RSD						
Mojonnier	25.58	0.275	0.968						
ASE	28.60	0.375	1.31						

Sample Preparation Comparison											
Technique	Soxhlet	Sonication horn	Bag Technology	SPE QuEChERS	Automated Soxhlet	Microwave	PLE	PSE	ASE 200 and 300	ASE 350	
Various sample sizes per batch	Limited	Yes	No	No	Limited	Limited	No	Limited	Limited	Yes	
Extraction of pretreated samples i.e. acid hydrolysis	Yes	No	Yes	No	Yes	Yes	No	No	No	Yes	
Automated rinsing of system	No	No	No	No	No	No	No	No	Yes	Yes	
Solvent extraction	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	
Solvent extraction and filtration	No	No	No	No	No	No	Yes	Yes	Yes	Yes	
Sequential extraction and flow-thru extraction technology	No	No	No	No	No	No	No	No	Yes	Yes	
Post-extraction (in-cell) cleanup	No	No	No	Yes	No	NO	Yes	No	Yes	Yes	

# **Choosing Dionex ASE** Your Choice Is No Longer Based Solely On Sample Size

### Which ASE is right for you?

The two Dionex ASE systems are designed to meet the extraction requirements of the full spectrum of laboratories performing solid and semisolid solvent extraction. Each Dionex ASE system uses elevated temperature and pressure to greatly accelerate extractions. Dionex ASE systems feature:

- Automated sample extraction using flow-through technology with pH-hardened pathways
- Automatic extract filtration
- Easy-to-fill sample cells with finger- or hand-tight fittings
- Easy-to-use collection vials and bottles
- Convenient front panel operation with multiple method storage
- Sensors for temperature, pressure, and solvent vapors ensure safe operation at all times
- Easy method transfer between systems
- Patented technology (patent numbers 5,843,311; 5,647,976; 5,660,727; and 5,785,856)
- Established methodologies
- Temperature range from ambient to 200 °C
- Samples cell sizes: 1, 5, 10, 22, 34, 66, and 100 mL
- Collection vial sizes: 60 or 250 mL
- Operating pressure: 1500 psi (100 bar)

## Dionex ASE 150 Accelerated Solvent Extractor

The Dionex ASE 150 is the entry-level ASE system designed for xuse in lower-throughput labs. This system offers fast and efficient extraction for a large range of sample sizes. Key features include:

- Automated extraction of a single sample
- Economically priced for lower throughput laboratories
- Sample cell sizes: 1, 5, 10, 22, 34, 66, and 100 mL
- Collection vial sizes: 60 or 250 mL
- Operating pressure: 1500 psi (100 bar)
- Small footprint requires less than 36 cm (14 in.) of bench space

### Dionex ASE 350 Accelerated Solvent Extractor

The Dionex ASE 350 is designed for high-throughput labs with large sample volume requirements. It is ideal for the busy environmental, biotech, pharmaceutical, chemical, food analysis, or research lab, supporting automation of up to 24 cells and sample cell volumes up to 100 mL. Key features:

- Unattended extraction of up to 24 samples
- Samples cell sizes: 1, 5, 10, 22, 34, 66, and 100 mL
- Collection vial sizes: 60 or 250 mL
- Operating pressure: 1500 psi (100 bar)
- Automatic rinsing of system between sample extractions
- Solvent saver mode for further reduction in solvent consumption
- Scheduling programming for automated method optimization
- Integration of ASE Solvent Controller into the dionex ASE 350 system
- Mixing or selection of three different solvent sources



Dionex ASE 150

Dionex ASE 350

Our goal is to provide you with solutions to your extraction challenges rather than just selling you an instrument. You get a complete solution, and a partner committed to your success.

To meet that goal, we offer a complete line of training, installation and warranty service, and comprehensive support programs.

#### **System Specifications**

### **Power Requirements**

Consumption: 500 VA watts max. Voltage: 100–120 or 220–240 VAC Frequency: 50/60 Hz

#### Pneumatic Requirements:

N2 at 1034–1340 kPa (150–200 psi) Air at 400–827 kPa (60–120 psi) (ASE 350 optional)

#### Dionex ASE 150

Dimensions (h x w x d): 56.1 x 35.6 x 50.8 cm (22.1 x 14.0 x 20.0 in.)

Weight:

# 34 kg (75 lb)

### Dionex ASE 350

Dimensions (h x w x d): 69.3 x 67.3 x 61.7 cm (27.3 x 26.5 x 24.3 in)

Weight:

65 kg (140.0 lb)

#### www.thermoscientific.com/dionex

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