Thermo Scientific

Dionex ASE Prep MAP

Product Manual

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Thermo SCIENTIFIC

Product Manual

for

Dionex ASE Prep MAP

Moisture Absorbing Polymer, 200 g (P/N 083475)

For Research Use Only. Not for Use in Diagnostic Procedures.

Revision History:

Revision 01, May, 2013, Original Publication.

Revision 02, June, 2013, Typo corrections.

Safety and Special Notices

Make sure you follow the precautionary statements presented in this guide. The safety and other special notices appear in boxes.

Safety and special notices include the following:

 **SAFETY**	***Indicates a potentially hazardous situation which, if not avoided, could result in death or serious injury.***
 **WARNING**	***Indicates a potentially hazardous situation which, if not avoided, could result in damage to equipment.***
 **CAUTION**	***Indicates a potentially hazardous situation which, if not avoided, may result in minor or moderate injury. Also used to identify a situation or practice that may seriously damage the instrument, but will not cause injury.***
 **NOTE**	***Indicates information of general interest.***
IMPORTANT	***Highlights information necessary to prevent damage to software, loss of data, or invalid test results; or might contain information that is critical for optimal performance of the system.***
Tip	***Highlights helpful information that can make a task easier.***

Contents

1. Introduction

Thermo Scientific™ Dionex™ ASE™ Prep MAP, moisture absorbing polymer (P/N 083475) is a consumable designed for sample to remove moisture from wet samples as well as from the solvent extract. MAP is a proprietary polymer which is a copolymer of an anionic and cationic monomer. This unique formulation allows moisture removal under a variety of ionic strength conditions and ASE extraction conditions. The polymer is a free flowing white granular material that can be easily mixed with Dionex ASE Prep DE, diatomaceous earth (P/N 062819) in a 1:1 ratio and used for the moisture removal applications under ASE extraction conditions.

Analyses of organic compounds is becoming increasingly important, and often with the need to isolate and analyze trace levels of compounds from a variety of matrices such as soil, sediment, animal tissue, fruits, and vegetables. Sample pretreatment constitutes an important step prior to analysis. The purpose of the sample pretreatment step is to selectively isolate the analytes of interest from matrix components and present a sample suited for routine analysis by established analytical techniques such as gas chromatography or high-pressure liquid chromatography. Typical sample pretreatment techniques include solid phase extraction, liquid-liquid extraction, solid-liquid extraction, dilution, evaporation, and distillation. Accelerated solvent extraction (ASE) is a technique used for extracting the analytes of interest from a solid, semisolid or an adsorbed liquid sample by performing extraction using an organic solvent and at elevated temperature and pressure. The elevated pressure elevates the boiling temperature of the solvent thereby allowing faster extraction to be conducted at relatively high temperatures. Thus the extraction process is significantly faster than traditional methods such as soxhlet extraction.

In some samples containing moisture or water such as soil samples or food samples (animal tissue, fruits, vegetables etc.) an additional step may be needed either before the extraction step or as a post extraction step to remove the moisture. Sample drying can be accomplished in several ways such as air drying and oven drying prior to extraction. However, these approaches are not suited when analyzing volatile or semi-volatile components as they would be removed from the sample prior to extraction or analysis.

Another common method for moisture removal is by using salts such as sodium sulfate, calcium chloride, magnesium sulfate, calcium sulfate and the like. These salts tend to associate to water molecules to form hydrated salts. Sodium sulfate for example tends to clump together when water is present.

Sodium sulfate is not suitable for in-cell moisture removal and extraction in ASE. Sodium sulfate can dissolve in hot solvent to a certain extent and can precipitate downstream in some instances clogging the outlet frit, tubes and valves. Moreover, sodium sulfate becomes an aggregate hard lump upon water absorption and is not easy to process during sample preparation for in-cell moisture removal and extraction.

The water absorbing ability of the Dionex ASE Prep MAP polymer is independent of the ionic strength of the sample matrix making it more suitable for sample preparation applications. The polymer absorbs water and the water is held in the polymer matrix by hydrogen bonding. The Dionex ASE Prep MAP has a high capacity for water removal and does not suffer from the limitations as outlined above for sodium sulfate salt.

2. Overview and Usage

This section describes detail information and usage of the Dionex ASE Prep MAP.

Moisture Removal Modes: The moisture removal polymer by itself can remove up to 5 g of water per gram of the polymer at room temperature. Addition of DE particularly when used in an ASE cell configuration results in improved removal of water under ASE extraction conditions. The water absorbing ability of the polymer increases with decreasing temperature. For example, at 100 °C 4 gm of MAP and 4 gm of DE can remove roughly 10 gm of water where as at room temperature about 2 gm of the polymer is adequate for this application.

The Dionex ASE Prep MAP polymer can be used in three modes.

1. In-cell Moisture Removal Mode:

   This mode is designed for inline moisture removal with the ASE instrument. The moisture absorbing polymer is combined with the Dionex ASE Prep DE preferably in a 1: 1 ratio to aid moisture removal. The addition of DE is recommended and results in improved flow and improved moisture removal. In this mode after the extraction is complete the collected solvent is expected to be free of moisture. The amount of Dionex ASE Prep MAP required can be estimated from the expected moisture content of the sample. Typically this mode is recommended for all samples where the extraction method is run at 125 ˚C and below. The benefit of this mode is there is no need to remove the moisture post extraction.

NOTE

It is important to grind the Dionex ASE Prep DE and mix this with Dionex ASE Prep MAP in a 1:1 ratio for use with a wet sample. The mixture along with the sample is loaded into the ASE cell for extraction.

2. In-vial moisture removal mode:

   This mode is designed for offline moisture removal such as with the collected solvent extract from the ASE extraction. The Dionex ASE Prep MAP polymer is added in this mode to the extract for moisture removal. The amount of polymer required can be calculated based on the estimated amount of moisture in the extract container. The amount of moisture absorbing polymer needed is 0.20 ± 0.05 g for absorbing one gram of water at room temperature. The extract can also dried by passing it through a bed of Dionex ASE Prep MAP placed on a filter paper. This mode is applicable to all ASE methods independent of temperature.

3. Combination mode:

   In this mode, the in-cell moisture removal is followed by in-vial moisture removal. If some break thru of moisture is observed in the extract during extraction then addition of a small amount of polymer in the collection bottle can result in complete moisture removal. In fact a small amount of the Dionex ASE Prep MAP polymer in the collection vessel always ensures that there would be no moisture present in the samples. This mode is particularly useful for samples with unknown moisture content and for extractions occurring above 125 ˚C. The mode is also recommended for use with water containing solvents.

2.1 Amount of MAP and DE Needed for Moisture Removal Under ASE Conditions

Table 1 shows moisture removal for a variety of cells under three different temperatures. The maximum water removal numbers for the various temperatures and cell sizes are also shown. The maximum water removals at the various temperatures are summarized below. In general for temperatures below 125 ˚C the in-cell mode is preferred. For temperatures above 125 ˚C the in-vial mode is recommended.

A. Maximum water removal at 100 ˚C is approximately 15 g
B. Maximum water removal at 125 ˚C is approximately 8 g
C. Maximum water removal at 150 ˚C is approximately 2 g
D. Maximum water removal at 175 ˚C and 200 ˚C is approximately 0.5 g

Table 1 In-Cell Moisture Removal by Using MAP and DE at Various Temperatures and Cell Sizes in ASE.

ASE extraction temperature, °C	Total water present in the cell, g	Amount of MAP, g	Amount of DE, g	Cell size, mL
100	5.05	2	2	34
125	2.54	2	2	34
150	2.15*	2	2	34
100	10.0	4	4	66
125	5.05	4	4	66
150	2.09*	4	4	66
100	15.1*	6	6	100
125	8.14*	6	6	100
150	2.14*	6	6	100

*Maximum water removal

Table 2 shows the amount of polymer required for in cell moisture removal with ASE under a variety of common temperatures. It is clear that as the temperature increases the water removal and the maximum water removal decreases.

Table 2 Polymer and DE Amounts Required for In-Cell Moisture Removal at Various Temperatures

Temperature °C	Amount of Polymer required per gm of water	Amount of DE required per gm of water
100	0.4 g	0.4 g
125	0.8 g	0.8 g
150	1.0 g	1.0 g

An example of a high moisture content sample is shown in Table 3. The moisture content of the sample is roughly 84%. The amount of MAP needed can be calculated based on the water content and using the information in Table 2. For example at 100 ˚C, the amount of polymer needed to remove 2 gm of water is roughly 0.8 gm.

Amount of MAP in gms = 0.4 * amount of moisture present in the sample in gms at 100 ˚C. Table 3 also provides some guidelines on the cell size needed for a given sample size.

CAUTION

The MAP may be harmful on contact with skin.
Use safety glasses, goggles and/or a face shield to protect eye.
Wear air purifying respirator to protect lungs if dust is generated.

Table 3 In-Cell Moisture Removal by the MAP and DE at 100 °C for Different Cell Sizes in ASE

Sample size, g	Water content, g	Amount of MAP needed, g	Amount of DE needed, g	Approximate volume occupied by the sample and the drying agents, mL	Recommended cell size to be used in ASE, mL
1.0-2.0	0.84-1.68	0.33-0.67	0.33-0.67	6-10	10
2.1-3.5	1.76-2.94	0.71-1.18	0.71-1.18	11-21	22
3.6-6.0	3.02-5.04	1.21-2.01	1.21-2.01	22-33	34
6.1-12.0	5.12-10.1	2.05-4.04	2.05-4.04	34-64	66
12.1-18.0	10.2-15.1	4.07-6.05	4.07-6.05	65-95	100

2.2 Moisture Removal Under High Ionic Strength Conditions

The moisture removal capacity of the Dionex ASE Prep MAP polymer was measured at room temperature with and without added salt solution (Table 4). The moisture removal capacity of the Dionex ASE Prep MAP was substantially unaffected by the salt concentration as shown in Table 4. In contrast, other moisture absorbing polymers that were polyacrylate based showed a lower moisture absorbing ability as the salt concentration increased. These polymers are also not suitable for extraction under ASE conditions since they give out water. In contrast the Dionex ASE Prep MAP is ideally suited for moisture removal under ASE conditions. Further the water absorbing ability of the Dionex ASE Prep MAP polymer is independent of the ionic strength of the sample matrix making it more suitable for sample preparation applications under ASE conditions.

Table 4 Moisture Removal Capacity of the MAP Versus a Commercial Polyacrylic Acid Based Polymer at Room Temperature.

Type of polymer	Polymer needed to absorb one gram of water, g	Polymer needed to absorb one gram of 2.91% NaCl solution (sea water concentration), g	Polymer needed to absorb one gram of 26.5% NaCl solution (saturation level concentration), g
MAP	0.20	0.19	0.18
Commercial Polymer that is Polyacrylate based	0.04	0.08	0.27

2.3 Samples

Typical samples such as wet soil, sediments and animal tissue based samples such as meat, fish, and food samples can be dried by using the MAP and DE for the in-cell mode (See section 2.1 and US EPA Method 3545, 1668 and 1699).

NOTE

Use the Tables in section 2.1 to determine the sample size.

2.4 Choice of Solvent

The Dionex ASE Prep MAP polymer is recommended for in cell moisture removal ASE applications from samples that are extracted using non polar solvents such as Hexane, dichloromethane, and combination of polar and nonpolar solvents. The polymer is not recommended for in cell moisture removal with polar solvents.

Typical solvents used along with the extraction temperatures are listed below.

A. Thermo Fisher Dionex App note 317: Base, neutral and acidic compound, volatile organic compound from soil, solvent- dichloromethane: acetone (1:1) at 100 °C.
B. Thermo Fisher Dionex App note 313: Polycyclic aromatic hydrocarbons from soil, solvent- dichloromethane: acetone (1:1) at 100 °C.
C. Thermo Fisher Dionex App note 320: Pesticide from soil/sediment, solvent- hexane: acetone (1:1) at 100 °C.
D. Thermo Fisher Dionex App note 334: Fat in meat, solvent- hexane or petroleum ether at 125 °C.
E. Thermo Fisher Dionex App note 332: Pesticide from Food, solvent- hexane: acetone (9:1) at 100 °C.

NOTE

The water removal efficiency may be compromised when using polar solvents such as methanol, ethanol, and acetone. Preliminary experiments to determine the moisture removal capacity by optimizing the polymer amount may be needed.

NOTE

The user can use in-vial moisture removal approach for any solvent composition containing acetic acid.

2.5 Example Application

2.5.1 Materials Needed for Sample Preparation

A. Test Sample (Oyster sample)
B. ASE cell
C. ASE Prep MAP
D. ASE Prep DE
E. Collection bottle
F. Analyte standard and spike solution
G. Mortar and pestle
H. Glass beaker, spoon, spatula etc

2.5.2 In-Cell Moisture Removal of an Oyster Sample

Sample preparation is challenging for a wet animal tissue sample such as an Oyster sample. The presence of water in such a sample can result in poor recoveries of the analyte of interest. A drying step is therefore needed before the extraction. In the current method a mixture of six organochlorine pesticides was spiked on a wet Oyster sample. The spike level was 500 ng/g of sample. The spiked oyster sample was either treated with MAP and DE as per the present method or by using sodium sulfate pursuing an in-cell extraction in ASE. The extraction was pursued at 100 °C using hexane: acetone (1:1) as solvents (following Thermo Scientific Dionex App Note 320). The extracts were analyzed by GC-ECD. The results are shown in Table 5. The results indicated that the MAP and DE were effective as a drying agent for a very wet animal tissue sample such as Oyster and excellent recoveries were achieved for the six organochlorine pesticides. In contrast the sodium sulfate treated sample showed poorer recoveries.

Table 5* In-Cell Moisture Removal of Oyster Sample by MAP And DE, and Sodium Sulfate at 100 °C In ASE Using Hexane: Acetone (1:1) as an Extracting Solvent.

Compound	Oyster (n = 3) dried with MAP and DE		Oyster (n = 3) dried with sodium sulfate**	
	Recovery %	Std error	Recovery %	Std error
Lindane	91	5.6	81	3.1
Heptachlor	93	8.5	64	1.5
Aldrin	94	4.7	66	2.8
Dieldrin	105	2.7	75	4.6
Endrin	106	4.7	70	5.0
DDT	114	5.6	69	2.4
Overall	101	5.3	71	3.2

* Data is courtesy of Department of Toxicology, Texas Tech University, Lubbock, TX, USA.

** In-cell drying with sodium sulfate is not recommended using the ASE

2.6 Sample Preparation Procedure

The general sample preparation steps are listed below.

A. Prepare the appropriate extraction cell; by fixing the bottom cell end cap and installing either two glass fiber filters (P/N 056781) or two cellulose filters (P/N 056780) depending on the extraction solvent (s).

B. Add the required amount of the Dionex MAP polymer and Dionex DE as outlined in section 2.1 into a mortar. The polymer and the DE should be ground to a powder form using a mortar and pestle.

C. Place an aluminum funnel on top of the cell to aid in the loading process. Add a small plug of the MAP/DE mixture from step b to the bottom of the cell. This layer ensures a reservoir of capacity to capture any breakthrough of moisture from the samples during extraction.

D. Mix the sample prior to sampling to ensure a homogenous distribution. Accurately weigh the sample in a beaker or in a mortar. Spike analytes and surrogates to the sample, by dispensing the liquid slowly and evenly, drop by drop directly onto the sample. **Note: Do not spike the wall of the container or glass surface of the beaker.**

E. Add the MAP/DE from step b) into the mortar containing the sample in step d. Mix the wet sample and the drying agents thoroughly by a spatula or by a pestle.

F. Load the sample and drying agent mixture into the cell. For improved accuracy the residual sample in the containers and handling spatula can be removed. For example, add small amount of DE to take out any residual sample from the beaker or mortar and load into the ASE cell. Rinse the beaker or mortar with 1-2 mL of extraction solvent and add it to the ASE cell. Rinse the spatula with 0.5-1.0 mL of extraction solvent and add it to the ASE cell.

G. Close the top end cap of the ASE cell and the cell is now ready for extraction.

H. Proceed with extraction, evaporation and concentration of the extract, clean up (if any) and analysis.

NOTE

Sample preparation is recommended to be performed in the fume hood.

NOTE

Use Tables in section 2.1 for the sample size, drying agent and corresponding cell size information.

2.7 Cleaning the Cell After the Extraction:

A. Remove the top end cap after extraction. Using a squirt bottle add water or solvent to remove any residual particles. Dry the end cap with a paper towel.

B. Invert the cell on a waste (or collection) container and tap the cell body with 'filter insertion tool' slowly and the used sample with drying agents will come out of the cell. Use a lab spatula or filter insertion tool to force out the residual mass.

C. Remove the bottom (other) end cap.

D. Remove all the residual mass plus filter from the cell.

E. Rinse the cell body (inside) with water or deionized water thoroughly and dry it with paper towel. Blow with pressurized lab air/ N_2, if necessary.

3. Troubleshooting

3.1 Low Recovery

A. Check the sample. Generally older samples tend to give lower recovery once they are opened and left under ambient conditions due to degradation. Use a fresh container of the sample when possible.
B. Check for any instrument leaks.
C. Check the evaporation temperature and ensure that is within the recommended values.
D. For better accuracy rinse with extracting solvent the sample handling containers such as beakers, mortar etc and tools such as pestle spatula etc.

3.2 Water In the Extract

A. Check the sample preparation steps and ensure the required amounts of the drying agents are used.
B. Increase the plug of drying agents at the bottom of the cell, if necessary.
C. Check the moisture content of the DE (Moisture specification = 1% (w/w)) and Dionex ASE Prep MAP (Moisture content is ≤ 3% (w/w)). Dry the MAP and DE in the oven, if the moisture content is outside these specifications.

NOTE

Use product manual of ASE 350, 150, 300, 200 and 100 for troubleshooting hints with any ASE related issue.