



Empore™ Extraction Disks

January 1996

Method Summary

FOOD APPLICATION

Extraction of Lead from Maple Syrup

Introduction

Solid phase extraction using Empore™ chelation disks greatly simplifies the analysis of high-solid liquid food products such as maple syrup for lead and other transition metals. The chelation disk (paired iminodiacetate ions on a styrene divinylbenzene support) selectively extracts and concentrates trace levels of metal ions while the high concentrations of sugars pass through unretained. The metals are eluted with a small volume of acid and can be directly analyzed by Atomic Adsorption (AA) or Inductively Coupled Plasma Spectroscopy (ICP).

The analysis of high solids containing liquids for metal ions is normally complicated because the metals must be separated from the dissolved organic solids. This typically involves ashing the sample to destroy the organic solids (sugars) or digesting the sample with strong acid at elevated temperatures. Sample preparation with the chelation disk is as simple as diluting with reagent water and adjusting the pH. The extraction step is a filtration through the sorbent disk with subsequent elution using an appropriate acid solution.

Method Summary

An aliquot of maple syrup is diluted and acidified with nitric acid. The sample is passed through a 47mm chelation disk followed by a reagent water wash. The disk is then eluted with nitric acid, diluted to a 15 mL final volume and analyzed by ICP.

Method Performance

Preliminary trials indicate the chelation disk is effective for concentration of lead from maple syrup. The data summarized herein is from limited trials using fortified reagent water and maple syrup samples.

	Spike Amt (µg/L)	Amt Detected (µg/L)	% Recovery
Lab Water Blank	N/A	<.05	N/A
Fortified Water 1	15	11.6	77
Fortified Water 2	15	11.3	76
Maple Syrup Blank	N/A	88	N/A
Fortified Syrup 1	100	165	77 ¹
Fortified Syrup 2	100	171	83 ¹

Est. Detection Limits 10-20 µg/L

¹ Recovery corrected for background levels

N/A = not applicable

Method Summary

The method below outlines the procedure used in the preliminary experimentation. It is intended for guidance only. The chelation disk is a hydrophilic medium. It is not necessary to condition the material with methanol prior to extracting and is desirable to allow the disk to go dry between steps.

When performing metals analyses with the chelation disks, metal-free extraction apparatus should be used. If glass is used, assure thorough rinsing with acid prior to extractions. Do not use the stainless steel (or PTFE-coated) support screens.

1. Place the chelating disk on the base of the extraction apparatus. Preswell the polymer-based chelation disk by wetting with acetone. Place the reservoir atop the acetone-wetted disk and clamp in place. Apply a vacuum to remove residual acetone.
2. **Prewash** – Wash the disk with 40¹ mL of 5M nitric acid. Wash excess acid from disk with two 40¹ mL washes of reagent water.
3. **Convert to Ammonium Form** – Add 10 mL 2M ammonium hydroxide to reservoir. Allow the disk to soak for a minute, then draw the ammonium hydroxide through. Wash twice with 40¹ mL aliquots of reagent water, then apply 5 mL of 1M ammonium hydroxide. Wash again with 40¹ mL reagent water.
4. **Sample Preparation** – Acidify maple syrup (150 grams) with 0.5 mL 5M nitric acid prior to adding spiking standard. Mix well and adjust to a volume of 380 mLs with reagent water. The dilution decreases the sample viscosity and allows better flows.

Note: When making spiked samples, acidify the matrix prior to adding lead solution.

Add 0.5 mL 1M ammonium acetate to all samples. Adjust the sample pH to 5.4 with 2M ammonium hydroxide or 2M nitric acid just prior to extracting.

5. **Extraction** – Pour the sample into the extraction glassware and apply full vacuum. When the extraction is complete, rinse the disk with two 40¹ mL aliquots of reagent water to remove residual matrix materials.
6. **Elution** – Using acid-washed glass receivers, elute the disk by applying 7 mL 5M nitric acid. Repeat using a second 7 mL aliquot of acid. (Elution volumes may be adjusted in order to keep the total volume below the final volume required for the instrumentation.)
7. **Analysis** – Adjust the final volume as desired and proceed to ICP or AA analysis.

¹Aliquots used for washes in this protocol were 40 mLs. From experience, 10 mL wash aliquots are generally sufficient.

Important Notice to Purchaser:

All statements, technical information and recommendations contained in this literature are based on tests conducted with 3M-approved equipment and are believed to be reliable. However, the accuracy or completeness of the tests are not guaranteed. THE FOLLOWING IS MADE IN LIEU OF ALL WARRANTIES, EXPRESS OR IMPLIED, INCLUDING THE IMPLIED WARRANTIES OF MERCHANTABILITY AND FITNESS FOR A PARTICULAR PURPOSE: The seller's and manufacturer's only obligation will be to replace the quantity of the product proved to be defective. Neither the seller nor 3M will be liable for any injury, loss or damage, direct or consequential, arising out of the use of or the inability to use the product. Before using, the user must determine the suitability of the product for his or her intended use.

3M

New Products Department
3M Industrial and Consumer Sector

3M Center, Building 220-9E-10
St. Paul, MN 55144-1000
800 328 5921



Recycled paper
40% pre-consumer
10% post-consumer

Printed in U.S.A.
© 3M 1995 78-6900-7360-2 (PRF224)