

In-Cell Cleanup Using ASE: Tips in Selection of Sorbents and Solvents



David Knowles, Bruce Richter, Eric Francis, Brian Dorich, Brett Murphy, Richard Carlson, and Jennifer Peterson, Dionex Corporation, Salt Lake City, UT, USA

INTRODUCTION

As laboratory analytical techniques become faster and more efficient, the sample preparation steps can become a bottleneck, specifically, the postextraction cleanup step. For example, lipid compounds not removed from the collected extract can have many adverse effects when injected onto a chromatographic column. To prepare samples from many matrices (soils, sediments, marine tissues), a gel permeation chromatography (GPC) cleanup is a common technique. While effective, this step can be time consuming. The work shown here demonstrates a method for performing cleanup inside the extraction cell using Accelerated Solvent Extractor (ASE[®]) systems.

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Figure 1. Dionex ASE[®] 150 and ASE 350 instruments.

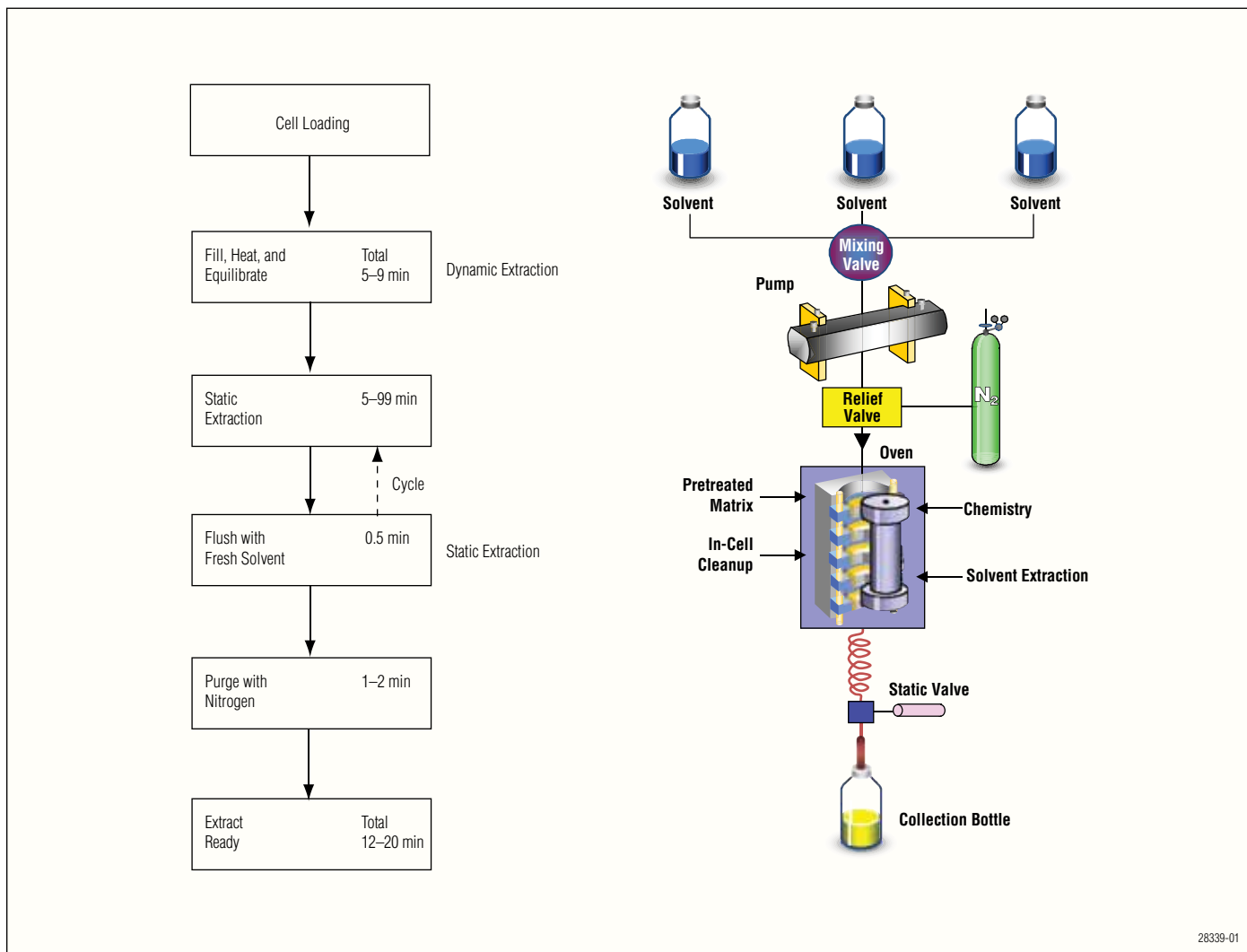


Figure 2. Automated solvent extraction schematic.

WHY DO WE WANT TO SELECTIVELY EXTRACT ANALYTES?

- To remove interferences
 - Improves peak identification
- To improve sensitivity
 - Allows trace analysis
- To accelerate overall sample preparation step
 - Eliminates GPC cleanup step
- Instrumentation concerns
 - Removes lipids before inject into mass spectrometer

WAYS TO AFFECT SELECTIVITY USING ASE

- Solvents
- Adsorbents

SOLVENT EFFECT ON SELECTIVITY

- Selective extractions
 - Blueberry extracts using ASE systems
 - Same sample extracted with different solvents
 - Fractionation of analytes
 - Elimination of interferences

EXTRACTION OF BLUEBERRIES USING DIFFERENT SOLVENTS

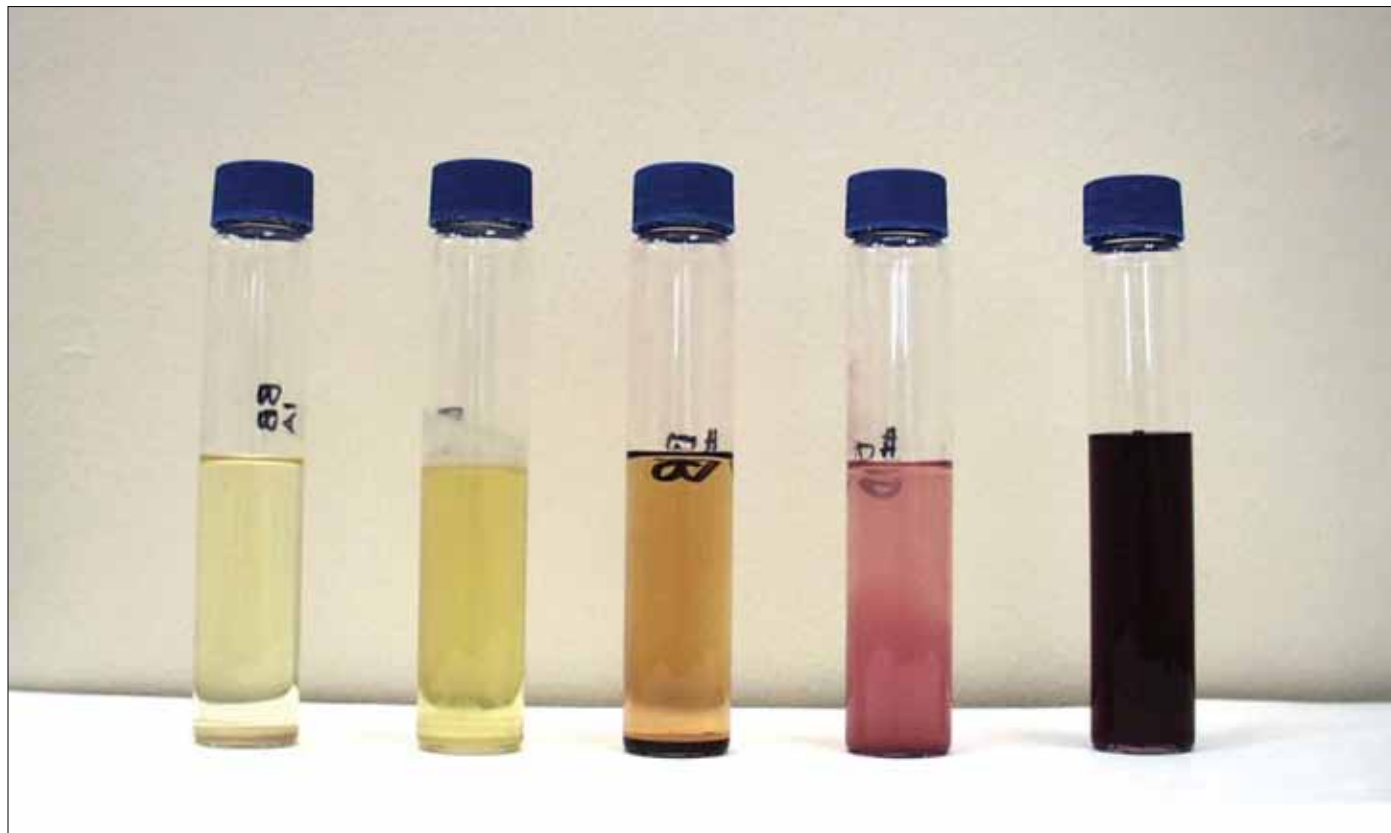


Figure 3. Hexane, DCM, Ethyl Acetate, Acetonitrile, and Ethanol.

Table 1. Adsorbents for In-Cell ASE Cleanup

Adsorbent	Interferences Removed	Analyte Group
Carbon	Organics	Dioxins
Copper	Elemental sulfur	Multi-residue pesticides
C18 Resins	Organics, lipids, and chlorophyll	Sulfonamides
Ion-Exchange Resins	Organics, metals, and ionic interferences	Anions, cations, and metal speciation
Acid-Impregnated Silica Gel	Lipids and oils	PCBs and PBDEs
Alumina	Lipids, chlorophyll, petroleum, and waste	Amines, perchlorates, and PCBs
Florisil™	Oils, lipids, and waxes	Pesticides and aromatics

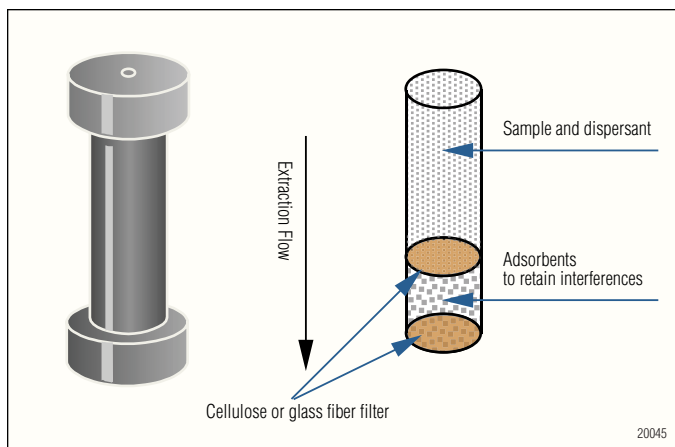


Figure 4. Schematic of in-cell ASE cleanup.

EXTRACTION OF PERCHLORATE IN SOIL WITH IN-CELL CLEANUP

ASE System Method

- ASE 200 system with 33 mL cells
 - Glass fiber filters
 - Absorbent layered in bottom of the cell
 - OnGuard® resins: RP
 - 1–5 g sample
 - Solvent DI water (18 MΩ-cm)
 - Temp 80 °C
 - Three 5-min static cycles
 - 25 min extraction time
- Extracts analyzed by IC/MS

INTEGRATED ASE EXTRACTION AND CLEANUP: PBDES IN SALMON



Figure 5. Extracts with and without in-cell cleanup of fish tissue using alumina, silica gel, and acidic silica gel (40% H_2SO_4).

Sonication Method

- Sonication bath
 - 1 g sample
 - 10 ml DI water(18 MΩ-cm)
 - Vortex 1 min
 - Sonicate 30 min
 - Centrifuge 5 min
 - Filter supernatant through 0.45 μm filter
 - Perform C18 column cleanup
- Extracts analyzed by IC/MS

ANALYSIS OF PERCHLORATE BY ION CHROMATOGRAPHY

LESS INTERFERENCE PROVIDES GREATER SENSITIVITY

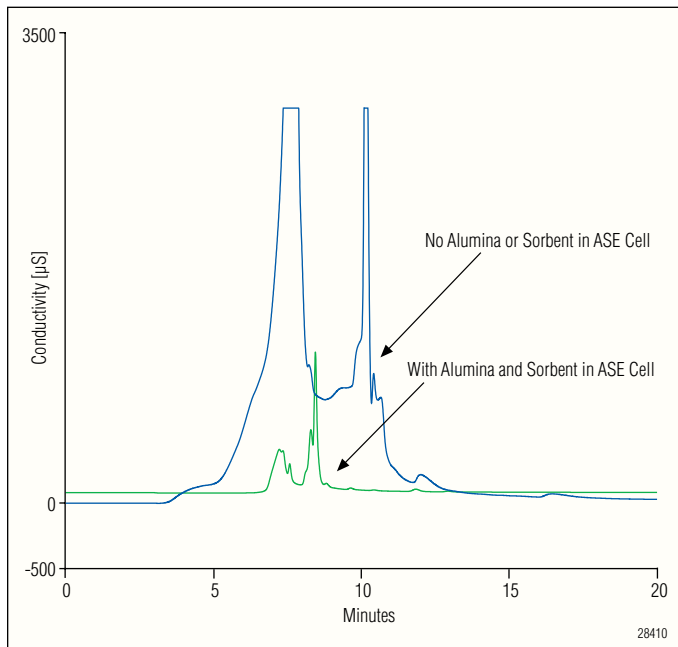


Figure 6. Effect of alumina and sorbent mixture on chromatography.

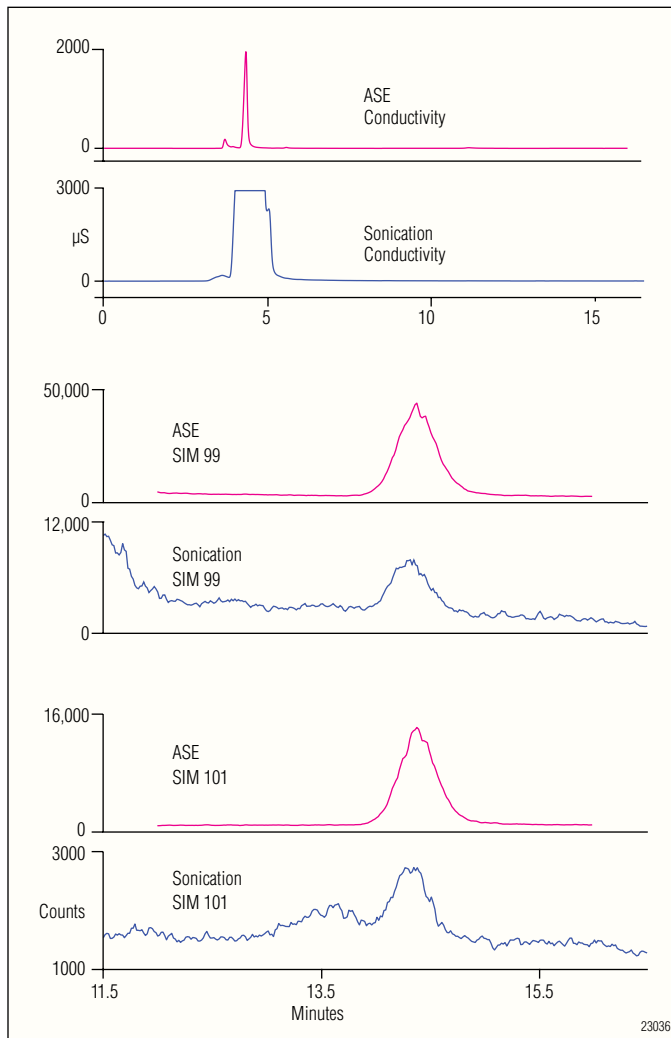


Figure 7. ASE system vs sonication; analysis of perchlorate in soil using IC and IC/MS.

CONCENTRATION OF PERCHLORATE IN $\mu\text{g/Kg}$

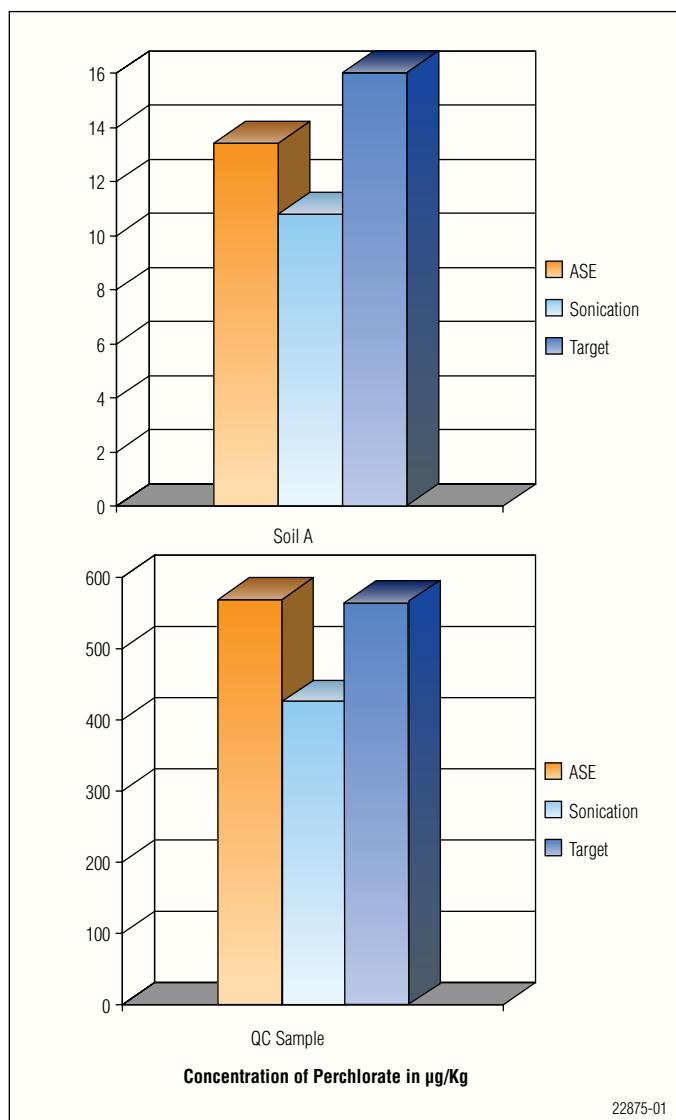


Figure 8. Perchlorate recovery: ASE system with in-cell cleanup vs sonication with off-line cleanup.

CONCLUSION

- Cleanup of unwanted interferences can be performed in-cell, eliminating additional sample handling and time.
- GPC cleanup for samples containing lipids can be reduced or eliminated, saving overall sample preparation time and costs.
- Sorbents can be used to provide cleaner extracts.
- Careful solvent choice can aid in selectivity of analytes being extracted.
- In-cell cleanup allows for complete automation of extraction and postextraction cleanup steps.
- This provides savings in overall costs and preparation time, and increases sample throughput.

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Dionex Corporation

1228 Titan Way
P.O. Box 3603
Sunnyvale, CA
94088-3603
(408) 737-0700

North America

U.S./Canada (847) 295-7500

South America

Brazil (55) 11 3731 5140

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Austria (43) 1 616 51 25 Benelux (31) 20 683 9768; (32) 3 353 4294
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