

Extraction of BNAs (Bases, Neutrals, and Acids) Using Accelerated Solvent Extraction (ASE)

Meets the requirements of U.S. EPA Method 3545

INTRODUCTION

Accelerated Solvent Extraction (ASE[®]) is a new extraction method that significantly streamlines sample preparation. A commonly used solvent is pumped into an extraction cell containing the sample, which is then brought to an elevated temperature and pressure. Minutes later, the extract is transferred from the heated cell to a standard collection vial for cleanup or analysis. The entire extraction process is fully automated and performed in minutes for fast and easy extraction with low solvent consumption.

Previously, the extraction of environmental materials such as soils, sludge, and other solid wastes required large amounts of solvents. Soxhlet, for example, uses from 250 to 500 mL of solvent for most environmental samples. Recent and anticipated changes in environmental regulations will severely restrict the amount of solvent usage in laboratories worldwide. For example, a recent U.S. executive order calls for major reductions of solvent usage in all federal laboratories. ASE was developed to meet the new requirements for reducing solvent usage in the preparation of solid waste samples.

The use of ASE for the extraction of BNA compounds from solid wastes provides a more convenient, faster, and less solvent-intensive method than previously available. Recoveries of these analytes by ASE are equivalent to recoveries from other more solvent-intensive methods such as Soxhlet. ASE also avoids the problem of localized heating and multiple washing procedures associated with sonication. ASE can extract a 10 g sample of a typical soil in about 12 min with a total solvent consumption of approximately 15 mL.

The procedures described in this application note meet the requirements for sample extraction as determined by U.S. EPA Method 3545. This method is applicable to the extraction of water-insoluble or slightly water-soluble volatile and semivolatile compounds in preparation for gas chromatographic or GC/MS measurement. This method is applicable to soils, clays, wastes, and sediments containing 250–12,500 $\mu\text{g}/\text{kg}$ of BNA compounds.

EQUIPMENT

ASE 200 Accelerated Solvent Extractor* with 11 or 22 mL stainless steel extraction cells
GC or GC/MS

Dionex vials for collection of extracts (40 mL, P/N 49465; 60 mL, P/N 49466)

**ASE 150 and 350 can be used for equivalent results.*

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SOLVENTS

Dichloromethane

Acetone

ASE 200 CONDITIONS

Oven Temperature:	100 °C
Pressure:	1500 psi*
Oven Heatup Time:	5 min
Static Time:	5 min
Flush Volume:	60% of extraction cell volume
Solvent:	Dichloromethane/acetone (1:1 v/v)
Nitrogen Purge:	1 MPa (150 psi) for 60 s

**Pressure studies show that 1500 psi is the optimum extraction pressure for all ASE applications.*

SAMPLE INFORMATION

Spiked samples were extracted both by the ASE 200 system and by a Perstorp Environmental Soxtec® (automated Soxhlet). Extracts were analyzed by SW-846 Method 8270A.

Note: All extractions and analytical work were performed by Mountain States Analytical, Inc. (Salt Lake City, Utah, USA). Matrix blanks, spikes, and spike duplicates were included for the low-level spikes; matrix spikes were included for all other concentrations.

SAMPLE PREPARATION

Spiking concentrations ranged from 250 to 12,500 µg/kg for the semivolatiles (BNA compounds). All spiked soils were prepared and certified by ERA (Environmental Resource Associates). Samples were ground to 100–200 mesh (150–75 µm). Wet samples were either mixed with ASE Prep DE (diatomaceous earth), P/N 062819, (10-g sample to 10-g ASE Prep DE) or air dried.¹ After grinding, a weighed sample was transferred to either an 11- or 22-mL extraction cell.

PROCEDURE

The procedure used in this application note follows the detailed method as described under the U.S. EPA SW-846 Method 3545.

Mix sample thoroughly, especially composite

samples. Dried sediment, soil, and dry waste samples should be ground or otherwise subdivided so that they can pass through a 1-mm sieve. Introduce a sufficient amount of sample into the grinding apparatus to yield at least 10 to 20 g after grinding. Air dry the sample at room temperature for 48 h in a glass tray or on hexane-cleaned aluminum foil, or dry the sample by mixing with ASE Prep DE until a free-flowing powder is obtained. Samples containing high boiling components may be dried by mixing with ASE Prep DE until a free-flowing powder is obtained. Air drying is not appropriate for the analysis of the more volatile organochlorine pesticides (e.g., the BHCs) or the more volatile of the semivolatile organics because of losses during the drying process.

Gummy, fibrous, or oily materials not amenable to grinding should be cut, shredded, or otherwise separated to allow for mixing and maximum exposure of the sample surfaces for extraction. If grinding of these materials is preferred, the addition and mixing of ASE Prep DE with the sample (1:1, w/w) may improve grinding efficiency.

For sediment and soils (especially gummy clay) that are moist and cannot be air-dried because of loss of volatile analytes, mix 5–10 g of sample with an equal amount of ASE Prep DE in a small beaker using a spatula. Use this approach for any solid sample that requires dispersion of the sample particles to ensure greater solvent contact throughout the sample mass.

Place a cellulose disk at the outlet end of the extraction cell. Weigh approximately 10 g of each sample into an 11-mL extraction cell or approximately 20 g into a 22-mL extraction cell. For samples mixed with ASE Prep DE, transfer the entire contents of the beaker to the extraction cell. Surrogate spikes and matrix spikes may be added to the appropriate sample cells.

Place extraction cells into the autosampler tray and load the collection tray with the appropriate number (up to 24) of 40-mL, precleaned, capped vials with septa. Set the method conditions on the ASE 200 system and initiate the run.

Collected extracts will be approximately 13–15 mL from the 11-mL extraction cells and 26–30 mL from the 22-mL size cells. The extract is now ready for cleanup or analysis depending on the extent of interfering coextractives.

DISCUSSION AND RESULTS

Examples of extraction of selected environmental samples are shown in this application note. This study illustrates the effectiveness of the ASE technique in obtaining recoveries of analytes equivalent to Soxtec. Results are summarized in Tables 1 and 2 for BNA compounds at three different spiking levels in three different soil types that were extracted according to the method presented.² ASE recoveries and RSD (%) values were all within the range expected from Soxhlet extractions.

Table 2. Average RSD (%) for BNAs for Three Soil Types

Matrix	ASE	Automated Soxhlet
Clay	9.1	2.5
Loam	16.1	4.7
Sand	13.4	7.4

Table 1. Average Recovery of BNAs from Three Soil Types^a—ASE Compared to Automated Soxhlet²

BNA Target Compound	Average Recovery (% of Soxhlet)	BNA Target Compound	Average Recovery (% of Soxhlet)
Phenol	102.0	2,6-Dinitrotoluene	90.3
bis(2-Chloroethyl)ether	101.9	Acenaphthalene	101.7
2-Chlorophenol	101.6	3-Nitroaniline	92.9
1,3-Dichlorobenzene	120.6	Acenaphthene	98.4
1,4-Dichlorobenzene	119.2	4-Nitrophenol	75.6
1,2-Dichlorobenzene	112.5	2,4-Dinitrotoluene	90.7
2-Methylphenol	104.7	Dibenzofuran	98.8
bis(2-Chloroisopropyl)ether	100.2	4-Chlorophenyl-phenylether	94.4
<i>o</i> -Toluidine	110.3	Fluorene	95.4
<i>N</i> -Nitroso-di- <i>n</i> -propylamine	98.1	4-Nitroaniline	99.1
Hexachloroethane	118.6	<i>N</i> -Nitrosodiphenylamine	96.8
Nitrobenzene	100.2	4-Bromophenyl-phenylether	95.8
Isophorone	101.7	Hexachlorobenzene	93.7
2,4-Dimethylphenol	109.8	Pentachlorophenol	81.2
2-Nitrophenol	96.3	Phenanthrene	100.6
bis(Chloroethoxy)methane	97.2	Anthracene	101.2
2,4-Dichlorophenol	98.6	Carbazole	99.1
1, 2,4-Trichlorobenzene	104.2	Fluoranthene	102.7
Naphthalene	106.1	Pyrene	105.2
4-Chloroaniline	108.1	3,3'-Dichlorobenzidine	116.5
Hexachlorobutadiene	104.7	Benz[<i>a</i>]anthracene	101.5
4-Chloro-3-methylphenol	99.7	Chrysene	102.9
2-Methylnaphthalene	102.1	Benzo[<i>b</i>]fluoranthene	99.0
Hexachlorocyclopentadiene	75.8	Benzo[<i>k</i>]fluoranthene	103.1
2,4,6-Trichlorophenol	95.9	Benzo[<i>a</i>]pyrene	99.9
2,4,5-Trichlorophenol	94.1	Indeno[1,2,3- <i>cd</i>]pyrene	102.7
2-Chloronaphthalene	96.2	Dibenz[<i>a,h</i>]anthracene	98.6
2-Nitroaniline	92.6	Benzo[<i>g,h,i</i>]perylene	66.4

^aAverages from extraction of sand, loam, and clay soils

REFERENCES

1. U.S. Environmental Protection Agency. *U.S. EPA Method 60014-81-055*, "Interim Methods for the Sampling and Analysis of Priority Pollutants in Sediments and Fish Tissue," Section 3.1.3.
2. Richter, B.; Ezzell, J.; Felix, D. "Single Laboratory Method Validation Report: Extraction of TCL/PPL (Target Compound List/Priority Pollutant List) BNAs and Pesticides Using Accelerated Solvent Extraction (ASE) with Analytical Validation by GC/MS and GC/ECD," Document 116064.A, Dionex Corporation, June 16, 1994.

Sextec is a registered trademark of Tecator (Perstorp, Inc.).
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LPN 0634-04 PDF 4/11
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