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Application Note 341

Extraction of Base/Neutrals and Acids (BNAs) from Large-Volume Samples Using Accelerated Solvent Extraction (ASE)

INTRODUCTION

Accelerated Solvent Extraction (ASE®) is an 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 bottle for cleanup or analysis. The entire extraction process is fully automated and performed in minutes for easy, fast extraction.

ASE extraction of base/neutral and acid (BNA) compounds from soils, sludges, and solid wastes is more convenient, faster, and less solvent-intensive than sonication and Soxhlet. Sonication and Soxhlet are both manual techniques that require large amounts of labor. Sonication requires 0.5–1.0 h for extraction; Soxhlet requires 2–48 h. Both sonication and Soxhlet use approximately 150–500 mL of solvent for most environmental samples.

Recoveries of BNAs by ASE are equivalent to recoveries from other more solvent-intensive methods such as Soxhlet. ASE also avoids the problems of localized heating and multiple washing procedures associated with sonication. ASE can extract a 30-g sample of a typical soil in about 17 min with a total solvent consumption of approximately 150 mL.

The procedures described in this application note meet the requirements for sample extraction as determined by U. S. EPA Method 3545(A). This method is applicable to the extraction of water-insoluble or slightly water-soluble volatile and semivolatile compounds in preparation for GC/MS measurement. This method is applicable to soils, clays, wastes, and sediments containing from 250–12500 µg/kg of BNA compounds.

EQUIPMENT

ASE 300 Accelerated Solvent Extractor* with 66- or 100 mL stainless steel extraction cells

Gas Chromatograph

Mass Spectrometer

Dionex collection bottles for extracts (P/N 056785)

Cellulose D-28 Filters (P/N 056780)

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

SOLVENTS

Dichloromethane (pesticide grade or equivalent)

Acetone (pesticide grade or equivalent)

REAGENTS

ASE Prep DE (diatomaceous earth)

Ottawa sand

EXTRACTION CONDITIONS

Oven Temperature:	100 °C
Pressure:	1500 psi*
Oven Heatup Time:	5 min
Static Time:	5 min
Static Cycles:	1
Flush Volume:	60%
Purge:	90–180 s
Solvent:	Dichloromethane/acetone (1:1 v/v)

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

SAMPLE PREPARATION

A method detection limit study was performed on Ottawa sand at a spike concentration ranging from 250 to 12500 µg/kg for BNA compounds. To mimic real-world samples, Certified Reference Material (CRM 105-100) was mixed with water (50% v/v). Wet samples were mixed with ASE Prep DE. After mixing, a weighed sample was transferred to either a 66- or 100-mL cell.

PROCEDURE

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

1. Weigh 30 g of soil, sediment, or solid waste into a beaker.
2. Mix sample with 5–50 g ASE Prep DE.
3. Place mixture into a 66- or 100-mL extraction cell containing a cellulose filter.
4. Add surrogates and/or spikes to sample and cap the extraction cell.
5. Place extraction cells onto upper carousel.
6. Place precleaned collection bottles into bottom tray.
7. Load appropriate method.
8. Start run.

Collected extracts will be approximately 100 mL for the 66-mL extraction cells and 150 mL for the 100-mL extraction cells. The extract is now ready for cleanup or analysis depending on the extent of interfering coextractables.

RESULTS AND DISCUSSION

This application note discusses the effectiveness of ASE in the extraction of BNAs from large-volume samples. Table 1 and Figure 1 show the average recovery of individual BNA compounds from CRM 105-100. Table 2 shows the recoveries of individual BNA compounds from seven replicates of spiked Ottawa sand.

Table 1. Average ASE Recovery of BNAs from CRM 105-100

Target Compound	Certified Value	Average Value	Average Recovery	RSD
Acenaphthene	387088	345833	89.4	2.20
Acenaphthylene	10093	14683	145.5	3.54
Anthracene	250928	286500	114.3	38.3
Benz[a]anthracene	151693	154833	102.1	4.96
Benzo[a]pyrene	50766	72833	143.5	3.60
Benzo[ghi]perylene	13658	14333	105.0	13.3
Carbazole	23812	25783	108.3	2.70
Chrysene	191278	188333	98.4	3.93
Dibenz[a,h]anthracene	6587	11517	174.9	3.90
Dibenzofuran	154956	206167	133.1	3.56
Fluoranthene	852017	677667	79.6	21.3
Fluorene	222583	247667	111.3	2.25
Indeno[1,2,3-cd]pyrene	12994	15550	119.8	9.26
2-Methylnaphthalene	16318	21750	133.3	2.50
Naphthalene	9488	13717	144.6	4.55
Pentachlorophenol	726795	1007000	138.6	5.38
Phenanthrene	697061	543667	78.0	3.43
Pyrene	640373	446333	69.7	3.62

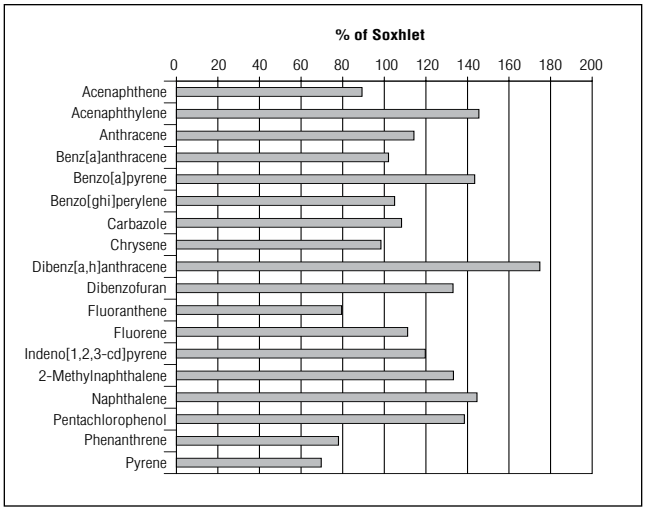

Figure 1. ASE recoveries of BNA from a certified reference material [CRM 105-100].

Table 2. BNA MDLs					
Target Compound	**Average Recovery**	**MDL (µg/kg)**	**Target Compound**	**Average Recovery**	**MDL (µg/kg)**
N-Nitrosodimethylamine	213.47	32.09	2-Fluorobiphenyl	1373.47	189.94
Pyridine	135.58	20.80	Dimethylphthalate	250.89	34.43
Phenol	250.18	40.47	2,6-Dinitrotoluene	234.08	33.87
Aniline	207.39	46.47	Diethylphthalate	264.86	36.74
Bis(2-chloroethyl)ether	227.80	27.53	Acenaphthalene	240.88	37.94
2-Chlorophenol	234.13	44.80	3-Nitroaniline	215.68	38.72
1,3-Dichlorobenzene	205.60	25.20	Acenaphthene	240.73	34.31
1,4-Dichlorobenzene	219.83	28.08	4-Nitrophenol	239.70	51.41
Benzyl alcohol	267.14	53.04	4,6-Dinitro-2-methylphenol	145.46	131.89
1,2-Dichlorobenzene	216.61	26.70	2,4-Dinitrotoluene	225.26	23.92
2-Methylphenol	246.23	27.21	Dibenzofuran	245.90	32.03
Bis(2-chloroisopropyl)ether	236.81	30.70	4-Chlorophenyl-phenylether	239.46	31.24
4-Methylphenol	244.04	58.68	Fluorene	246.26	26.96
N-Nitroso-di-*n*-propylamine	235.72	40.84	4-Nitroaniline	229.59	36.66
Hexachloroethane	200.41	32.29	N-Nitrosdiphenylamine	253.92	26.60
Nitrobenzene	240.02	45.63	4-Bromophenyl-phenylether	232.44	52.04
Isophorone	245.49	34.65	Hexachlorobenzene	237.40	47.00
2,4-Dimethylphenol	224.87	49.67	Pentachlorophenol	196.30	64.13
2-Nitrophenol	227.41	35.68	Phenanthrene	253.85	39.17
Bis(chlorethoxy)methane	238.21	31.51	Anthracene	251.91	37.14
2,4-Dichlorophenol	243.08	33.62	Carbazole	264.81	38.44
1,2,4-Trichlorobenzene	225.20	34.40	Fluoranthene	269.99	53.00
Naphthalene	242.54	35.80	Pyrene	237.01	47.73
4-Chloroaniline	210.67	54.57	3,3'-Dichlorobenzidine	206.98	50.03
Hexachlorobutadiene	218.38	37.10	Benz[a]anthracene	263.91	44.48
4-Chloro-3-methylphenol	249.94	32.44	Chrysene	257.24	48.94
2-Methylnaphthalene	267.90	25.23	Benzo[b]fluoranthene	234.56	37.82
Hexachlorocyclopentadiene	192.86	42..45	Benzo[k]fluoranthene	247.14	72.08
2,4,6-Trichlorophenol	221.32	47.19	Benzo[a]pyrene	247.50	38.33
2,4,5-Trichlorophenol	233.53	34.03	Indeno[1,2,3-cd]pyrene	234.86	37.79
2-Chloronaphthalene	234.37	40.80	Dibenz[a,h]anthracene	231.11	46.17
2-Nitroaniline	235.22	39.51	Benzo[g,h,I]perylene	241.89	37.26
Azobenzene	251.86	23.88	Di-n-butylphthalate	364.89	48.76
Benzidine	109.15	142.76	Butylbenzylphthalate	265.86	38.86
Bis(2-ethylhexyl)phthalate	263.88	29.45	Di-n-octylphthalate	227.91	26.94

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LPN 1203-04 PDF 6/11