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Accelerated Solvent Extraction (ASE) of the Organic Components of Granular and Liquid Detergents

INTRODUCTION

Organic solvent extraction is the standard technique in the detergent industry for the isolation of organic components from inorganic matrices and the separation of ionic and nonionic surfactants. For example, analysis of the alcohol-soluble portion of detergents is routinely performed to verify the integrity of the manufacturing process. Traditional extraction of this material is labor-intensive and uses large amounts of liquid solvent. With Accelerated Solvent Extraction (ASE®), manufacturers can streamline the extraction process to quickly verify product quality and provide feedback information into the production cycle.

ASE is an automated extraction technique that reduces the time and the volume of organic solvent required to perform traditional sample preparation. ASE uses a combination of elevated pressure and temperature with common liquid solvents to increase the efficiency of the extraction process. Each extraction is performed in minutes, using a fraction of the solvent volume required for conventional techniques such as Soxhlet.

During the extraction process, the extraction solvent is delivered into a cell containing the sample, which is then brought to an elevated temperature. Pressure is applied to maintain the solvent in its liquid state. Following extraction, the extract containing the target analytes is moved from the cell to a collection vial for analysis. With ASE, up to 24 samples can be processed unattended.

EQUIPMENT

Dionex ASE 200 Accelerated Solvent Extractor*
equipped with 11, 22, or 33 mL cells
Analytical balance
Dionex vials for extract collection (40 mL, P/N 49465;
60 mL, P/N 49466)
Cellulose filter disks (P/N 49458)

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

SOLVENT

Ethanol (95%, HPLC grade or better)

EXTRACTION CONDITIONS

Extraction Solvent: Ethanol
Temperature: 150 °C
Pressure: 1500 psi*
Heat-Up Time: 7 min
Static Time: 3 min
Flush Volume: 60% of cell volume
Purge Time: 60 s
Static Cycles: 1–3
Total Extraction
Time: 12–18 min per sample

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

SAMPLE PREPARATION

Weigh and directly add dry, granular detergents to the ASE extraction cells containing a cellulose filter. Prepare liquid detergent samples by mixing each with Hydromatrix in a ratio of 1:1 (v/v).

PROCEDURE

Weigh the collection vials prior to extraction. Following extraction, remove the ethanol by nitrogen evaporation with gentle heating. Cool the vials to room temperature. Reweigh the collection vials to determine the weight percent extracted.

RESULTS AND DISCUSSION

The conventional manual extraction method used to determine total alcohol extractables is a multistep procedure requiring 700 mL of solvent and 4 hours to complete. The ASE extraction method used for this analysis was complete in 12–18 minutes per sample, with a total solvent usage of 15 mL per sample (11 mL cell size). Five granular detergent samples were extracted using ASE and the conventional technique. Comparison of the results is shown in Table 1. The average recovery by ASE compared to the conventional method was 101.6%, indicating that the two techniques generated equivalent results. For the granular samples, the absolute standard deviation of the ASE extract was 0.19% (n=5) compared to 0.56% for the conventional method. Inspection of the IR spectra (Figure 1) indicates no significant variation in the composition of the extracts generated by the two techniques.

ASE was then compared to the same method for the extraction of two liquid detergent samples (Table 2).

Sample	% Extractable by Weight	
	Std. Method	ASE
1	22.16	22.40
2	34.10	33.49
3	38.50	39.22
4	21.80	22.76
5	30.10	30.69
Average	29.33	29.71
Std. Dev.	0.56%	0.19%

Sample	% Extractable by Weight	
	Std. Method	ASE
1	44.35	45.35
2 (lab 1)	53.30	55.87
2 (lab 2)	57.2	39.22

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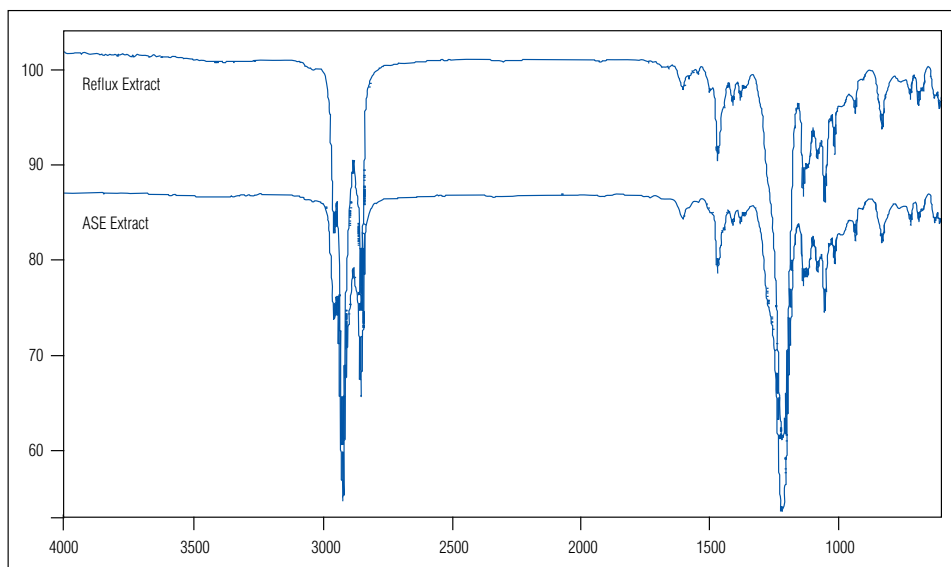


Figure 1. IR spectra comparison of extracts generated using ASE and conventional extraction.

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