Application Note 21162

# The Analysis of Banned Azo Dyes in Textiles Using a Solid Supported Liquid-Liquid Extraction

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## Key Words

HyperSep SLE, Azo dyes, solid supported liquid-liquid extraction

## Goal

Azo dyes can be extracted from textiles using solid supported liquid-liquid extraction. A method is presented for performing this extraction. This approach has several advantages over conventional liquid-liquid extraction which are described below.

## Introduction

Azo Compounds contain a functional group R-N=NR', where R,R' is an alkyl or aryl. The aryl azo compounds undergo n-delocalisation producing vivid colours such as reds, oranges and yellows. These are commonly referred to as azo dyes and are used as food colouring agents. In the food industry these are more commonly known as E numbers. Some of these dyes have been banned due to the toxicity of their degradation products which have been found to be mutagens and carcinogens. The extraction of such compounds from textiles can be achieved in different ways, one of the most common being liquid-liquid extraction.

Solid supported liquid-liquid extraction (SLE) provides time and cost benefits over conventional liquid-liquid extraction (LLE) whilst maintaining the ability to provide clean extracts free of matrix interferences. SLE lends itself to the extraction of components of moderate to low polarity (logP >2) and has several advantages over LLE, including:

1. Uses less solvent

2. Does not produce emulsification

3. Easily automated and takes less time

4. Requires small sample volumes

SLE utilizes a solid support of packed diatomaceous earth to support an aqueous sample. The aqueous sample is first loaded onto the support and allowed to adsorb. Small volumes of extraction solvent are then passed through the packing to allow the analytes to partition into this. This can be carried out several times and the individual extraction volumes combined and dried down before being reconstituted and analyzed.

## Experimental

### Chemicals and Reagents

- Fisher Scientific™ HPLC grade water (P/N W/0106/17)
- Fisher Scientific citric acid (P/N C/6200/53)
- Fisher Scientific sodium hydroxide (P/N BPE359-212)
- Fisher Scientific sodium dithionite (P/N S/3800/53)

### Sample Handling Equipment

- Thermo Scientific™ SPE Manifold (P/N 60104-233)
- NSC Mass Spec Certified 2 mL clear vial with blue bonded PTFE silicone cap (P/N MSCERT4000-34W)
- Thermo Scientific Reacti-Therm Heating Module (P/N TS-18823)
- Thermo Scientific Reacti-Vap Evaporator (P/N TS-18826)

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## Sample Preparation

### Cartridge type

HyperSep SLE 20000mg/60 mL (P/N 60109-2000-60-7)

### Pretreatment

Cut representative sample into small pieces of 5 mm × 5 mm and mix. Transfer 1.0 g (accurate to 0.01 g) of sample into a reactor and add 16 mL of citrate buffer (0.06 mol/L, pH 6.0) at 70±2 °C. Seal the reactor and shake up until all the sample is soaked into the liquid. Put the reactor in a water bath at 70±2 °C for 30 minutes to soak the textiles thoroughly. Add 3.0 mL of sodium dithionite solution (200 mg/mL - prepared fresh daily), seal and shake. After a further 30 minutes in the water bath, cool the reactor to room temperature in 2 minutes.

### Application

Press the sample in the reactor with glass rod, and transfer the liquid onto the HyperSep SLE cartridge.

### Adsorption

Draw the liquid onto the cartridge bed and leave to adsorb (15 minutes).

### Elution

Elute the cartridge with diethyl ether four times (20 mL × 4). For each time, wash the reactor with 20 mL of ether and load onto cartridge. Control the flow rate. Collect the eluate in a round flask.

### Post-treatment

Evaporate the eluate to 1 mL by rotary evaporator at 35 °C and dry under a slow stream of nitrogen.

## Separation Conditions

The experimental parameters stated will require optimization with your system.

| | |
|---|---|
| Column | Thermo Scientific™ TraceGOLD™ TG-5MS, 30 m × 0.25 mm × 0.25 µm (P/N 26098-1420) |
| Injection temperature | 250 °C |
| Column temperature | 50 °C (0.5 min) 20 °C/min 150 °C (8 min) 20 °C/min 230 °C (20 min) 20 °C/ min 260 °C (5 min) |
| MS interface temperature | 270 °C |
| MS scan range | 35~350 amu |
| Injection mode | splitless |
| Carrier gas | He(≥99.999%) |
| Flow rate | 1.0 mL/min |
| Injection volume | 1 µL |
| Ionization source | EI |
| Ionization voltage | 70 eV |





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