Rapid Determination of Azo Dyes in Textiles Using ASE and HPLC with MS and UV Detection

Instrument: Agilent 6130B Quadrupole LC/MS with Agilent 1200 Series HPLC System

ASE Conditions

Preheat: 20 min (purge closed)
Pressure: 1500 psi
Static: 5 min
Temp: 100 °C
Cycles: 2
Purge: 100 s
Flush: 30%
Solvent: 90% CH_3OH with 10% 1 M KOH

Extraction time per sample: 40 min
Solvent amount used per sample: 17 mL

Analytical Conditions

Column: Acclaim® 120 C18, 3 µm, 120 Å, 2.1 × 150 mm
Column Temperature: 30°C
Injection Volume: 10 µL
Gradient: See Dionex Application Note 360 (3)
UV: 240 nm, 278 nm
Ionization Mode: APCI, positive polarity
Nitrogen: 3 bar
Corona Current: 10 µA
Source Temperature: 400°C
Cone Voltage: Varies per analyte. See Table I.

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LC Conditions

Time	Flow Rate (µL/min)	30 mM Ammonium Acetate, pH 4	Acetonitrile	Methanol	Curve
0.00	250	91	5	4	
25.00	250	69	27	4	5
40.00	250	52	40	8	5
45.00	250	15	40	45	5
48.00	250	15	40	45	5
48.10	250	91	5	4	5

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Reference: http://www.dionex.com/en-us/webdocs/67364-AN360_UltiMate3000_AzoDyes_Textiles_05June08_LPN2007_01.pdf