

# Automated Solid Phase Extraction (SPE) of Steroids and Phytoestrogens in Water

## **INTRODUCTION**

Steroids and estrogens are endocrine-disrupting compounds that have the potential to interfere with reproductive systems. As such, drinking water is routinely monitored for these compounds.

## **INSTRUMENTATION USED FOR SAMPLE PREPARATION**

Dionex AutoTrace® instrument

## **SAMPLE PREPARATION**

- A 500 mL sample of water is taken for the analysis.
- The solvent MTBE (methylterbutylethylene ) is used as an eluent.
- Screen for compounds, such as chrysin, equol, glycitein, testosterone, progesterone, estrone, estradiol, and ethynylestradiol.
- The sample eluent will need to be split into 2 x 2 mL samples. The phytoestrogens can be done directly by LCMS but the steroids need to be evaporated to dryness and reconstituted in 250 µL methanol prior to LCMS analysis.

## **AUTOTRACE METHOD AND CONCLUSION**

All solvent lines are purged and primed with solvent first. A maximum of five solvents can be used to run a wide range of methods. Independent lines separate the aqueous and organic waste solvents. The instrument sample rack has six positions that can take volumes from 10 mL to 2000 mL and six sample collection positions for the eluent. This method offers an automated rugged and reproducible solution for cleaning up the samples to concentrate and remove interferences.

No.	Method: Estimated time 1h 41 min
1	Process six samples using the following method steps:
2	Condition column with 5.0 mL of MTBE into solvent waste.
3	Condition column with 5.0 mL of CH <sub>3</sub> OH into solvent waste.
4	Condition column with 5.0 mL of CH <sub>3</sub> OH/H <sub>2</sub> O 40:60 into solvent waste.
5	Load 500.0 mL of sample into column.
6	Rinse column with 5.0 mL of CH <sub>3</sub> OH/H <sub>2</sub> O 40:60 in aqueous waste.
7	Wash syringe with 5.0 mL of CH <sub>3</sub> OH.
8	Dry column with gas for 15 min.
9	Collect 5.0 mL fraction into sample tube using CH <sub>3</sub> OH.
10	Collect 5.0 mL fraction into sample tube using CH <sub>3</sub> OH/MTBE 10:90.
11	Wash syringe with 5.0 mL of CH <sub>3</sub> OH.
12	End

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## PARAMETERS:

### Flow Rates

Cond Flow:	15.0 mL/min
Load Flow:	10.0 mL/min
Rinse Flow:	20.0 mL/min
Elute Flow:	5.0 mL/min
Cond Air Push:	15.0 mL/min
Rinse Air Push:	20.0 mL/min
Elute Air Push:	5.0 mL/min

### SPE Parameters

Push Delay:	5 sec
Air Factor:	1.0
Autowash Vol.:	1.00 mL

### Instrument Parameters

Max. Elution Vol.:	12.0 mL
Exhaust Fan On:	Yes
Beeper On:	Yes

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