

# Application Note

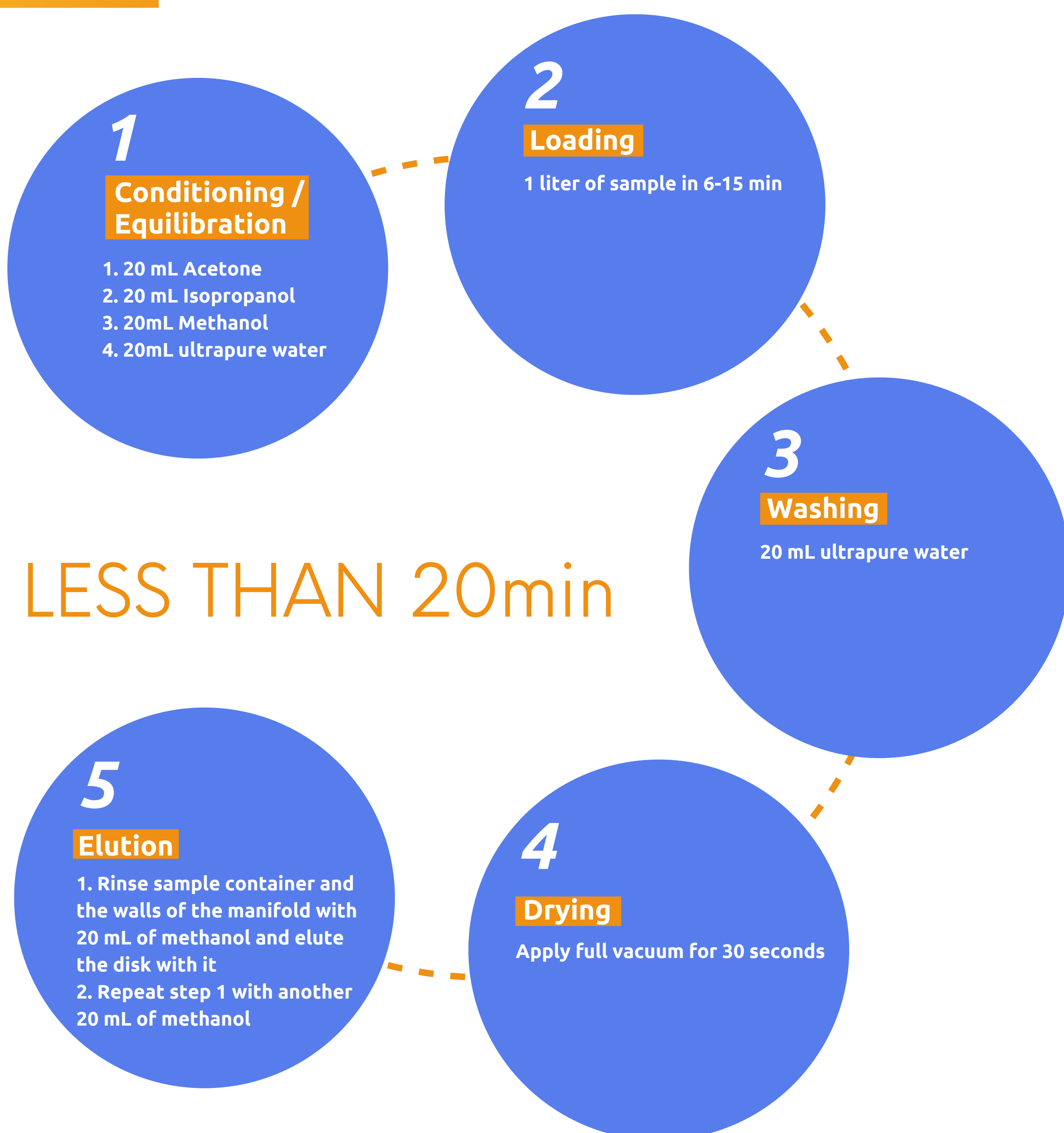


**Analysis of endocrine disruptors at trace levels in large volumes of water using AttractSPE® Disks HLB**

This application note describes the cleanup and analysis of 3 endocrine disruptors (Bisphenol A, Beta-estradiol, Ethinyl estradiol) at trace levels using AttractSPE®Disks HLB 47mm in 1 liter of reagent water and tap water. So sample was concentrated 1000-times.

1L of reagent water or tap water is spiked at 26.7 ng/L with bisphenol A, and at 267ng/L with beta-estradiol and ethinyl estradiol. For each experiment, presence of these compounds on non-spiked water were found negligible.

## STEPS



IN LESS THAN 20min



Elution solutions were concentrated by evaporation under vacuum at 45°C. After **AttractSPE®Disks HLB** procedure, samples were analyzed by LC-MS/MS. Results are presented in table 1.




 <b>MOLECULE</b>	 <b>REAGENT WATER</b>		 <b>TAP WATER</b>	
	<b>RECOVERY</b>	<b>RSD<sub>r</sub>(n = 2)</b>	<b>RECOVERY</b>	<b>RSD<sub>r</sub>(n = 2)</b>
<b>BISPHENOL A</b>	<b>98%</b>	<b>5%</b>	<b>86%</b>	<b>7%</b>
<b>BETA-ESTRADIOL</b>	<b>96%</b>	<b>1%</b>	<b>90%</b>	<b>5%</b>
<b>ETHYNIL ESTRADIOL</b>	<b>96%</b>	<b>1%</b>	<b>87%</b>	<b>7%</b>

Table 1. Recovery yields for 3 endocrine disruptors in 1L of reagent or tap water, using **AttractSPE®Disks HLB** 47mm.

- ✓ **Fast flow rate (1L in less than 15min)**
- ✓ **Excellent Recovery (> 85%)**
- ✓ **Good reproducibility (RSDR < 10%)**



## HPLC Method with LC – MS/MS

Analysis by HPLC – MS/MS (QTRAP 4000)

HPLC Column: Silact™HPLC LC - EDC 50\*2.1 mm (3µm)

Delay column Silact™HPLC - DELAY - EDC for Endocrine disruptors analysis - 50x2.1mm

Column temperature: 40°C

Flow rate: 0,3mL/min

Injection volume: 5µL



(min)



(%water)



(%Methanol)

0	80	20
1	80	20
7	5	95
11	5	95
12	80	20
18	80	20

Table 1. HPLC gradient for the analysis.

Analyte	Q1	Q3	DP (V)	EP (V)	CE (V)	CXP (V)
Bisphenol A (Q)	153	109	-45	-10	-14	-7
Bisphenol A (q)	153	81	-45	-10	-18	-1
β-estradiol (Q)	271.2	145.2	-105	-10	-58	-9
β-estradiol (q)	271.2	182.9	-105	-10	-54	-7
Ethinylestradiol (Q)	295.2	144.9	-100	-10	-56	-5
Ethinylestradiol (q)	295.2	183.1	-100	-10	-56	-13

### Mass parameters

Ion source: ESI Negative

Curtain gas: 30

Collision gas: High

IonSpray

voltage: -4500 V

Source temperature: 600°C

GS1: 30

GS2: 30

Table 2. MRM transitions for the analysis

# CONCLUSION

**AttractSPE®Disks HLB** show excellent performances with recovery yields higher than 85% for all evaluated compounds. SPE Disk format allowed a fast treatment of large sample volumes (~20 minutes). The method makes possible a 1000-times concentration of the sample.

### AttractSPE®Disks HLB

SPE-Disks-HLB-25.T1.40 for 40/pk – 25mm

SPE-Disks-HLB-47.T1.20 for 20/pk – 47mm

SPE-Disks-HLB-90.T1.10 for 10/pk – 90mm

HPLC Delay Column DELAY-EDC-50.2.1

HPLC Column LC-EDC-50.2.1

**AttractSPE®Prefilter Glassfiber** for 47mm disks (also available for other diameters)

PF-GF-50.T1.47.1 for 50/pk – 1µm

PF-GF-50.T1.47.3 for 50/pk – 3 µm

SPE disks manifold 47mm (also available for 90mm):

- 1 station ACC-DISKSPE-G47-1

- 3 stations ACC-DISKSPE-G47-3

- 6 stations ACC-DISKSPE-G47-6