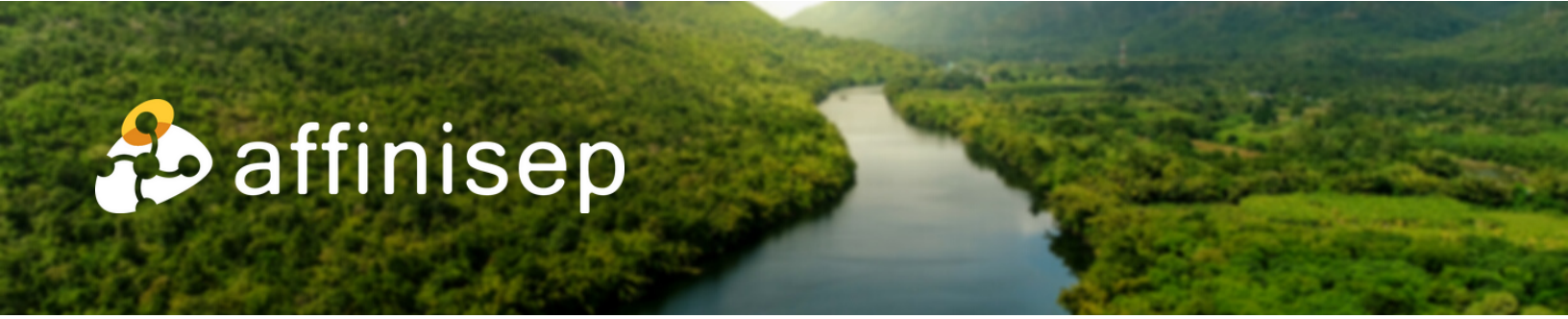


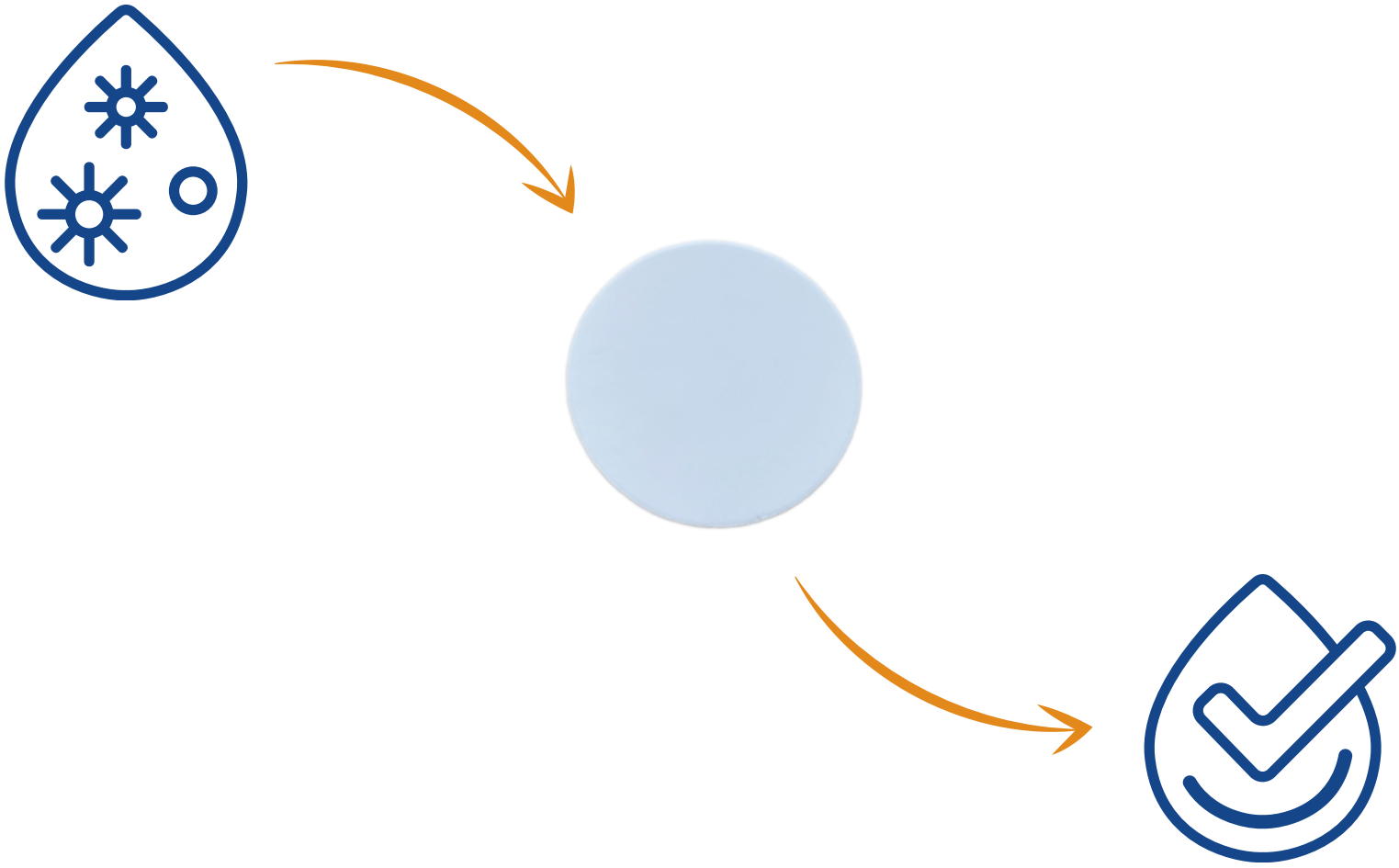
Application Note

Rapid and quantitative analysis of chlorinated acid herbicides in large water volumes using AttractSPE®Disks

AttractSPE®Disks make possible the loading of large water volume thanks to a fast flow rate and a high surface area of exchange. Our innovative SPE disks allow the best interactions with analytes and a maximal flow rates without any channeling. Attract®SPE Disks reduce extraction time while loading high volume of water. A high enrichment is obtained by our SPE disks with excellent recoveries (>90%). AttractSPE®Disks is also the perfect membrane to use for the passive sampler Chemcatcher® thanks to a very good hold and ease to use. This application note describes a rapid and quantitative analysis of chlorinated acid herbicides in large water volumes using AttractSPE®Disks.

47mm AttractSPE® Disks – Anion Exchange – SR (SAX) were used for this study

Loading solution : For both ultrapure water and tap water, one liter is spiked at 1 µg/L with aminopyralid, clopyralid, and picloram. One liter of water (not spiked) was also performed as a blank control.



PURIFICATION PROTOCOL

Place the AttractSPE® Disks – Anion Exchange – SR onto the SPE disk manifold.

Note: A glass microfiber (1 µm or 3 µm) can be added on top of the disk to prevent clogging from particulates in the water sample.

CONDITIONING/EQUILIBRATION

1. 50 mL methanol (soak disk for 1 minute)
2. 50 mL ultrapure water

LOADING

1. For both ultrapure water and tap water, one liter is spiked at 1 µg/L with several very common herbicides of Picolinic acid family such as aminopyralid, clopyralid, and picloram. One liter of water (not spiked) was also performed as a blank control.

WASHING

1. 50 mL ultrapure water
2. Dry disk 1 minute under vacuum

ELUTION

1. 50 mL 3% formic acid in methanol freshly prepared (soak disk for 1 minute)

ANALYSIS

The eluate was then agitated and diluted (1:9 ratio) with ultrapure water prior to analysis.

Note: The eluate can also be evaporated to concentrate the analytes and improve the limit of quantification.

SPE Disk manifold for AttractSPE® Disks

After the cleanup procedure, the molecules were simultaneously analyzed by LC-MS/MS. The results obtained are presented in Table 1.

	PRESENCE IN BLANK CONTROL		% RECOVERY	
	Ultrapure water	Tap water	Tap water	Ultrapure water
AMINOPYRALID	**<0.1 µg/L**	**<0.1 µg/L**	**102%**	**80%**
CLOPYRALID	**<0.1 µg/L**	**<0.1 µg/L**	**102%**	**90%**
PICLORAM	**<0.1 µg/L**	**<0.1 µg/L**	**108%**	**87%**

Table 1. Percent recovery of Aminopyralid, Clopyralid and Picloram at a concentration of 1 µg/L in ultrapure water and in tap water after the AttractSPE® Disks – Anion Exchange – SR (SAX)cleanup.

Recoveries ranging from 80% to 108% for the three molecules were observed, demonstrating the success of the purification method using AttractSPE® Disks – Anion Exchange – SR

LC CONDITIONS	MS CONDITIONS
LC Dionex U3000	Qtrap 4000 ESI+ MS/MS
Column : Hypersil Gold 150*2.1 mm (3 µm) + guard Hypersil Gold 1 cm at 30°C	Curtain gas : 20
	CAD: High
Injection volume : 20 µL	IS : 4500 V
T° sampler : 10°C	Temperature : 550°C
Flow rate : 0.3 mL/min	GS1/GS2 : 50/50

TIME (MIN)	% WATER 0.1% FORMIC ACID	% ACETONITRILE 0.1% FORMIC ACID	ANALYTE	RETENTION TIME (MIN)	Q1	Q3	CE (V)
0	97	3	Aminopyralid	4.1	207.0	161.1	33
1	97	3			207.0	134.0	45
7	42	58	Clopyralid	5.9	192.0	146.1	33
9	42	58			192.0	110.1	51
10	97	3	Picloram	6.9	241.0	213.0	29
15	97	3			241.0	195.0	23

Table 2. Conditions of analysis with LC-MS/MS.

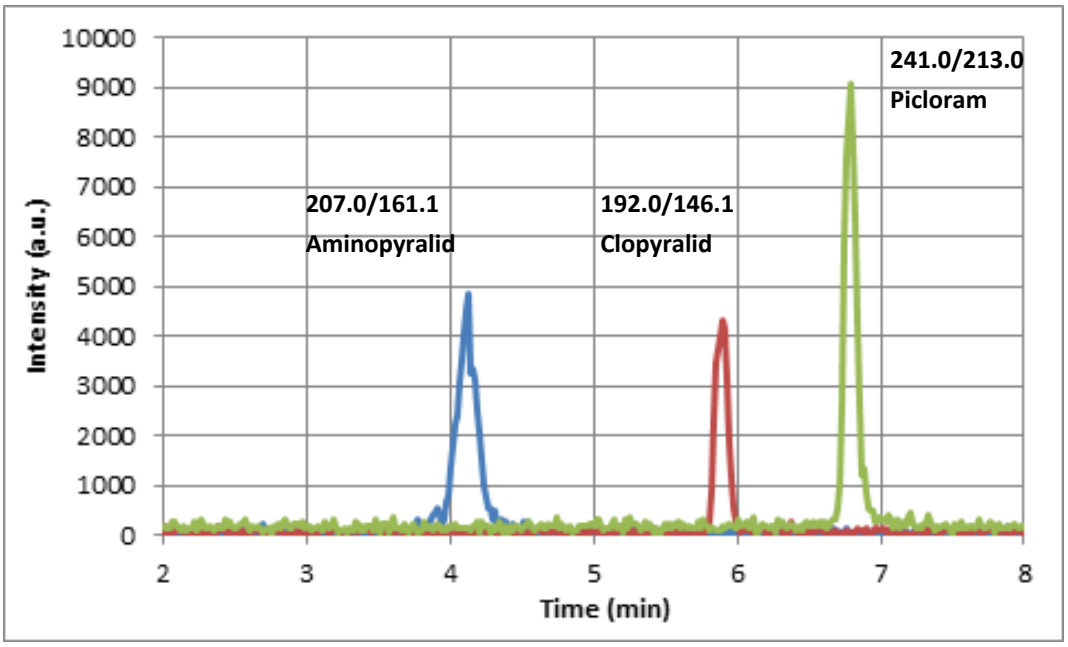


Figure 1. LC-MS/MS chromatogram of aminopyralid, clopyralid, and picloram at 5 µg/L after cleanup with AttractSPE® Disks – Anion Exchange – SR (SAX).

Conclusion

AttractSPE® Disks – Anion Exchange - SR have shown excellent performances in the detection of Aminopyralid, Clopyralid and Picloram with excellent recovery yields above 80%. SPE Disk format allowed a fast treatment of large sample volumes (~20-25 min). The method makes possible a 1000-times concentration of samples.

AttractSPE® Disks – Anion Exchange – SR

- *SPE-Disks-AN-25.T1.40 for 40/pk – 25mm*
- *SPE-Disks-AN-47.T1.20 for 20/pk – 47mm*
- *SPE-Disks-AN-90.T1.10 for 10/pk – 90mm*

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*

Related Products

AttractSPE® Disks Passive Sampler Anion exchange - SR - 10/pk

- DBPS.AN.90.40.kit.10

AFFINIMIP® SPE Picolonic herbicides
6mL – 50/pk

- FS115-03B