affinisep

Application Note

LC-MS/MS detection of glyphosate/AMPA/glufosinate in apple juice without prior derivatization after AFFINIMIP® SPE Glyphosate cleanup

A 6 mL AFFINIMIP® SPE Glyphosate cartridge was used for this study.

Loading solution: 5 mL of clear apple juice is diluted with 15 mL of ultrapure water. 200µL of formic acid is added and the solution is stirred. The pH is adjusted to 6–8 with 35% ammonia solution. The solution is then spiked with glyphosate, AMPA, and glufosinate at 16.7 µg/L each.

CONDITIONING

1. 6 mL ultrapure water

LOADING

1. 6 mL of loading solution at 1.5 mL/min

WASHING

1. 12 mL ultrapure water

ELUTION

1. 8 mL HCl 0.1M (in water)

ANALYSIS

Elutions are collected in polypropylene vials, evaporated under vacuum at 60°C for 2 hours, and dissolved in 1 mL of mobile phase containing 0.8mM of EDTA-Na2. The solution is then analyzed by LC-MS/MS (Table 3).

Note: We recommend the use of plastic labware to avoid potential adsorption of the analytes on glassware.

Results

After the AFFINIMIP® SPE Glyphosate procedure, the molecules were simultaneously analyzed by LC-MS/MS without derivatization. Apple juice without added glyphosate, AMPA, or glufosinate was also tested as a blank control. The results obtained are presented in Table 1.

ANALYTE	CONCENTRATION IN BLANK (DILUTED APPLE JUICE) (µg/L)	SPIKE LEVEL (µg/L) (DILUTED APPLE JUICE)	RECOVERY FROM SPIKED SAMPLE
GLYPHOSATE	<0.8 ug/L	16.7	96%
AMPA	ND	16.7	86%
GLUFOSINATE	ND	16.7	92%

Table 1. Recovery of glyphosate, AMPA, and glufosinate in diluted apple juice spiked at 16.7 µg/L after purification with AFFINIMIP® SPE Glyphosate. (ND = Not detected)

Recoveries ranging from 86% to 96% for the three molecules were observed, demonstrating the success of the purification method using AFFINIMIP® SPE Glyphosate

LC CONDITIONS	MS CONDITIONS
LC Dionex U3000	Sciex Qtrap 4000 ESI- MS/MS
Column: Acclaim Trinity Q1 100 mm x 3 mm ID (3 µm) + prefilter	Curtain gas: 30
	CAD: High
Injection volume: 20 µL	IS: -4500V
T° sampler: 10°C	Temperature: 700°C
Flow rate: 0.5 mL/min	GS1/GS2: 50/50

TIME (MIN)	SOLVENT A	SOLVENT B	ANALYTE	RETENTION TIME (MIN)	Q1	Q3	CE (V)
0	100%	0%	Glyphosate	1.8	168.0	62.9	-32
3	100%	0%			168.0	78.9	-50
3.2	0%	100%	AMPA	1.6	110.1	62.8	-24
6	0%	100%			110.1	78.8	-34
6.2	100%	0%	Glufosinate	1.2	179.9	63.1	-58
10.2	100%	0%			179.9	95.0	-24
Solvent A : 50mM Ammonium formate, pH 2.9 (adjusted with formic acid) Solvent B : Acetonitrile							

Table 2. LC-MS/MS conditions for tested analytes.

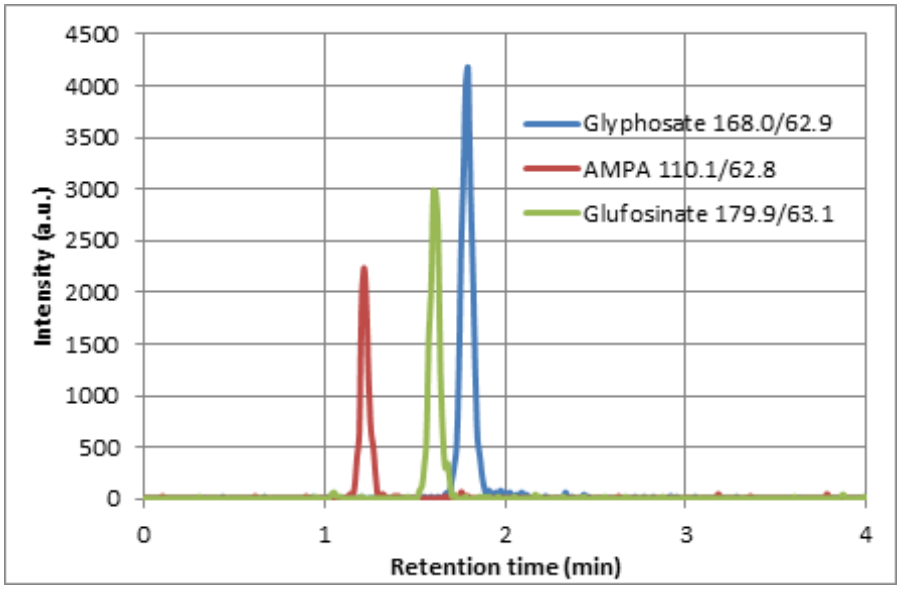

Figure 1. Typical LC-MS/MS chromatogram obtained for the three main ion transitions for glyphosate, AMPA, and glufosinate using AFFINIMIP® SPE Glyphosate.

Conclusion

AFFINIMIP® SPE Glyphosate has shown excellent performances for the enrichment and cleanup of glyphosate, AMPA, and glufosinate from apple juice. AFFINIMIP® SPE Glyphosate demonstrated a high selectivity for the three molecules, producing high recoveries of the three compounds (above <80%). In addition, the protocol is very simple, fast, and easily automated.

References:

[1]Application notebook for glyphosate including tests in various matrices available at:

https://www.affinisep.com/spe-kits-applications/spe-kit-for-sample-preparation/affinimip-spe-selectives-mip-spe-cartridges/affinimip-spe-glyphosate-ampa/

PART NUMBER OF PRODUCTS USED IN THIS APPLICATION NOTE:		
PRODUCT:	**QUANTITY:**	**PART NUMBER:**
AFFINIMIP® SPE GLYPHOSATE - 6ML CARTRIDGES	50/PK	FS113-03B
AFFINIMIP® SPE PATULIE - 6ML CARTRIDGES	50/PK	FS102-03B-200MG