



AFFINISEP

Application note.



Application Note AFFINIMIP® SPE
Patulin Apple Juice LC – MS/MS

Patulin analysis in Apple juice using AFFINIMIP® SPE Patulin

PROTOCOLE OF PURIFICATION

Sample preparation

Clear apple juice was diluted by two with ultrapure water – 2% Acetic acid to form the loading solution.

Purification with a 3mL-100mg AFFINIMIP® SPE Patulin cartridge

Equilibration

- 2 mL Acetonitrile
- 1 mL Ultrapure water

Loading

- 4 mL of loading solution (0,5mL/min)

Washing of interferences

- 1 mL NaHCO₃ - 1% solution
- 2 mL ultrapure water (immediately)

Drying 30 sec under full vacuum (if evaporation of elution solution)

Elution

- 2 mL Acetonitrile

Elutions are then homogenized, diluted by 5 with water 0.1% Acetic acid, and analyzed.

Note: An evaporation can also be processed in order to reach concentrations lower than 1 µg/Kg. See application note of Patulin in apple juice (LC-UV) for optimized protocol.



ANALYSIS

HPLC Method with LC – MS/MS

Analysis by HPLC – MS/MS (QTRAP 4000)




Column: Patulin HPLC column - 150x 2.1mm (3µm)

Column temperature: 30°C

Flow rate: 0,2mL/min

Injection volume: 20µL

Table 1. HPLC gradient for the analysis.

 (min)	 (water)	 (Acetonitrile)
0	100	0
2	100	0
10	80	20
12	80	20
13	20	80
16	20	80
17	100	0
25	100	0

RESULTS

Table 3. Recovery at a concentration of 10µg/Kg

Analyte	Recovery (%)	RSDr (%) (n=4)
Patulin	81	6

-  Shorter cleanup – No evaporation required
-  Excellent Recovery (> 80%)
-  Good repeatability (RSDr < 10%)

Table 2. MRM transitions for the analysis

Analyte	Q1	Q3	DP (V)	EP (V)	CE (V)	CXP (V)
Patulin Q	153	109	-45	-10	-14	-7
Patulin q	153	81	-45	-10	-18	-1

Mass parameters

Ion source: ESI Negative

Curtain gas: 40

Collision gas: Medium

IonSpray voltage: -3000 V

Source temperature: 550°C

GS1: 50

GS2: 50

Catalog number

HPLC Column

LC-Pat-150.2.1

3mL format

FS102-02 for 25 cartridges

FS102-03 for 50 cartridges

6mL format

FS102-02B-200mg for 25 cartridges

FS102-03B-200mg for 50 cartridge