APPLICATION BRIEF

Food/Nutraceutical

Detection of Acrylamide in Potato Chips Using UHPLC/SQ MS

Acrylamide is a known carcinogen found in heated and fried foods. The most common methods for acrylamide analysis are GC/MS, GC/FID, GC with a nitrogen specific detector or LC/MS/MS.

For GC analysis, acrylamide is often brominated to facilitate extraction into an organic solvent. We describe a UHPLC/SQ MS method that does not require a tedious derivatization and is a less expensive alternative to the LC/MS/MS assay.

Experimental Conditions

Target Analyte: Acrylamide

Sample Preparation Conditions

Potato chips obtained from a grocery store were ground to a fine powder in a food processor. Water (10 mL) was added to the chips (1 g), spiked with 1,2,3,C13-acrylamide as the internal standard (50 ng/mL) and was gently shaken for ~20 min to extract acrylamide from the chips. The resulting solution was centrifuged at ~8000 RPM for 30 min. Three layers were observed from centrifugation. The middle aqueous layer (1 mL) was gently removed to avoid sampling any of the upper oily layer. The extract was loaded on a mixed mode MCAX SPE cartridge (Supelco, DSC-MCAX, 300 mg/3 mL) stacked in a series with the C18 cartridge (Supelco, DSC-18, 1g/6 mL) using SPE tube adaptors. Prior to sample loading, the cartridges were conditioned with methanol (2 mL) followed by DI water (2 mL). The sample loaded on the preconditioned cartridges was pulled through with vacuum followed by a water wash (1 mL). The MCAX cartridge was disposed of along with the eluate. The acrylamide on the C18 cartridge was eluted with 2 mL of methanol. The methanol eluate was then concentrated under a stream of nitrogen at 30 °C to ~500 µL and reconstituted to 1 mL with methanol. The samples were filtered through 0.45 µm filters prior to UHPLC/SQ MS analysis. Three separate extractions of potato chips were analyzed to obtain an average concentration of acrylamide in the chips.

Liquid Chromatography Conditions

Pump Type:	PerkinElmer® Flexar™ FX-15
Column:	Thermo Hypercarb® (2.1 mm x 100 mm, 3 µm)
Mobile Phase:	A: water containing 0.1% formic acid B: acetonitrile containing 0.1% formic acid
Flow Rate:	0.25 mL/min
Injection Volume:	3 µL/min in partial fill mode

Isocratic Conditions:	Time (min)	%A	%B
	0-5	98	2

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Mass Spectrometer Conditions	
Ionization:	Ultraspray™ ESI – Positive mode
Selected Ion Monitoring (SIM) Mode:	$[M+H]^+$ of acrylamide and 1,2,3,C13-acrylamide monitored at 72.1 m/z and 75.1 m/z respectively; dwell time of 250 ms each
Capillary Exit Voltage:	100 V

Results

A calibration curve for acrylamide was run over the range of 3.9-250 ng/mL with 1,2,3,C13-acrylamide as internal standard (50 ng/mL). Figure 1 shows the SIM ion trace of the lowest standard of acrylamide (S/N > 3 RMS) and the spiked internal standard of 1,2,3,C13-acrylamide. The instrument detection limit for acrylamide on the Flexar SQ 300 MS was determined to be 3.9 ng/mL.

The recovery of the internal standard 1,2,3,C13-acrylamide taken through the solid phase extraction procedure was estimated at 75.4% (RSD of 9.5%, n=3).

Figure 2 shows SIM ion traces of acrylamide and its internal standard in a potato chip extract. The summary for three separate analyses of potato chip extracts is shown in Figure 3.

Conclusions

A method has been developed for the measurement of acrylamide using off-line SPE extraction followed by UHPLC/SQ MS analysis with the Flexar SQ 300 MS. The ESI source is able to handle ~100% water and the instrument detection limits of 3.9 ng/mL were achieved for acrylamide analysis.

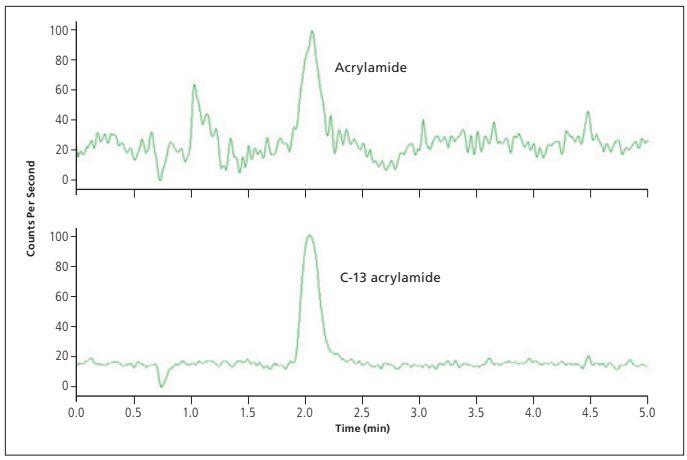

Figure 1. SIM ion trace of the lowest standard of acrylamide (3.9 ng/mL, S/N = 10) and SIM ion trace of the internal standard 1,2,3,C13-acrylamide (50 ng/mL) were monitored simultaneously.

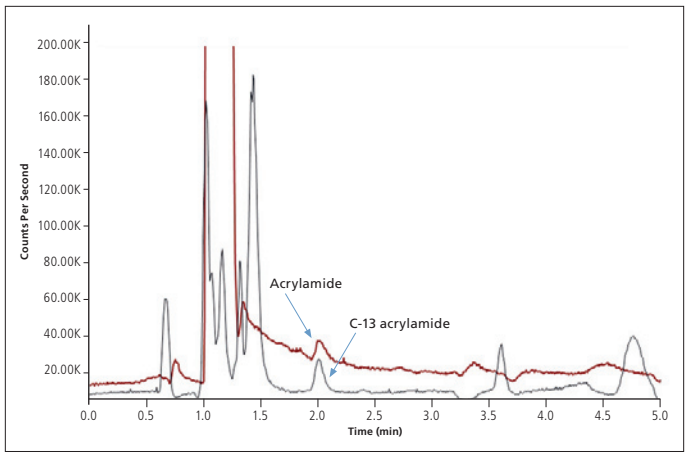

Figure 2. SIM chromatograms of a potato chip sample.

Sample	µg/Kg
Extract 1	63.2
Extract 2	71.3
Extract 3	63.9
Average concentration of acrylamide	66.1
% RSD	6.8

Figure 3. Results of quantitative analysis on potato chip extracts.

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