Analysis of Acrylamide in Potato Chips by SPE and GC-MS

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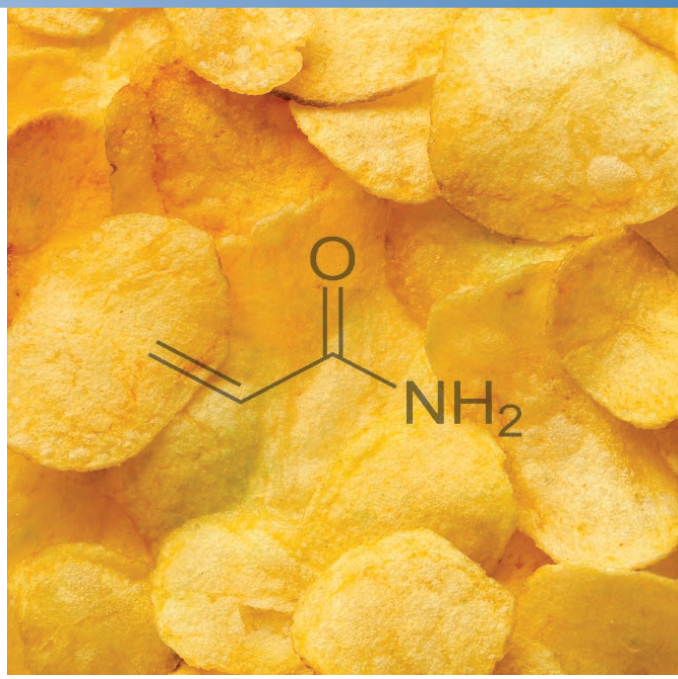
Key Words
Hypercarb SPE, food, acrylamide, 2-propenamide, capillary GC, porous graphic carbon (PGC), polyethylene glycol (PEG) GC column

Abstract
Acrylamide is an endogenous compound, formed when heating starchy or sugary foods. The production of potato chips can result in its formation. The method reported here detects acrylamide at the low ng/g levels at which it is produced. Potato chips were extracted using porous graphitic carbon for solid phase extraction (SPE). Analysis of acrylamide was performed using GC-MS on a polyethylene glycol phase GC column. A standard addition calibration curve was used to estimate the level of acrylamide in potato chips at 450 ng/g.



Introduction

Acrylamide (2-propenamide) is a potential human carcinogen. This toxic compound is usually formed as a by-product of Maillard reactions during the heating of carbohydrate-rich food. The World Health Organization (WHO) has set a safe limit of 500 ng/mL acrylamide in drinking water. Higher levels of 100–1000 ng/g are determined in some foods such as potato chips or french fries.

The extraction of acrylamide from potato chips is carried out using a Thermo Scientific™ HyperSep™ Hypercarb™ SPE cartridge. Hypercarb SPE material is 100% porous graphic carbon (PGC) and offers retention of highly polar compounds that are not usually retained by traditional reversed phase C18 columns. HyperSep Hypercarb SPE can produce clean samples by removing potential matrix interferences.

The analysis of acrylamide was carried out using a GC-MS in electron ionization (EI) mode. Quantitative measurement in food can be difficult as matrix-derived ions can interfere with acrylamide fragment ions of *m/z* 71, 55, and 41 when using this mode. Acrylamide often requires derivatization to improve sensitivity on a mass spectrometer. In this case, acrylamide is injected without derivatization onto a Thermo Scientific™ DSQ™ II mass spectrometer and an ultra low bleed Thermo Scientific™ TraceGOLD™ TG-WaxMS™ 30 m × 0.25 mm × 0.25 µm GC column.

Acrylamide is a highly polar water soluble compound having a logP value of -0.65 [1]. Such highly polar compounds are not readily amenable to GC, therefore a polar GC column is required. The TraceGOLD TG-WaxMS column is a polyethylene glycol-phase GC column that allows the analysis of polar compounds.

Experimental Details

Consumables		Cat. No.
Cartridge type:	HyperSep Hypercarb SPE cartridge, 500 mg/6 mL	**10047-152**
Column:	TraceGOLD TG-WaxMS, 30 m × 0.25 mm × 0.25 µm	**10055-728**
Septum:	Thermo Scientific BTO, 17 mm	**10056-066**
Liner:	Thermo Scientific™ Splitless FocusLiner™, 3 × 8 × 105 mm	**101710-690**
Column ferrules:	100% graphite ferrules for Thermo Scientific™ TRACE™ injector, 0.1–0.25 mm i.d.	**10055-758**
Column ferrules:	Graphite/Vespel® for transfer line 0.1–0.25 mm i.d.	**10055-746**
Vials and closures:	Assembled Advanced Vial Closure System vial and cap With ID Patch. Red PTFE/White Silicone	**89239-442**
Syringe filter:	Thermo Scientific™ Target2™ 30 mm GMF syringe filter membrane, 3.1 µm pore size	**66064-840**
Plastic syringe:	Thermo Scientific 3 mL plastic disposable syringes	**66064-758**

Sample Handling Equipment
HyperSep glass block manifold

Instrumentation
Thermo Scientific™ TRACE GC Ultra™ gas chromatograph
Thermo Scientific™ DSQ™ II single quadrupole mass spectrometer
Thermo Scientific™ TriPlus™ Autosampler

Chemicals and Reagents
HPLC grade water
HPLC grade methanol
Analytical grade formic acid

Sample Pretreatment

The potato chips were finely crushed with mortar and pestle and 1 g was weighed into a vial. A 1 g portion of the sample was spiked with 25, 50, 100, 250, 500, and 1000 ng/g of acrylamide standard in 2% formic acid / water. The sample was then filtered through a filter membrane.

Sample Preparation	
Compounds:	Acrylamide and acrylamide-d_3 (internal standard)
Matrix:	Potato chips
Conditioning stage:	Add 4 mL methanol, 4 mL water, and 4 mL 2% formic acid / water to the SPE cartridge.
Application stage:	Apply 1 mL of extract in 2% formic acid / water under vacuum at 1 mL/min to the SPE cartridge.
Washing stage:	Add 1 mL water to the SPE cartridge and dry for 20 min under vacuum.
Elution stage:	Apply 4 mL methanol to the SPE cartridge.
Additional stage:	Evaporate methanolic extract and reconstitute with 1 mL of 1 µg/mL of internal standard in methanol to the SPE cartridge.

Separation Conditions	
Carrier gas:	Helium
Split flow:	50 mL/min
Column flow:	1.2 mL/min, constant flow
Oven temperature:	80 °C, 10 °C/min, 250 °C
Injector type:	Split/Splitless
Injector mode:	Splitless (1 min), constant septum purge
Injector temperature:	230 °C

Instrumentation	
Transfer line temperature:	150 °C
Source temperature:	200 °C
Ionization conditions:	EI
Electron energy:	70 eV
SIM scan parameters:	*m/z* 71 for acrylamide and *m/z* 74 for acrylamide-d_3
Start time:	4.0 min
Dwell time:	0.1 s

Injection Conditions	
Injection volume:	2 µL
Pre- and post-needle injection dwell time:	0.5 s

Data Processing	
Software:	Thermo Scientific™ Xcalibur™ software

Results

A standard addition calibration curve was constructed for acrylamide in matrix over the range 25–1000 ng/g. Standard addition calibration was chosen because acrylamide is endogenous in cooked foods and a suitable blank matrix was unavailable.

The amount of acrylamide present in the potato chips was calculated to be 450 ng/g. The chromatogram in Figure 1 shows the acrylamide peak in potato chips and acrylamide-d_3 internal standard spiked in potato chips.

The acrylamide concentration was calculated using the integrated response ratio of acrylamide/acrylamide-d_3 (*m/z* 71/74). The acrylamide in the potato chips was calculated from the intercept of the x axis. An excellent linearity was demonstrated for this method with a coefficient of determination (R^2) of 0.999.

The accuracy of the back calculated concentrations for the amount of acrylamide spiked in potato chips was less than 10% (see Table 1).

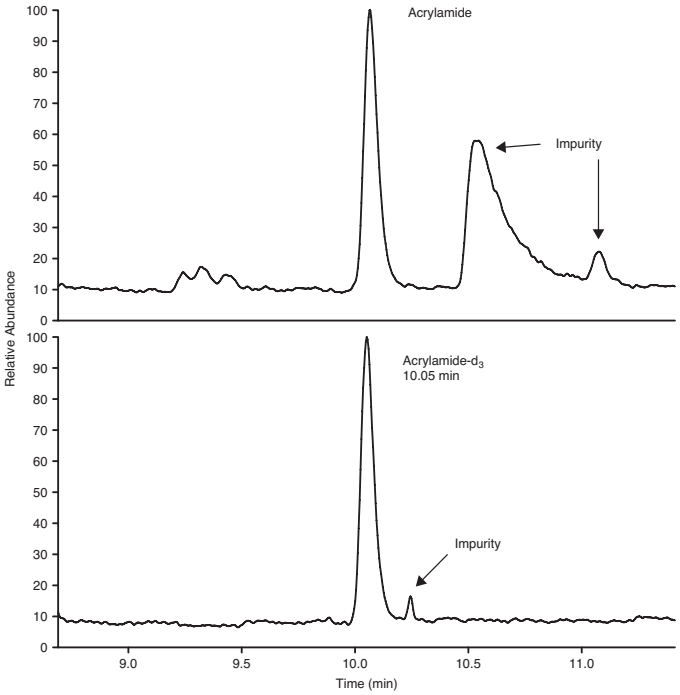


Figure 1: TIC of chromatogram of 1 µg/mL spiked acrylamide (*m/z* 71) and acrylamide-d_3 (*m/z* 74) extracted from potato chips

Specified Concentration (µg/mL)	Calculated Concentration	% Difference
0.25	0.225	-9.83
0.50	0.469	-6.22
1.00	0.983	-1.70
2.50	2.520	0.79
5.00	4.979	-0.41
10.0	10.037	0.37

Table 1: Accuracy data for the standard addition calibration curve for spiked acrylamide in potato chips

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