

# Determination of Acrylamide in Fried Potato Chips by Discovery DSC-MCAX (Mixed Cation) and DSC-18 (C18) SPE, and Discovery HS F5 (Pentafluoropropylphenyl) LC-MS

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## Introduction

In April 2002, researchers in Sweden announced that acrylamide is formed in starchy foods (e.g. potato chips, bread, cereals, etc.) when cooked at high temperatures through traditional methods (e.g. frying, baking, etc.). Because acrylamide is a suspect cancer-causing agent, many agencies worldwide have since confirmed Sweden's findings. Current methodologies for isolating and quantifying acrylamide in fried foods require extensive sample preparation, and derivatives must be formed prior to retention and analysis. In this study, a mixed-mode cation-exchange (Discovery DSC-MCAX) in series with a C18 SPE phase (Discovery DSC-18) is used to extract acrylamide from fried potato chips prior to LC-MS analysis.

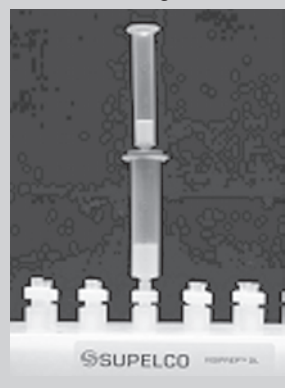
## Sample Prep and LC-MS Methodology

Extraction of acrylamide in fried potato chips is difficult due to the high level of endogenous matrix interferences inherent with fried starchy food products. Although acrylamide is readily soluble in water, starches and oils are also released during initial aqueous extraction. As a result, sample clean up is necessary to prevent starches and oils from interfering with HPLC and/or LC-MS analysis.

In this study, two SPE tubes connected in series via an SPE tube adapter were necessary to remove potato chip matrix

interferences prior to LC-MS analysis (Figure A). As the sample is loaded through the first MCAX cartridge, the combination of hydrophobic and ionic interaction derived from the mixed-mode phase retains starch and oil interferences. As the sample exits the first cartridge and is delivered into the second C18 SPE cartridge, acrylamide is weakly retained on the C18 packing.

Figure A. Discovery DSC-MCAX/DSC-18 SPE Stacked Configuration



The larger C18 SPE bed weight retards acrylamide prior to methanol elution. The eluate is then evaporated and reconstituted in water prior to LC-MS analysis. For a detailed method, please refer to Tables 1 and 2.

## Results/Discussion

Because Discovery DSC-MCAX SPE utilizes two retention mechanisms (reversed-phase and cation-exchange), the majority of the matrix interferences were adsorbed on the first cartridge. Acrylamide was eluted onto the subsequent C18

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Table 1. SPE Conditions

<b>SPE:</b> Discovery DSC-MCAX, 300mg/3mL (Cat. No. 52784-U) Discovery DSC-18, 1g/6mL (Cat. No. 52606-U) SPE Tube Adapter for 1, 3, & 6mL tubes (Cat. No. 57020-U)
<b>Conditioning:</b> Stack Discovery DSC-MCAX SPE on top of Discovery C18 SPE using tube adapter (Figure A). Condition stacked SPE with 1.0mL of methanol followed by 1.0mL DI H <sub>2</sub> O. Pull dry with vacuum
<b>Sample Prep:</b> Finely grind 2.0g of potato chips, place in 20mL vial and add 10mL water. Mix using vortex until mixture forms a thick paste. Place sample in centrifuge vials and spin at 16.1 rcf for 5 min. Extract aqueous portion of sample leaving oil layer and solids.
<b>Sample Load:</b> Load 1.0mL of aqueous extract onto conditioned SPE, pull through with vacuum.
<b>Sample Wash:</b> Wash stacked SPE with 1.0mL of water
<b>Sample Elute:</b> Remove MCAX SPE and dispose of eluate. Elute acrylamide from C18 SPE with 2.0mL methanol. Concentrate samples using at 30°C using N <sub>2</sub> stream. Reconstitute to 0.50mL with DI H <sub>2</sub> O.

Table 2. Chromatographic and Mass Spec Conditions

### LC Conditions:

**Column:** Discovery HS F5, 15cm x 4.6mm ID, 3µm particles  
**Cat. No.:** 567507-U  
**Mobile Phase:** 100% Ultra Pure Water  
**Temperature:** 35°C  
**Flow Rate:** 0.3mL/min  
**Detection:** ESI+  
**Injection Volume:** 5µL  
**Standard:** 0.048 µg/mL acrylamide in water

### MS Conditions:

**Source:** ESI+  
**Selective Ion Mode:** 72m/z  
**Capillary:** 3.20kV  
**Cone:** 30.00V  
**Extractor:** 3.00V  
**RF Lens:** 0.4V  
**Source Temp.:** 150°C  
**Cone Temp.:** 20°C  
**Desolvation Temp.:** 350°C  
**Cone Gas Flow:** 103.0L/hr  
**Desolvation Gas Flow:** 213.0L/hr

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SPE cartridge using water. The cartridges were then separated, and acrylamide was eluted off the larger C18 bed with methanol. Good recovery and RSDs were observed (Table 3) when measured against external acrylamide calibration standards in water. The detection limit of this assay is 15 µg/kg (15ppb).

In general, small basic polar compounds are poorly retained on traditional reversed-phase HPLC columns. In this assay, a pentafluorophenyl HPLC column (Discovery HS F5, 3 µm) offered good retention and selectivity for acrylamide. Unlike standard C18 HPLC phases, the F5 column offered excellent retention and peak shape using a completely aqueous mobile phase (Figure B).

In conclusion, a relatively simple sample prep method was developed using a mixed-cation SPE phase connected in tandem with a C18 SPE phase. This method eliminates the need for extensive derivatization to produce sufficient retention and detection. The unique selectivity of Discovery HS F5 HPLC offered good retention of acrylamide while maintaining good peak shape with a totally aqueous system.



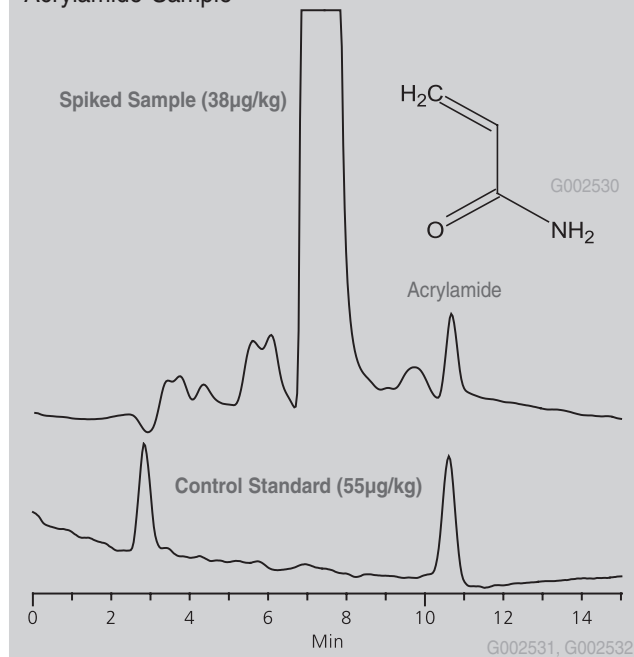
## Related Information

For more information request *Determination of Acrylamide in Fried Potato Chips by SPE and LC-MS*, T404108 (HKA).

Table 3. Recoveries and RSD of Acrylamide Standards

Concentration (µg/kg)	Recovery ± RSD (n = 3)
10	78 ± 7%
55	93 ± 5%

Figure B. Chromatogram of Control and Spiked Acrylamide Sample


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