Acrylamide Analysis by Gas Chromatography

Introduction

How much acrylamide is in that French fry? Is this potato chip safe to eat? Since the release of a report by Sweden's National Food Administration in April 2002, consumers have had something else to think about when choosing what to eat. Acrylamide is a toxic and potentially cancer-causing chemical, although the toxicological effects on humans are still under investigation. The topic of acrylamide in foods, especially in fried and baked goods, has generated a significant amount of interest in 2002. The United Kingdom's Food Standards Agency, the Center for Science in the Public Interest (CSPI), and the U.S. Food & Drug Administration (FDA) are among the groups that have begun testing for acrylamide in food products.

Researchers are postulating that acrylamide is formed in relatively high concentrations when carbohydrate-rich foods such as potatoes, rice, and cereals are cooked at high heat.[1,2] This seems to be particularly true when the products are fried. Raw or boiled starchy foods do not seem to form detectable amounts of acrylamide. Of the products tested, the highest levels of acrylamide were found in potato chips and French fries, on the order of 400-1200 ppb. By comparison, the World Health Organization (WHO) has specified a maximum concentration of 0.5 μg/L (0.5 ppb) acrylamide in drinking water.[1]

The FDA has published a draft method for the analysis of acrylamide in foods by LC/MS/MS.[3] The procedure calls for a reversed phase C18 column and a highly aqueous mobile phase (0.1% acetic acid, 0.5% methanol). Because many of the sample matrices can be quite complex, solid phase extraction is used to remove interferences prior to the chromatographic analysis. Positive ion electrospray is used for the mass spectral interface, with quantification based on comparison to a ^{13}C isotopically labeled internal standard. The method has been validated for a limited number of matrices, such as potato chips and French fries, and public and private researchers are in the process of validating the LC/MS/MS approach for other food products.

Gas chromatography (GC) has been used to quantify acrylamide in a variety of industrial and environmental applications. With increasing interest in acrylamide analysis, we investigated the feasibility of using GC to screen for this compound in food samples. GC is a low-cost, efficient way to detect semivolatile compounds, and as an analytical tool is available in many food laboratories. In this note, we describe a GC approach to analyzing acrylamide, and discuss sample pretreatment using solid phase extraction.

Experimental

Virtually any PerkinElmer capillary column gas chromatograph can be used for this application (Clarus 500, AutoSystem XL, AutoSystem, etc.)

We used the following GC conditions in analyzing both acrylamide standards and food samples:

Column:	Elite-Wax ETR - 15 m, 0.53 ID, 0.50 μm film
Inj.:	1.0 μL, 0.5 min. hold
Liner:	2 mm splitless with wool
Injector temp.:	260 °C
Carrier gas:	helium, constant pressure
Linear velocity:	62 cm/sec. @ 100 °C
Oven temp.:	100 °C (hold 0.5 min.) to 200 °C @ 15 °C/min.
Detector:	FID @ 260 °C

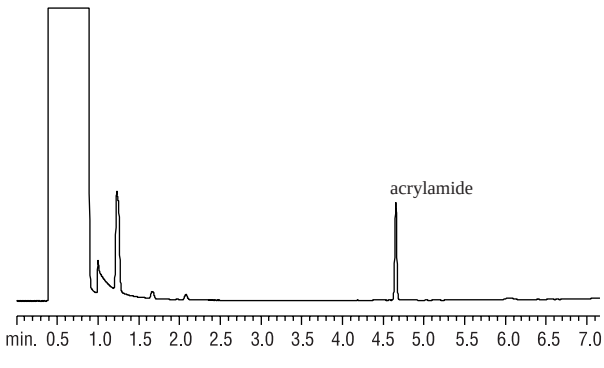
Sample preparation procedure

The procedure we used in our analysis of potato chips was as follows:

1. Analytically weigh 1 g crushed potato chips.
2. Combine chip sample with 10 mL 0.1% formic acid solution and mix on a wrist action shaker for 20 minutes.
3. Refrigerate extract for easier removal of oily top layer.
4. Filter supernatant through a 0.45 µm nylon syringe filter; remove and store for cleanup and analysis.
5. Condition CarboPrep™ 200 SPE tube, 6 mL, 500 mg:
   a. 2 mL acetone
   b. 2 mL 0.1% formic acid
6. Apply 2 mL of filtered, extracted chip solution to SPE tube. Allow sample solution to pass through tube with only gravity flow.
7. Wash SPE tube:
   a. 0.5-1.0 mL water; pass through tube quickly.
   b. Use vacuum for up to 1 minute to dry excess water from tube.
8. Elute with 2 mL of acetone, using gravity only. Eluate is ready for GC-FID analysis.

Figure 1

An Elite-Wax ETR column is an excellent choice for acrylamide analysis by capillary GC.

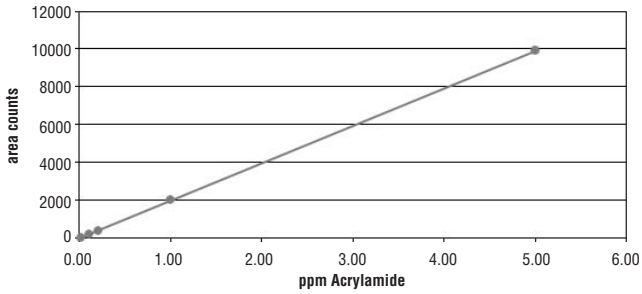


GC_FF00642

Elite-Wax ETR:	15 m, 0.53 ID, 0.50 µm
Sample:	25 µg/mL acrylamide standard in water
Inj.:	1.0 µL, 0.5 min hold
Liner:	2 mm splitless with wool
Inj. temp.:	260 °C
Carrier gas:	helium, constant pressure
Linear velocity:	62 cm/sec. @ 100 °C
Oven temp.:	100 °C (hold 0.5 min.) to 200 °C @ 15 °C/min.
Det.:	FID @ 260 °C

Figure 2

Acrylamide standard solutions were tested over a concentration range of 0.02 - 5 ppm (20 - 5000 µg/L) in water. A plot of peak counts vs. concentration shows a wide linear range for the GC assay, with $R^2 = 0.99996$.



Chromatographic conditions listed in text.

Results

The chromatogram produced by injecting 1 µL of a 25 µg/mL (25 ppm) acrylamide standard is shown in Figure 1. Figure 2 is the linearity plot for standard solutions over a range of 20-5000 ppb. The sample preparation method we followed was based on the draft U.S. Food & Drug Administration method *Detection and Quantitation of Acrylamide in Foods* dated June 20, 2002.[3]

The chromatogram we obtained from the analysis of a reduced fat potato chip extract is shown in Figure 3. The chip sample was spiked with 100 µg (50 µg/mL) of acrylamide for this analysis.

Conclusion

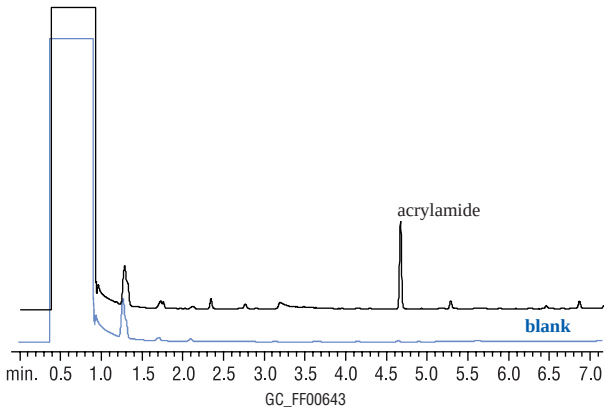
Gas chromatography offers a rapid, cost-effective approach to screening for acrylamide in food samples such as potato chips. The Elite-Wax ETR capillary column exhibits excellent selectivity for acrylamide, even when analyzing complex matrices, such as food samples. Detection limits on the order of 0.01 µg/mL (10 ppb) in solution can be achieved. Graphitized carbon SPE sample preparation cartridges provide excellent flow properties for rapid cleanup of samples, using either vacuum pressure or gravity. The chromatographic grade packing material demonstrates reproducible recovery. This strong adsorbent has a wide range of selectivity, resulting in high capacity, even for analytes not usually well retained by reversed phase C18 adsorbents. For additional sensitivity, extracted acrylamide can be brominated, then quantified using an electron capture detector (ECD).[4]

References

1. Hileman, Bette, *C&E News*, July, 2002.
2. Schildhouse, Jill, *Food Product Design*, July, 2002.
3. http://www.cfsan.fda.gov/~dms/acrylami.html
4. U.S. EPA Method 8032A

Figure 3

Acrylamide can be detected in reduced fat potato chips, using an Elite-Wax ETR capillary GC column.



Elite-Wax ETR:	15 m, 0.53 ID, 0.50 µm
Sample:	1 g of potato chip spiked with 100 µg acrylamide
Inj.:	1.0 µL, 0.5 min hold
Liner:	2 mm splitless with wool
Inj. temp.:	260 °C
Carrier gas:	helium, constant pressure
Linear velocity:	62 cm/sec. @ 100 °C
Oven temp.:	100 °C (hold 0.5 min.) to 200 °C @ 15 °C/min.
Det.:	FID @ 260 °C

Product listing

A wide variety of Elite-Wax ETR columns is available, a subset of which is listed below:

Elite-Wax ETR Columns (Fused Silica)

(Crossbond® Carbowax®—provides oxidation resistance) Stable to 250 °C

ID (mm)	df (µm)	temp. limits	15-Meter	30-Meter	60-Meter
0.25	0.25	40 to 250 °C	N9316547	N9316549	N9316551
0.25	0.50	40 to 250 °C	N9316548	N9316550	
0.32	0.25	40 to 250 °C	N9316552	N9316555	N9316559
0.32	0.50	40 to 250 °C	N9316553	N9316556	N9316560
0.32	1.00	40 to 240/250 °C	N9316554	N9316557	N9316561
0.53	1.00	40 to 240/250 °C	N9316567	N9316569	N9316571
0.53	2.00	40 to 220/230 °C	N9316568	N9316570	

CarboPrep SPE cartridges

CarboPrep SPE cartridges are available through Restek Corporation.

PerkinElmer Life and Analytical Sciences
710 Bridgeport Avenue
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