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Applicationsnote

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 US 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.

Methodology & Results

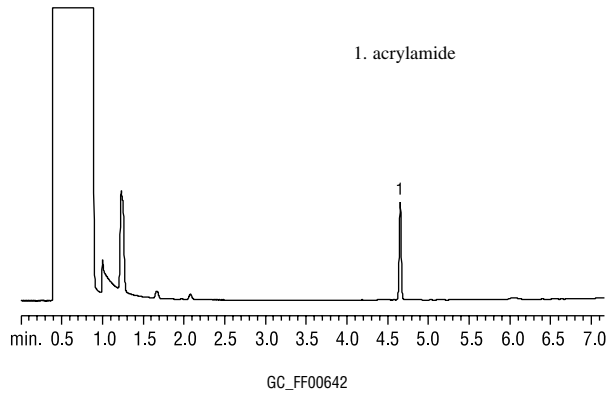
We used the following GC conditions in analyzing both acrylamide standards and food samples:

Column: Stabilwax® - 15m, 0.53 ID, 0.50µm film (cat.# 10637)
Inj.: 1.0µL, 0.5min. hold
Liner: 2mm splitless with wool (cat.# 20830)
Injector temp.: 260°C
Carrier gas: helium, constant pressure
Linear velocity: 62cm/sec. @ 100°C
Oven temp.: 100°C (hold 0.5min.) to 200°C @15°C/min.
Detector: FID @260°C

The chromatogram produced by injecting 1µL of a 25µg/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]

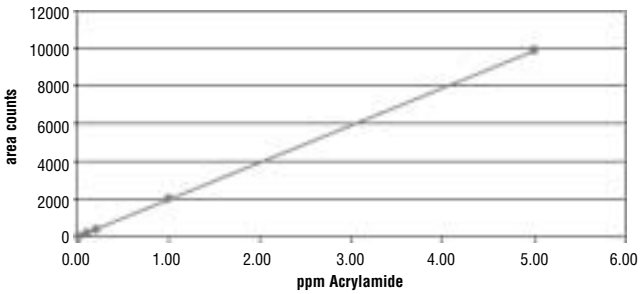
Figure 1
A Stabilwax® column is an excellent choice for acrylamide analysis by capillary GC.



Stabilwax® 15m, 0.53 ID, 0.50µm (cat.# 10637)

Sample:	25µg/mL acrylamide standard in water
Inj.:	1.0µL, 0.5min hold
Liner:	2mm splitless with wool (cat.# 20830)
Inj. temp.:	260°C
Carrier gas:	helium, constant pressure
Linear velocity:	62cm/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.

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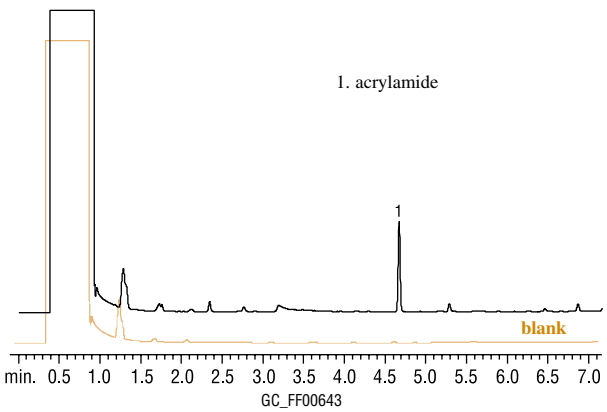
The procedure we used in our analysis of potato chips was as follows:

1. Analytically weigh 1g crushed potato chips.
2. Combine chip sample with 10mL 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 0.45µm nylon syringe filter (cat.#26071); remove and store for cleanup and analysis.
5. Condition CarboPrep™ 200 SPE tube, 6mL, 500mg (cat.#26087):
   a. 2mL acetone
   b. 2mL 0.1% formic acid
6. Apply 2mL 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 - 1.0mL water; pass through tube quickly.
   b. Use vacuum for up to 1 minute to dry excess water from tube.
8. Elute with 2mL of acetone, using gravity only. Eluate is ready for GC-FID analysis.

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 100µg (50µg/mL) of acrylamide for this analysis. The amount of acrylamide in the reduced fat chip sample alone was below the quantitation limit of this procedure.

Figure 3

Acrylamide can be detected in reduced fat potato chips, using a Stabilwax® capillary GC column.



Stabilwax® 15m, 0.53 ID, 0.50µm (cat.# 10637)
Sample: 1g of potato chip spiked with 100µg acrylamide
Inj.: 1.0µL, 0.5min hold
Liner: 2mm splitless with wool (cat.# 20830)
Inj. temp.: 260°C
Carrier gas: helium, constant pressure
Linear velocity: 62cm/sec. @ 100°C
Oven temp.: 100°C (hold 0.5 min.) to 200°C @ 15°C/min.
Det.: FID @ 260°C

Discussion

Gas chromatography offers a rapid, cost-effective approach to screening for acrylamide in food samples such as potato chips. The Stabilwax® capillary column exhibits excellent selectivity for acrylamide, even when analyzing complex matrices, such as food samples. Detection limits on the order of 0.01µg/mL (10 ppb) in solution can be achieved. CarboPrep™ 200 sample preparation cartridges provide excellent flow properties for rapid cleanup of samples, using either vacuum pressure or gravity. The chromatographic grade, graphitized carbon 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. US EPA Method 8032A

Product Listing

Ordering Information | Stabilwax® Columns (Fused Silica)

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

ID	df (µm)	temp. limits	15-Meter	30-Meter	30-Meter 6/pk.	60-Meter
0.25mm	0.10	40 to 250°C	10605	10608		10611
	0.25	40 to 250°C	10620	10623		10626
	0.50	40 to 250°C	10635	10638		10641
0.32mm	0.10	40 to 250°C	10606	10609		10612
	0.25	40 to 250°C	10621	10624		10627
	0.50	40 to 250°C	10636	10639		10642
	1.00	40 to 240/250°C	10651	10654	10654-600	10657
0.53mm	0.10	40 to 250°C	10607	10610		10613
	0.25	40 to 250°C	10622	10625		10628
	0.50	40 to 250°C	10637	10640		10643
	1.00	40 to 240/250°C	10652	10655	10655-600	10658
	1.50	40 to 230/240°C	10666	10669		10672
	2.00	40 to 220/230°C	10667	10670		

CarboPrep™ SPE Cartridges

SPE Cartridge	Tube Volume, Bed Weight	Surface Area	qty.	cat#
CarboPrep™ 200	3mL, 250mg	200 m^2/gm	50-pk.	26088
CarboPrep™ 200	6mL, 500mg	200 m^2/gm	30-pk.	26087
CarboPrep™ 90	3mL, 250mg	90 m^2/gm	50-pk.	26091
CarboPrep™ 90	6mL, 500mg	90 m^2/gm	30-pk.	26092

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