Measurement of Acrylamide in Potato Chips by Portable FTIR Analyzers

Application note

Food

Author

Alan Rein[1]
Professor Luis Rodriguez-Saona[2]

[1] Agilent Technologies,
Danbury CT, USA.

[2] Department of Food Science
and Technology
Ohio Sate University, OH, USA.

Introduction

Acrylamide is a compound that forms in french fries, potato chips, cereal, bread and coffee when they are fried, roasted or baked. The formation is dependent on cooking conditions, for example, high temperature over-cooking of fried chips results in very high levels of acrylamide. The substance, which forms from the reaction of sugar and amino acids in food, is reported to be a likely human carcinogen and formal regulation of acrylamide levels is under consideration worldwide. The compound is on California's Proposition 65 carcinogenic substances list, which requires a warning label on food products that contain elevated levels of a posted substance. Following legal action by the State of California, major potato chip manufacturers have agreed to reduce the level of acrylamide in potato chips to 275 ppb over the next several years. Acrylamide levels are of concern to European Union countries as well, and have been monitored in food for the past two years.

The current methods for determining the level of acrylamide in foods use GC-MS and LC-MS/MS, which require significant sample preparation, specialized instrumentation and knowledgeable operators. Food producers often turn to third party laboratories for the measurement of acrylamide. Thus, there is a need for rapid, accurate and real-time measurement of acrylamide to allow monitoring and controlling of acrylamide formation during food processing.

In a recent article [1], it was shown that a portable FTIR gave equivalent and in some cases better measurement of acrylamide when compared to a traditional lab FTIR and NIR spectrometers. This result was accomplished using a method that requires far less sample preparation than the LC-MS/MS method. In this application note, we summarize the findings of that work and demonstrate that FTIR spectroscopy employing compact, easy to-use FTIR analyzers provides acrylamide level measurements that are equivalent to the referenced mass spectroscopy method with less sample preparation.

Figure 1a. The Agilent 5500 FTIR analyzer equipped with diamond ATR sampling technology for use in at-site labs measuring acrylamide.

Figure 1b. The Agilent 4500 battery powered FTIR analyzer for similar measurements in out-of-lab applications.

This combination of technology and methodology provides more rapid, at-site acrylamide measurements and provides information to help control the cooking process conditions. The FTIR procedure also provides an easier and faster quality control method for measuring acrylamide levels in finished foodstuffs.

Compact at-site FTIR systems to develop and deploy methods for detecting acrylamide

Portable FTIR analyzers have significant value to the food industry. With increasing globalization of food sources, quality and safety is of paramount importance to consumers, regulatory agencies and the companies involved in growing and processing food and food ingredients. The value of portable FTIR analyzers arises from the ability to:

- Identify and verify food ingredients when and where needed.
- Eliminate adulterated, counterfeit or out-of-spec ingredients.
- Detect, identify and measure contaminants in food and food ingredients.
- Get answers at collection, receiving and processing sites, as well as at farms or fields.
- Aid in keeping harmful or out-of-spec ingredients and foods "at the factory gate".
- Allow actionable, on the spot decisions to proactively improve quality, safety and production efficiency.

Agilent offers a family of highly compact FTIR spectrometers and analyzers (Figure 1a and 1b), which all utilize the same optics, software and sampling technology [2]. If multi-purpose methods development and QA/QC is of primary interest, the Cary 630 FTIR spectrometer (not shown) is most suitable. For deployment of the FTIR based acrylamide method, the Agilent 5500 FTIR analyzer is recommended for at-site lab analysis. For analyses of acrylamide in non-lab environments, the Agilent 4500 battery powered FTIR analyzer is ideal. For the acrylamide FTIR method, these systems all employ diamond ATR sampling technology.

Experimental

Sixty-four different samples of potato chips were analyzed for acrylamide by infrared spectroscopy and by the referee mass spectroscopy method. To prepare the sample for infrared analysis, a hydraulic press compressed the potato chips into a "chip cake". This process forces excess oil out of the potato chips and reduces the strong infrared absorbance bands arising from the vegetable oil component. For each 10 g sample of potato chips, three chip cakes were prepared and the spectra were recorded. The chip cake was ground into a powder that was placed on a single reflection diamond ATR equipped portable Agilent FTIR analyzer. The resultant spectra consist of 64 co-added interferograms recorded at 4 cm^{-1} resolution in the 4000–700 cm^{-1} wavelength region.

Analyzer control and data acquisition was executed using Agilent Technologies MicroLab software and collected spectra were imported into Pirouette software (Infometrix Inc., Woodville WA) for multivariate analysis. The spectra were normalized and second derivative transformed via a Savitsky-Golay second order polynomial filter with a 25-point window prior to the partial least squares regression analysis (Figure 2).

Quantitative results were obtained by correlating the IR spectra with the acrylamide concentrations, as determined by the reference LC-MS/MS method. For independent validation studies of regular potato chips (not seasoned or sweet potato chips), 75% of the sample set was used for generating the calibration and the remainder served as the independent validation set.

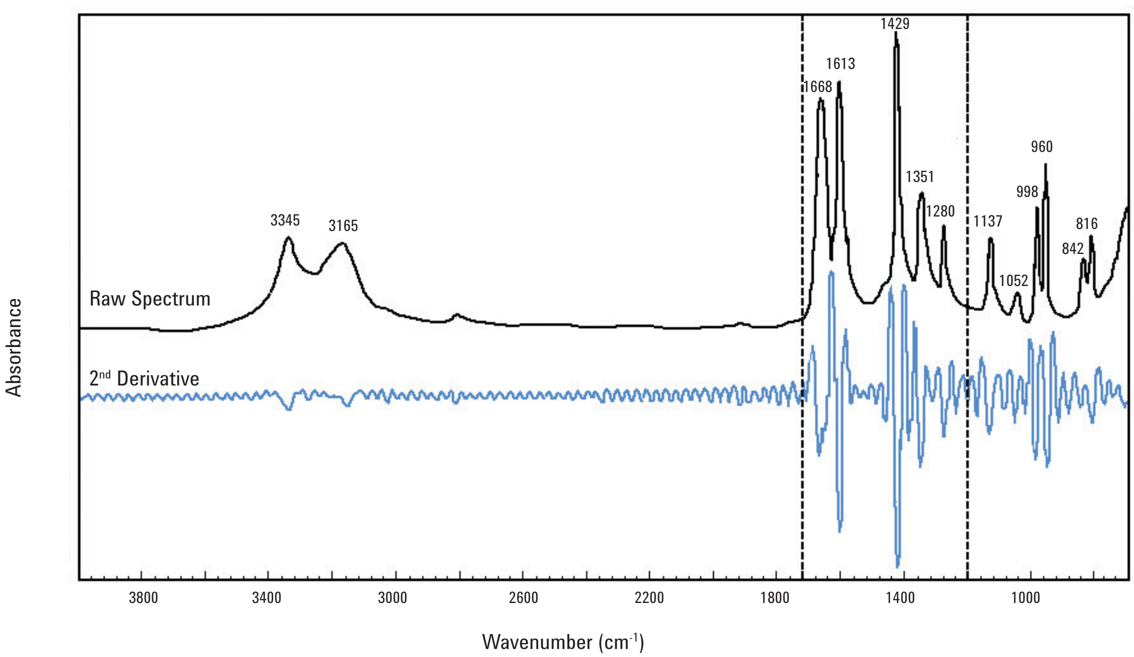

Figure 2. MIR spectrum and second derivative plot of acrylamide.

Results and Discussion

The acrylamide levels in potato chips, as determined by the reference LC-MS/MS method varied from 169 to 2453 µg/kg (169 to 2453 ppb). LOD and LOQ concentrations for the mass spec method were calculated to be 18 and 55 micrograms/liter.

Loading plots of the first two factors in the PLS correlation showed that the highest relevant variation in the calibration set for acrylamide content was in the range of 1201–1699 cm^{-1}.

The strong 1730 cm^{-1} absorbance band in the spectrum of potato chip cake (Figure 3) arises from fat, and is excluded in the PLSR model.

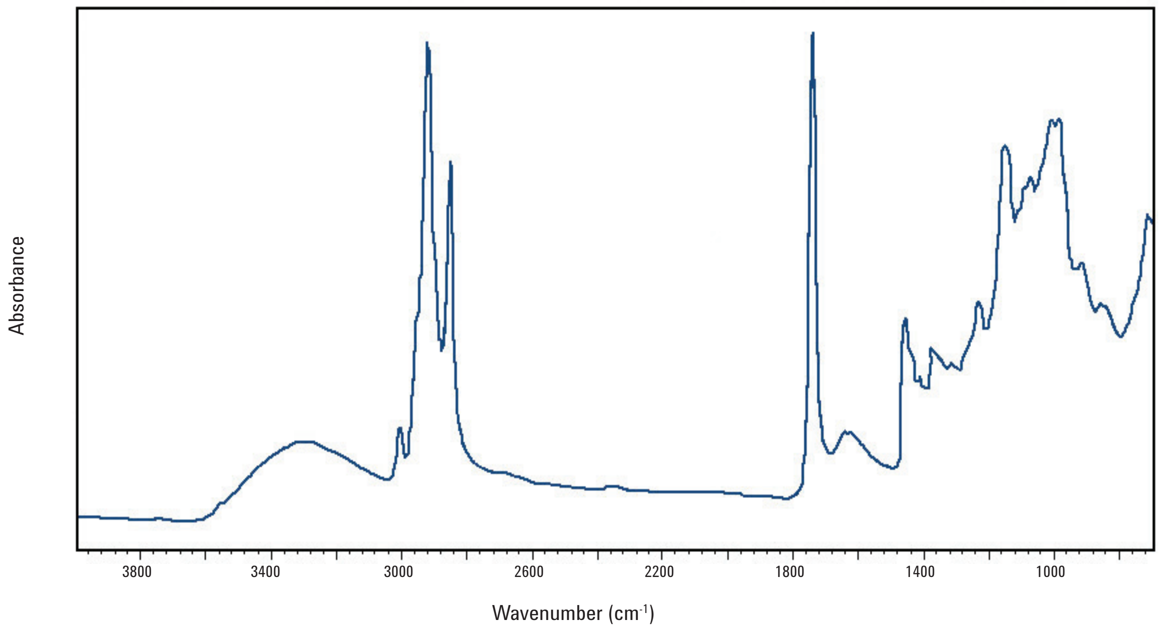

Figure 3. Spectrum of regular potato chip cake as measured by portable FTIR analyzer equipped with single reflection diamond ATR sample technology.

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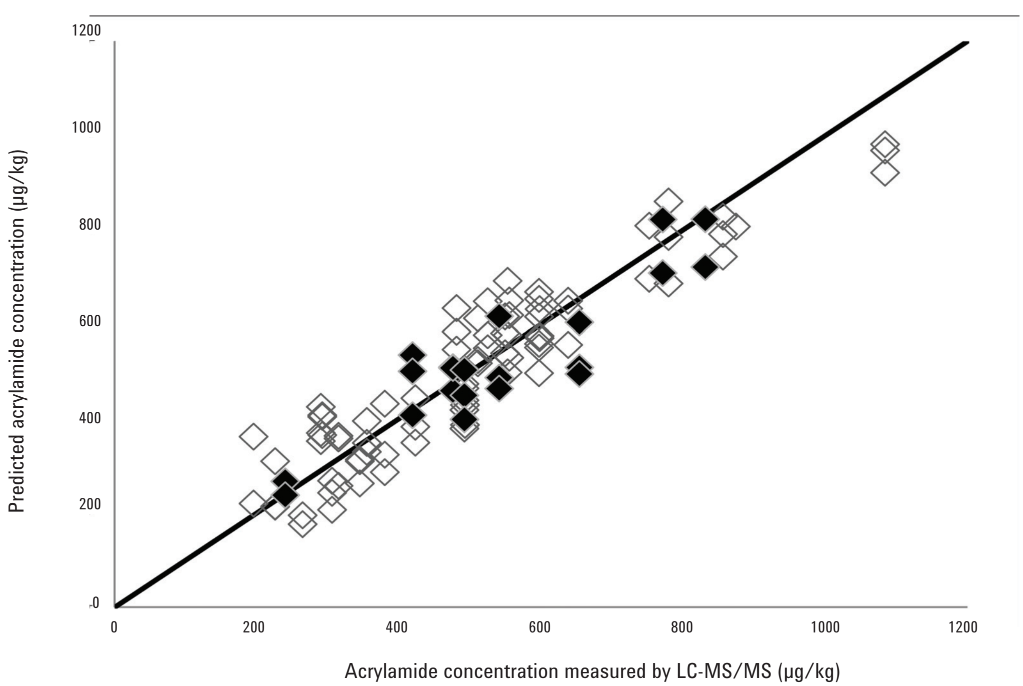

Figure 4. Correlation between infrared estimated levels and reference acrylamide values. The white squares represent samples in calibration groups; black squares represent samples used in validation groups.

Table 1. Calibration, cross-validation and prediction results of PLSR models developed by using mid-infrared instruments.

Sensor	Potato Chip Type		Factors	SE (µg/L)	r
Portable Cary 630 MIR	[a]Regular	Calibration	7	65	0.95
		Cross Val.	7	74	0.93
		Prediction*	7	75	0.90
	[b]Seasoned	Calibration	7	59	0.96
		Cross Val.	7	75	0.92
	[c]Sweet	Calibration	7	74	0.99
		Cross Val.	7	98	0.98

[a] "Regular" refers to potato chips containing only potatoes, vegetable oils and salt.
[b] "Seasoned" refers to potato chips containing additional ingredients.
[c] "Sweet" refers to sweet-potato chips.
* independent variable predictions made on regular potato chips only

The portable FTIR analyzer system equipped with single reflection diamond ATR sampling interface performed exceptionally well (Figure 4 and Table 1) with a SEP value for regular potato chips of 75 µg/kg (regular chips contain only potatoes, vegetable oils and salt).

Seasoned and sweet potato chip groups had SECV of 75 and 98 µg/kg, respectively. These ppb level measurements are consistent with current "acceptable" amounts of acrylamide in potato chips.

Conclusion

Agilent portable FTIR analyzers provide quantitative measurement of acrylamide levels in potato chips that is equivalent to, or better than, results obtained from traditional bench lab MIR or NIR spectrometers. The measurement of acrylamide in potato chips by FTIR provides a simpler analysis method for this potentially hazardous by-product than the current mass spectroscopy approach. The FTIR method eliminates consumables such as solvents, cartridges and columns and is far more efficient than the mass spectroscopy method. The level of user expertise necessary to obtain reliable results is not as critical with the FTIR method and, most importantly, it can be deployed wherever acrylamide needs to be measured, whether in a lab, a potato chip production facility, or for spot check measurements to determine levels in consumer product.

Acknowledgement

The data and results presented in this application note were provided by Professor Luis Rodriguez-Saona's research group at the Food Science and Technology Department of Ohio State University.

References

[1] Ayvaz, H. and Rodriguez-Saona, L., Application of Handheld and Portable Spectrometers for Screening Acrylamide Content in Commercial Potato Chips, submitted for publication to the Journal of Agricultural and Food Chemistry (2013).

[2] Agilent's FTIR family—lab results, anywhere you want" Agilent Publication 5991-1405EN, October 26, 2012.

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Published June 21, 2013
Publication number: 5991-2532EN

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