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Analysis of Acrylamide in Food by GC/MS

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A positive chemical ionization GC/MS method for the analysis of acrylamide monomer in foods will be described and future areas for development offered. The method had a limit of detection of 5 ppb and was found to show good linearity to 1000 ppb. PCI SIM using ammonia as a reagent gas provided the best blend of sensitivity and selectivity.

To adequately assess this risk to humans, food levels need to be accurately measured and compiled. This prompts the need for development of analytical methods for extraction and quantitation of acrylamide. The United States Food and Drug Administration (FDA) has published an LC/MS/MS method for acrylamide, and others have also published methods using either GC, GC/MS, or LC/MS (7,8). Published detection limits for acrylamide in food range from 10–50 ppb depending on instrumentation. Here, the Trace DSQ™ is used to analyze acrylamide with linear dynamic range extending from 5–1000 ppb.

Experimental Conditions

One gram of unknown sample and 100 μ L of $^{13}\text{C}_3$ -Acrylamide solution (2000 ng/mL) were combined in 10 mL water. Standards were prepared in water as well, with 100 μ L internal standard added to each. Samples were mixed for 20 min, then centrifuged. The supernatant was filtered using 0.45 μ m nylon syringe filters. 200–300 μ L of brominating reagent were added to 3 mL of the filtered sample and standards, which were gently mixed, and then allowed to react in an ice bath for 1 h. One drop of 1.0 N sodium thiosulfate was added to each sample to decompose any remaining bromine. Samples were extracted with 2 mL ethyl acetate, then centrifuged for 10 min. The organic layer was transferred to autosampler vials for analysis. Bromination of acrylamide according to this method yields 2,3-dibromopropionamide. Analysis of the more stable 2-bromopropenamide, created in situ from thermal decomposition in the injector, using ammonia as a reagent gas, provided stable adduct ions that were used.

The Trace DSQ was set up using the PTV injection port in splitless mode, utilizing programmed transfer, evaporation, and cleaning phases and a 0.3 min splitless duration. The oven program ramped from 40–220 °C at 30 °C/min. A 15 m \times 0.25 mm i.d. \times 0.25 μ m Stabilwax™ Crossbond® Carbowax® column (Restek Corp) was chosen as the analytical column. Ammonia was used as the reagent gas with a flow rate of 2.0 mL/min.

Results

The use of ammonia in PCI to analyze the 2-bromopropenamide form of acrylamide leads to the formation of two primary adduct ions, at m/z 167 and 169 (Figure 1) for the unlabeled form and 170 and 172 for the ^{13}C -labeled compound. A method was set up using these masses, and calibration from 5–1000 ppb was performed in PCI, with a correlation coefficient of 0.9998 (Figure 2).

Conclusion

The reported method has a linearity range from 5 ppb to 1000 ppb. Use of the Trace GC Ultra with a PTV enables the user to program evaporation, transfer, and cleaning steps that facilitate sample transfer to the analytical column and help eliminate carry-over and matrix interference. Future developments will focus on even lower detection limits with larger injection volumes.

References

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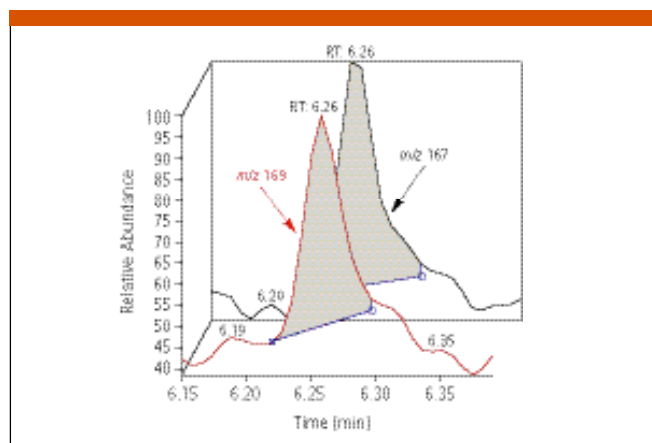


Figure 1: EIC for m/z 167 and 169, PCI SIM for 2-bromopropenamide, 5 ppb standard.

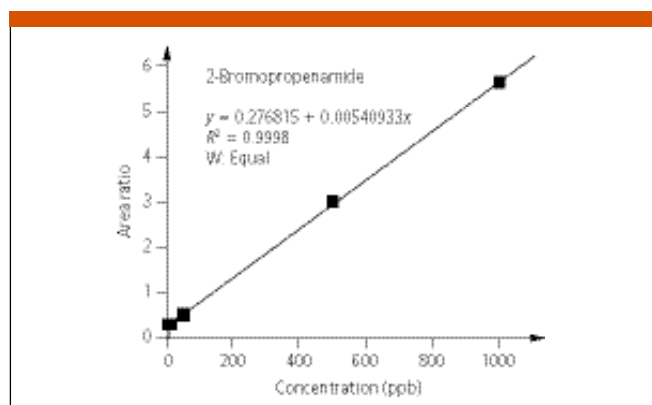


Figure 2: Calibration curve for brominated samples in PCI SIM using ammonia for reagent gas, from 5–1000 ppb, $r^2 = 0.9998$.

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