



C H E M I R

Analytical Services

Confronting Food & Beverage Quality Failures with Analytical Chemistry

Quality issues or product failures can cause manufacturing shut-downs, customer complaints or even legal disputes. These problems include contamination, off-flavors/colors/odors, toxic substances, migration/leaching and packaging failures. When these non-routine problems occur, food and beverage manufacturers may need to call on an outside analytical laboratory. These facilities feature state-of-the-art instrumentation and experienced scientists that can quickly interpret data and provide reliable answers.

Chemir Analytical Services has helped many food and beverage manufacturers confront tough quality issues. The following are case studies illustrating common quality failures and how our scientists found answers.

Product Contamination from Outside Sources

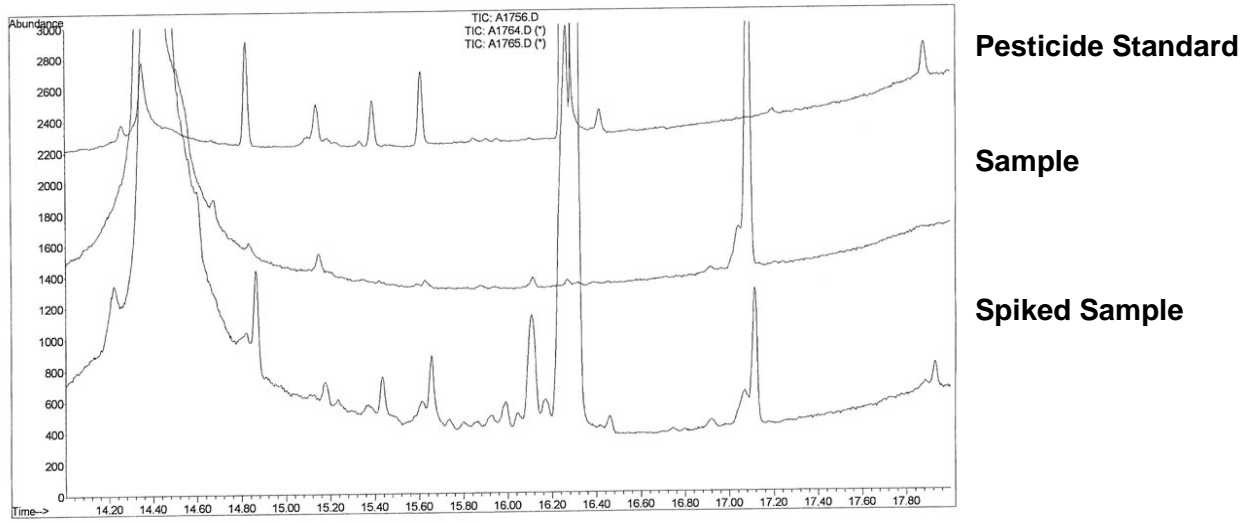
Often, a food or beverage product can become contaminated by an outside source. This can be identified by an off-flavor, color, or odor or even by visual indicators such as particulates or unexpected separations. Possible causes of product contamination can be migration or leaching from the packaging or process, incompatible raw materials, or improper storage.

Possible Pesticide Contamination

A door at a meat processing plant was left open while a pesticide was being sprayed. The processor came to Chemir to determine if their product had been contaminated. Chemir was contacted on a Sunday afternoon and results were needed by 8am Monday morning because 2000 butchers were holding operations.

We obtained a sample of the actual pesticide used, which was a mixture of 3 main components. First, samples of the meat from different locations were pooled to allow for faster sampling. While the samples were being shipped to our laboratory, Chemir scientists quickly performed method development experiments to ensure low limits of detection. Once the samples arrived, the meat was extracted using methylene chloride. Gas Chromatography/Mass Spectrometry (GC/MS) was then used to screen the methylene chloride extracts, looking for these 3 components. When none of the analytes were observed in the meat extracts, a portion of the meat was intentionally spiked with the pesticide at levels below the allowable limits (ppb levels) to confirm the effectiveness of the test method. All 3 analytes were observed in the spiked sample at the appropriate levels.

Figure 1: GC/MS total ion chromatograms from the analysis of the pesticide, sample and pesticide spiked sample

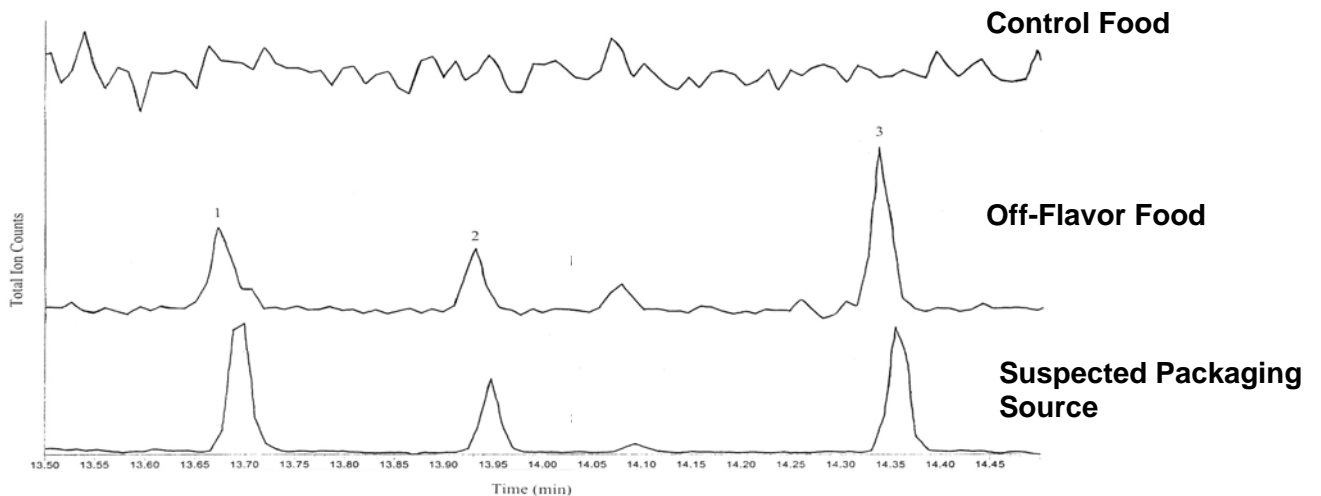


Out of the 36 samples analyzed, all were found to be free of the pesticide components below detection limits. After a conference call with local meat inspectors as well as the USDA in Washington DC, the testing protocol was accepted and the meat was released. The processor was able to resume operations on Monday morning.

An Off-Flavor Food

A client determined their food product had an off-flavor and suspected the secondary packaging material was the contamination source. Our scientists performed comparative analysis of a control batch, the contaminated batch, and the suspected packaging source using Solid Phase Microextraction (SPME) GC/MS. The results clearly identified the secondary packaging as the source of the offending components.

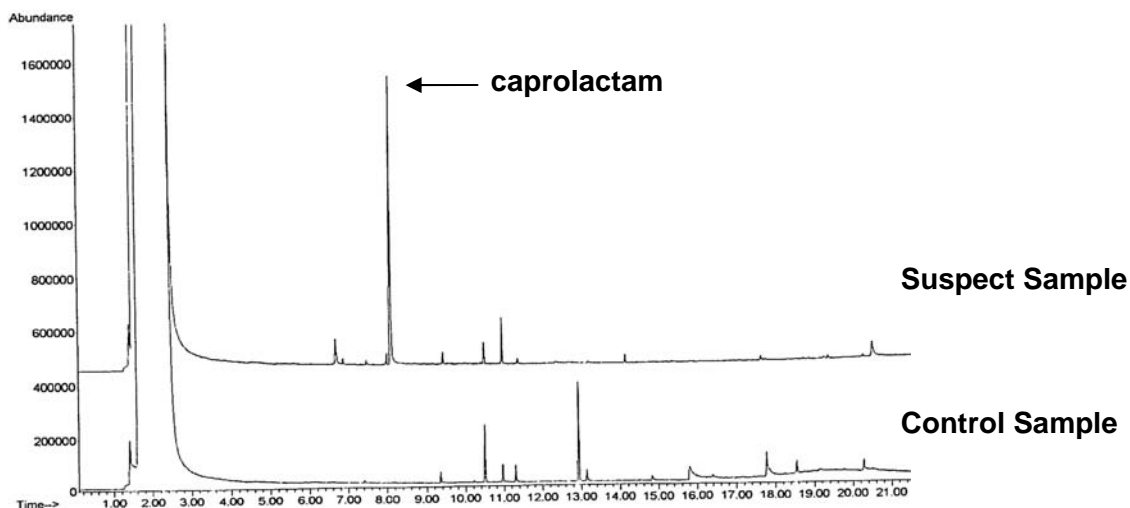
Figure 2: GC/MS total ion chromatograms of the control food, off-odor food, and the suspected source



Ink Migration

A client observed an off-flavor in a pre-packaged, alcohol based beverage. Comparative analysis was performed on suspect and control samples of the product by GC/MS to look for potential contaminants or impurities contributing to the off-flavor.

Figure 3: GC/MS total ion chromatograms of the suspect and control sample



Significant levels of caprolactam were found in the suspect sample, but not in the control. Caprolactam is an alcohol-soluble component of the ink used on the outside surface of the lids. During manufacturing, the lids were stacked, allowing for transfer of ink components onto the food-contact side of the lids. This led to the migration of caprolactam into the alcohol based product. By inserting a slip sheet between layers of lids, the manufacturer was able to prevent future issues.

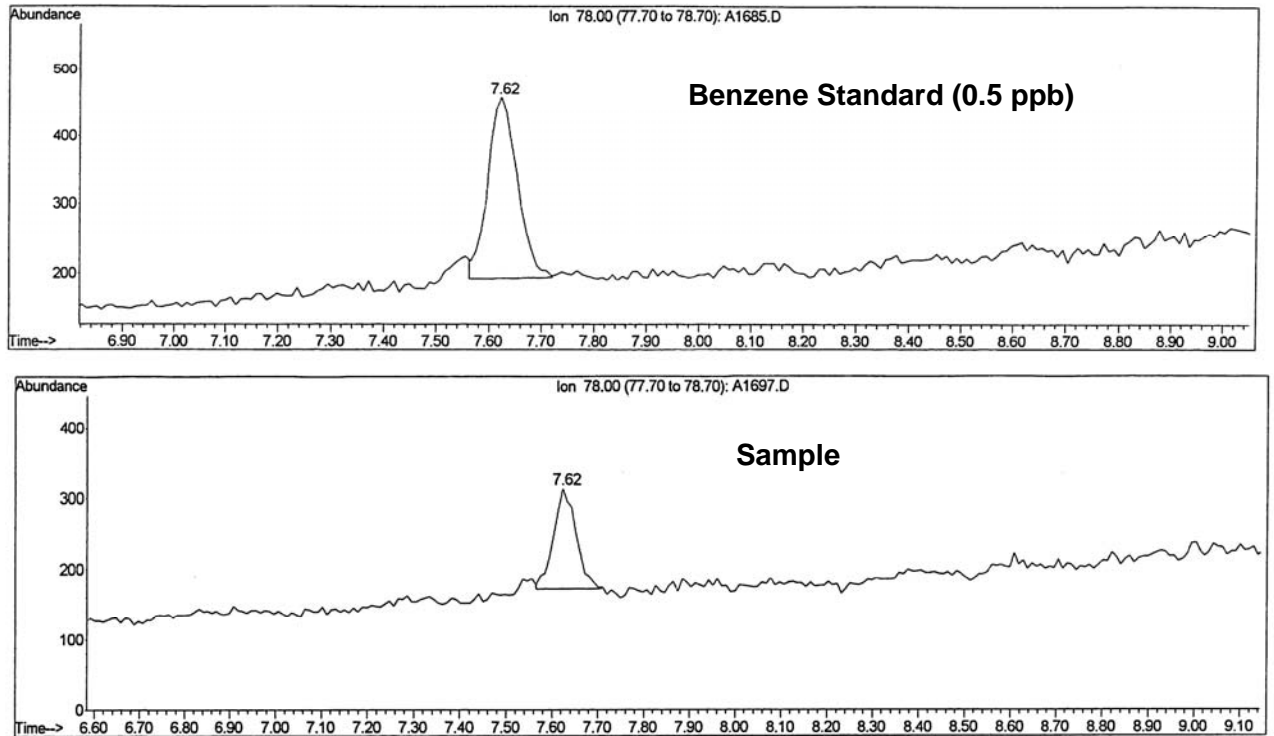
Toxic Chemicals

Occasionally, toxic chemicals are generated in a product by degradation or a cross-reaction. Possible causes can be contaminated raw materials, incompatible raw materials or improper storage of raw materials or final products.

Benzene in Beverages

Due to media and consumer concerns regarding benzene in beverages, a client decided to be proactive and asked Chemir to test their orange drink for benzene before they put it on the market. We performed headspace GC/MS on samples at room temperature and samples that were incubated for two weeks at elevated temperatures. As shown by the figure below, we found very little benzene in the drinks. The amount observed was slightly below the reporting limit of 0.5 ppb.

Figure 4: GC/MS extracted ion chromatograms of a 0.5ppb benzene standard and the orange drink



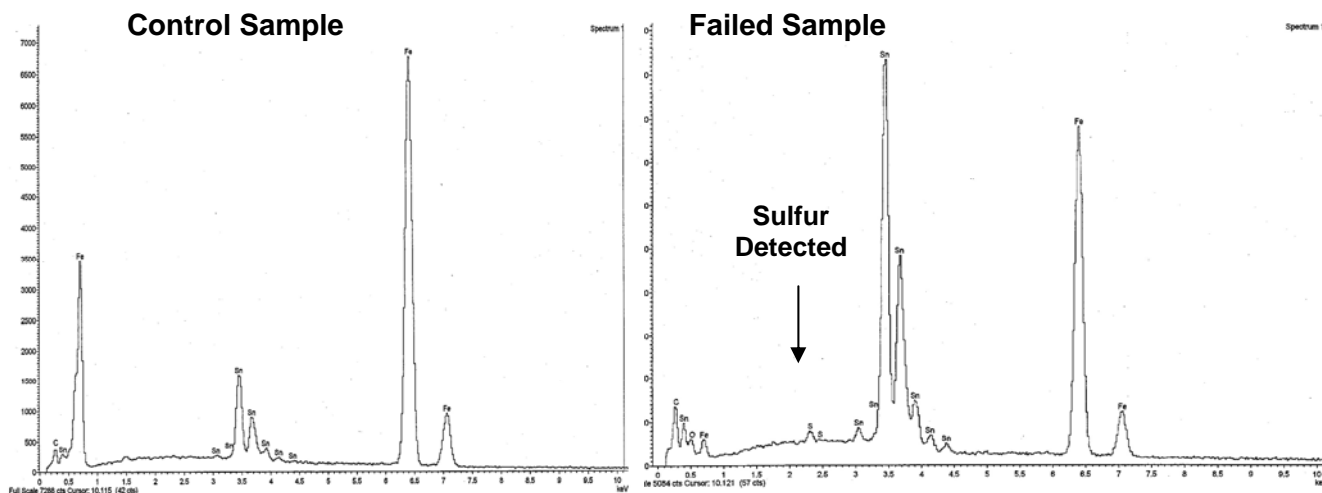
Packaging Failures

Often, packaging failures such as packaging degradation, seal failure, or contamination can cause issues for food processors. Possible causes can include material failure and/or substitution, contamination, or processing issues.

Peeling Cans

An epoxy liner was delaminating from the inside of a manufacturer's metal can. The scientists at Chemir performed a comparison study between a control and failed sample. We investigated the possibility of an incorrect epoxy application by cross sectional analysis and microscopy for film thickness. Insufficient epoxy curing was investigated by Modulated Differential Scanning Calorimetry (DSC). Finally, possible contamination was inspected by solvent extraction with GC/MS, Fourier Transform Infrared (FTIR) Spectroscopy and surface analysis using Energy Dispersive X-Ray Analysis (EDXA).

Figure 5: EDXA spectral data of the control metal surface and the suspect metal surface

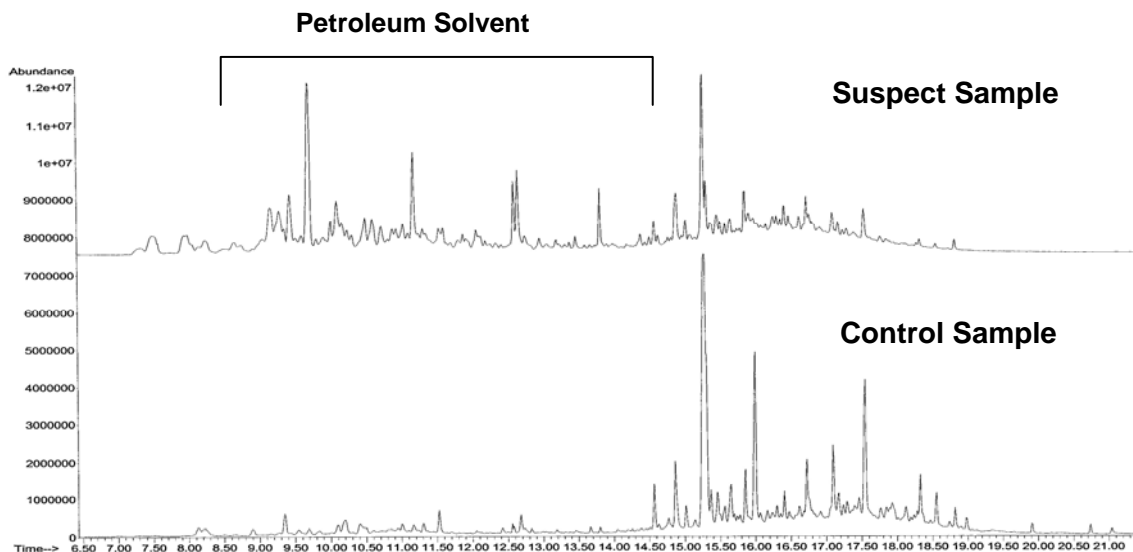


The only detectable difference between the samples was the presence of sulfur on the metal surface of the failed sample. Our client was able to bring the data to the can manufacturer so they could fix the problem.

Off-Odor Boxes

A food manufacturer found an off-odor in boxes imported from China. Our scientists performed headspace GC/MS to determine the cause of the odor. We found a profile consistent with a low molecular weight hydrocarbon solvent such as mineral spirits or naphtha. The client used this data to confront the box manufacturer. The manufacturer confirmed that they had accidentally used naphtha in place of isopropanol to clean the boxes prior to shipping.

Figure 6: SPME GC/MS total ion chromatograms of off-odor box and control box



Conclusion

Most companies are familiar with working with outside laboratories for routine testing, such as nutritional analyses, microbiological testing, or shelf life studies. However, when unusual problems occur, non-routine laboratories are called upon to troubleshoot quality issues and product failures.

Rather than specializing in one technique, a non-routine laboratory has experience solving complex problems that require unique and inventive approaches, often under a time crunch. These labs will often have Ph.D. scientists designing methods and experiments, as well as supervising the testing and interpreting the results.

Additionally, the potential for legal claims can make it necessary to use an independent laboratory. Patent infringement, product failures, product recalls or other legal issues may require a third party to review reports, interpret technical data or provide expert testimony.

As quality issues in the food and beverage industry continue to make the news and concern us all, it is important to keep in mind the reliable and cost-effective resources available in independent analytical laboratories.