

Ensuring Food Ingredient Quality and Consistency with a Novel Mass Detection Technology

BACKGROUND

Kalsec® is the leading global producer of natural spice and herb flavor extracts, colors, antioxidants, and advanced hop products for the food and beverage industry. Kalsec's high-quality, innovative ingredient solutions enable their customers to improve the taste, appearance and shelf-life of a highly diverse range of end products. The company maintains research and production operations in the United States, the United Kingdom, China, and Kuala Lumpur, including Application Centers staffed with experts in analytical chemistry and sensory evaluation with extensive food industry experience.

Kalsec's Analytical Research & Development laboratory is located at the company headquarters in Kalamazoo, Michigan. The lab has wide-ranging responsibilities, which include supporting the analytical needs of QC, R&D, Manufacturing, Sales, and Procurement groups. The lab of five full-time scientists analyzes approximately 300 to 500 samples per month that represent all product lines and require multiple assays. Technologies routinely employed by the laboratory include liquid chromatography (LC), gas chromatography (GC), photo diode array (PDA), fluorescence (FLR), mass spectrometry (MS), and fourier transform infrared spectroscopy (FTIR).

A critical role of the Analytical R&D laboratory is the development, optimization and validation of new sample test methods that are transferred to QC. The Quality Control laboratory continually faces a very high volume of samples and as a result runs a three shift operation with 12 technicians. The lab is tasked with both in-process and final product quality assessments and utilizes several techniques for its analyses such as HPLC-UV, GC, titrations, and wet chemistry.



TECHNOLOGY

- ACQUITY UPLC® H-Class System
- ACQUITY® QDa® Mass Detector
- Empower® 3 Chromatography Software



kalsec®



All peppers contain capsaicinoids, the most abundant being capsaicin (8-methyl-N-vanillyl-6-nonenamide), an active component that produces a burning sensation when in contact with the tissues of mammals.

CHALLENGE

Kalsec®'s broad product portfolio includes a line of capsicum extracts that are used in a wide variety of food applications. Capsicum is a genus of flowering plants more commonly known as peppers. Kalsec® extracts chili peppers for chili extract and sweeter peppers for paprika extract. All peppers contain capsaicinoids, the most abundant being capsaicin (8-methyl-N-vanillyl-6-nonenamide), an active component that produces a burning sensation when in contact with the tissues of mammals. The capsaicinoids family also includes dihydrocapsaicin, nordihydrocapsaicin, homocapsaicin, and homodihydrocapsaicin, all of which contribute to the heat associated with chili peppers. Given that Kalsec® produces both chili extracts (high levels of capsaicinoids) and paprika extracts (low levels of capsaicinoids) the laboratories must have the ability to quantitate over a broad concentration range in the presence of high levels of matrix.

Previously, Kalsec®'s Analytical R&D laboratory had developed a 15-minute HPLC-UV/FLR assay for the quantification of low-level capsaicinoids. The method is routinely used by QC as part of the capsicum extract (including paprika extracts) formulation process. Addition of capsicum extracts to food products enables precise management of the amount of heat experienced by consumers, which is an important characteristic that can drive product desirability and value in the marketplace.

Unfortunately, given that a typical flavor extract is quite complex, and is often comprised of a mixture of several spices and herbs, the legacy HPLC-UV/FLR assay could not consistently quantify target compounds of interest. Whether utilizing PDA or fluorescence detection, QC technicians were challenged with significant matrix interference and the presence of co-eluting analytes. The inherent limitations of the assay ultimately compelled the Analytical R&D team to seek out an alternative methodology, one that could help improve both data quality and decision making in capsicum extract production.

“ The UPLC-MS based assay can be completed in 4 minutes compared to the legacy 15-minute HPLC-UV/FLR method – a 73% reduction in run time.”

SOLUTION

In an effort to develop a new assay for capsaicinoids that would address the shortcomings of the legacy HPLC-UV/FLR approach, Analytical R&D implemented a method utilizing UltraPerformance Liquid Chromatography® (UPLC) separation coupled with MS detection. This integrated system was comprised of the following technologies:

- **ACQUITY UPLC H-Class System** – Provides the flexibility of a quaternary-based HPLC with the performance advantages of UPLC. The H-Class simplifies method development and method transfer, enabling laboratories to run existing HPLC methods, and then transition to UPLC separations on the same system.
- **ACQUITY QDa Mass Detector** – A novel mass detector that can be integrated into existing liquid chromatography configurations, allowing any scientist to generate high quality mass spectral data without advanced training. With the ACQUITY QDa, an analyst has the ability to detect and quantify compounds with no UV response, at levels not amenable to or accessible by optical detectors (PDA or FLR).
- **Empower 3 Chromatography Software** – A compliance-ready chromatography data system (CDS) for advanced data acquisition, processing, and reporting that allows laboratories to simplify the management of chromatography test results from multi-vendor instruments.

The new UPLC-QDa capsaicinoids assay was successfully optimized and validated by Analytical R&D and then transferred to a second UPLC-QDa system in Kalsec®'s QC laboratory. The method is now used for all in-process and batch release analyses of capsicum extracts. Given the quality of results obtained with the new method and ease of implementation, the Analytical R&D team is now investigating leveraging the UPLC-QDa system for additional assays in other product areas.



BUSINESS BENEFITS

Kalsec®'s decision to transition from a HPLC-UV/FLR based method for capsaicinoid quantification to one that combines UPLC with the latest mass spectrometry technology is representative of an ongoing commitment to produce highly innovative ingredient solutions. As a result of implementing this new approach to sample analyses, the company has noted several scientific and operational benefits, including:

Sample throughput and productivity

- The UPLC-MS based capsaicinoids assay can be completed in 4 minutes compared to the legacy HPLC-UV/FLR method which required 15 minutes – a 73% reduction in run time. For Kalsec®, the rapid analysis time means greater sample throughput, faster method development, and the opportunity for analysts to allocate time to additional projects.
- The new mass spectrometry technology was easily incorporated into the QC laboratory workflows. Despite having no previous experience with MS systems, QC technicians were able to have the UPLC-QDa system up and running quickly.

Lower operating costs

- Solvent consumption for analyses was reduced from 22.5 mL per run with traditional HPLC to 2.8 mL per run with the ACQUITY UPLC H-Class System – a decrease of 88%. This has translated to lower costs for the acquisition and disposal of solvents.

Data quality

- The inclusion of mass detection in the new method has improved sensitivity, enabling the quantitation of capsaicinoids present at very low levels in some of the formulations. Additionally, co-eluting analytes that had appeared as single peaks and could have been missed by optical detection are now discernible. With ACQUITY QDa, the QC technicians have far more confidence in their ability to effectively characterize samples.

Product quality and decision making

- Variances to the capsicum extracts composition encountered during manufacturing can now be investigated and adjusted more rapidly, helping to ensure product consistency and quality.
- The shorter analysis times achieved with the UPLC-QDa method have allowed the QC laboratory to determine whether the final product is within desired specification and release the lot to shipping at an accelerated pace.

Waters

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Waters Corporation
34 Maple Street
Milford, MA 01757 U.S.A.
T: 1 508 478 2000
F: 1 508 872 1990
www.waters.com