

MEETING THE CHALLENGES  
OF RAW MATERIAL, IN-PROCESS,  
AND FINAL PRODUCT FOOD TESTING



Waters

THE SCIENCE OF WHAT'S POSSIBLE.™



## QUALITY CONTROL AND NUTRITIONAL TESTING IN FOOD MANUFACTURING

### GLOBAL DEMAND AND COMPETITION

On-going consumer demand for better food quality and nutritional content coupled with increasing global competition has generated a need for increased compositional testing and quality control (QC) by food manufacturers and processors. Powerful, yet cost-effective, analytical solutions are required to meet this demand and to ensure companies maintain a competitive edge in a market that has proved very sensitive to food and product safety scares.

### CONSUMER PROTECTION

Effective testing is essential for food manufacturers:

- To protect consumer health and ensure consumers receive the quality of food they expect
- To ensure claims of nutritional and 'value-added' content of foodstuffs are substantiated
- To ensure compliance with food regulations operating in each country
- To build and protect brand image and reputation

### FOOD QC TESTING

Extensive testing of food and beverages is required in manufacturing, ranging from testing for contaminants (accidental or deliberate), to monitoring and developing the composition, to end product testing such as taste and flavor to ensure quality and consistency. Recent food safety scares have highlighted the importance of effective QC for both consumer protection and for minimizing the business risks associated with a product recall or failure.

### COMPOSITION AND LABELING

To meet regulatory requirements for the declaration of food ingredients on product labels, manufacturers must analyze foods for a variety of compounds, including:

- Amino acids
- Carbohydrates and sugars
- Fats
- Additives and preservatives
- Vitamins and minerals

## FUNCTIONAL FOODS

In many modern populations, eating habits stemming from busy lifestyles do not support a well-balanced diet. This has resulted in functional ingredients, such as those in Table 1, being included in commodity products where they are then marketed as 'functional foods'. As one of the fastest growing sectors in the food industry, functional foods allow the consumer to take advantage of a variety of supplemental nutritional benefits. The development and production of functional foods is a high priority for many leading food manufacturers.

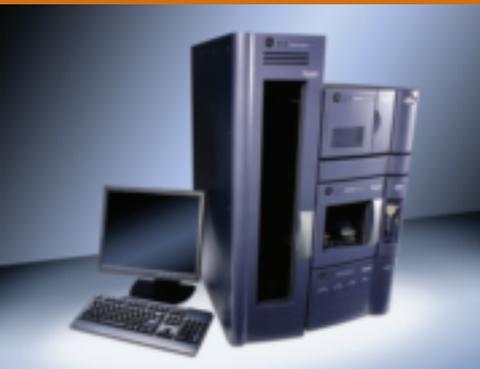
Functional food ingredient	Health benefit
Essential omega-3 fatty acids (Fish oil : LC-PUFAs: EPA, DHA)	Heart health & mental acuity, may help reduce osteoporosis
Soy isoflavones (genistein)	May help with heart health and breast cancer
Probiotics prebiotics, synbiotics	Digestive health
Carotenoids: Lycopene, Lutein, Zeaxanthin, , $\alpha$ & $\beta$ -carotene	Natural antioxidants
Polyphenols	Antioxidant characteristics. May reduce the risk of cardiovascular disease and cancer
Anthocyanins	May help against cancer
Superfruits	Antioxidant content benefits
Folic acid	Reduce neural tube defects (NTD)
Vitamins	Promote & regulate metabolic reactions within the body (growth, health, and reproduction)
Gingko biloba	Mental acuity & brain health

Table 1. Functional food ingredients and associated health benefits.

## THE WATERS SOLUTION

Waters will help your laboratory meet the challenges of food QC and nutritional testing with solutions that offer the sensitivity, stability, reproducibility, and versatility required to ensure you can profitably manage lab requirements. With end-to-end solutions including chromatography, mass spectrometry, column and sample prep chemistries, and data management software, our technologies continue to push the limits of key food testing applications. Look to Waters as a partner that delivers scientific expertise, flexible and superior service, and a commitment to your success.

## UPLC: FOR UNRIVALED SENSITIVITY, RESOLUTION, AND SPEED



UltraPerformance LC® (UPLC®), the industry standard in separation science, significantly reduces the time and cost per sample from analytical processes while improving the quality of results.

Compared to HPLC systems, UPLC run times are up to nine times faster, have up to twice the peak resolution, and are up to three times more sensitive.

UPLC synergistically combines unique instrumentation capabilities, column chemistries, and software for data acquisition and processing that yield more information, increase sample throughput, alleviating workload challenges.

When coupled with Waters' high performance mass spectrometry systems, the ACQUITY UPLC® System provides a powerful solution for your food testing needs.

## ANALYTICAL CHALLENGES IN FOOD MANUFACTURING

Today food manufacturers must meet the challenges presented by rapidly increasing demand, the global nature of food trade, the need to ensure consumer safety, and the desire to build brand value and commercial competitiveness.

The sourcing of raw materials can include products from diverse geographies. Ensuring the consistency of supply and product composition is essential in maintaining product safety, quality, and build trust in customer perception

### Raw material consistency

Many manufacturers are now seeking to profile raw materials using rapid and powerful analytical techniques capable of detecting contaminants and impurities before materials are processed.

Profiling materials of differing origin and detecting a wide range of potential impurities can prove time-consuming and costly, yet is an increasingly necessary requirement for leading food companies.

### In-process monitoring

Monitoring of manufacturing processes has long been recognized as critical to cost-effective operation and optimization of resources. Many plants have invested in real-time monitoring and feedback of physical parameters but it is increasingly recognized that monitoring of chemical components can also be of real value.

Advances in separation technology today make "at-line" or even "online" monitoring a realistic proposition, enabling rapid, robust chromatographic analysis of in-process samples.

### Final product quality

In addition to the established requirements for QC testing of final product composition and quality, the demand for new products with added nutritional value has added a further testing burden to manufacturers. QC labs are increasingly faced with demands for improving workflow by delivering rapid methods, faster method development, and enhanced method performance.

### Confidence in results

Throughout the production process, there is a high demand for simple, repeatable, and reliable testing techniques. Labs need to be able to test complex food samples consistently and routinely and have high confidence in the results and instrumentation.

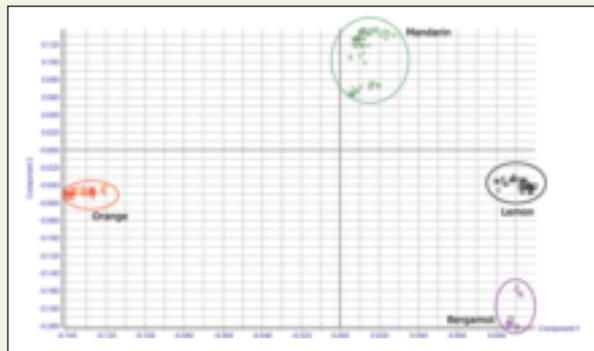


Figure 1. Scores plot illustrating differentiation of 25 essential oil samples.

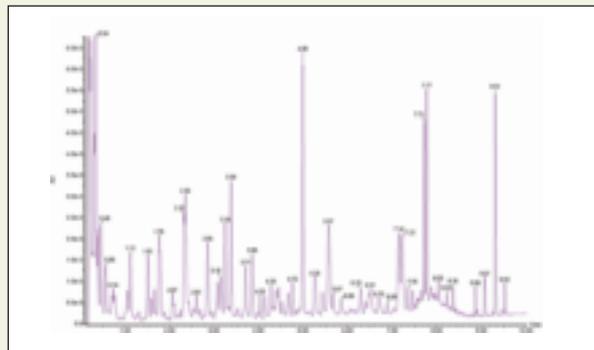


Figure 2. UPLC separation of complex mixture of polyphenol compounds in fruit juice.



## FOOD QC WORKFLOW

### Raw material testing

Verifying ingredient quality with methods ranging from composition to contamination analysis

### In-process / production testing

Assessing manufacturing processes, reaction monitoring and intermediate products

### Final product testing

Ensuring end product meets expected quality; and is safety and labeling compliant

## ANALYTICAL TESTING IN FOOD MANUFACTURING

### Raw Material testing

#### Issues:

- Material quality
- Material purity
- Seasonal variations
- Adulteration
- Traceability

#### Business impact:

- Variability can affect processing efficiency and final product quality, resulting in reduced production capacity and product value.
- Traceability of ingredient history is essential for meeting certain certifications such as Halal and Kosher production requirements as well as for avoiding allergen contamination. Failures in such systems can result in costly product recalls and damage to brand image.

### In-process / production testing

#### Issues:

- Process water quality
- Reaction monitoring
- Troubleshooting
- Process optimization

#### Business impact:

- Process downtime is very costly and undesirable. Effective monitoring of key parameters can avoid downtime and support optimization of methods which reduce cost and improve productivity.
- In batch production processes, monitoring ensures efficient removal of previous product which can facilitate production scheduling and avoid cross-contamination problems.

### Final product testing

#### Issues:

- Ensuring product quality
- Checking compositional requirements/claims
- Meeting labeling requirements

#### Business impact:

- Food alerts associated with contaminated, or non-conforming products can prove disastrous. Product recalls are very costly and damage to consumer confidence seriously affects brand image and purchasing decisions.
- Increasing differentiation of new products based on “added nutritional value” should be supported with evidence from testing.

## WATERS SOLUTIONS

### ACQUITY UPLC

- Improves lab efficiency and delivers excellent return on investment for QC labs.
- Fast turnaround for release of final products or evaluation of raw materials.
- Fast, high resolution of complex mixtures for effective profiling and contaminant detection.
- Dedicated solution for fast, reliable amino acid analysis (AAA)
- Rapid, simplified method development and validation supported by Empower™ 2 Software.
- The PATROL™ UPLC Process Analyzer System allows direct integration of UPLC into the production process reducing the impact of turnaround times

### Mass spectrometry detection

- ACQUITY® SQD offers a simple step up from core detection techniques to leverage the added benefits of mass spectrometry detection.
- IntelliStart™ System tools facilitate walk-up use and adoption by inexperienced users.
- Well-suited for complex component profiling and detection of components in complex sample matrices.
- Excellent troubleshooting tool enables detection and confirmation of compounds using mass spectral information.
- Supports regulatory compliance offering excellent quantitative analysis capabilities.
- TQD tandem quadrupole system provides ultimate quantitative performance for demanding trace-level analysis in complex matrices.

### Sample preparation products for complex food matrices

- Solid Phase Extraction (SPE) can be used for extraction of target compounds, enrichment, and clean-up. Removal of interferences from complex food matrices can significantly improve method performance and robustness thereby maximizing laboratory productivity.
- Waters® Sep-Pak® products offer a range of conventional SPE chemistries with many literature and validated methods available for food analysis. The highly reproducible adsorbent and packing quality ensures identical performance from every batch of cartridges meeting the critical demands of QC laboratories.
- Waters Oasis® products offer premium performance and the fastest, simplest, and cleanest approach to SPE method development based on patented water-wettable copolymer sorbents with excellent capacity and stability across the pH range.

### Empower 2 Software

- Advanced data collection, processing, and reporting platform for QC laboratory.
- Flexible configuration with scalability for single workstation, small group, or network.
- Supports enhanced workflow through method development and validation software.
- Integrated management of data, advanced security, and audit trails support compliance and regulatory requirements.

## OASIS SAMPLE PREP SOLUTIONS FOR COMPLEX FOOD MATRICES



Waters' Oasis sample extraction products allow simple and rapid preparation methods to be developed.

Patented water-wettable copolymer sorbents maintain proper wetting, have greater capacity, and demonstrate excellent stability over the entire pH range.

The result is solid phase extraction (SPE) that yields superior recovery, reproducibility, retention, and selectivity for a wide variety of compounds.

Waters has developed two complementary SPE procedures for rapid isolation and cleanup of, for example, Sudan dyes in chili products.

For oily matrices such as chili oil, a normal-phase cleanup uses the Waters Sep-Pak Alumina B.

For non-oily matrices such as chili sauce and chili powder, a mixed-mode anion-exchange procedure uses the Waters Oasis MAX sorbent.

## RAW MATERIALS

### Rapid detection of critical impurities

Adulteration of animal feedstuffs with melamine is one of the leading product safety stories of recent years.

Melamine, and other compounds were added to increase the apparent protein content of the feed but ultimately resulted in the poisoning and death of a large number of domestic animals. The net effect was a loss in excess of \$70 Million dollars and several lawsuits.

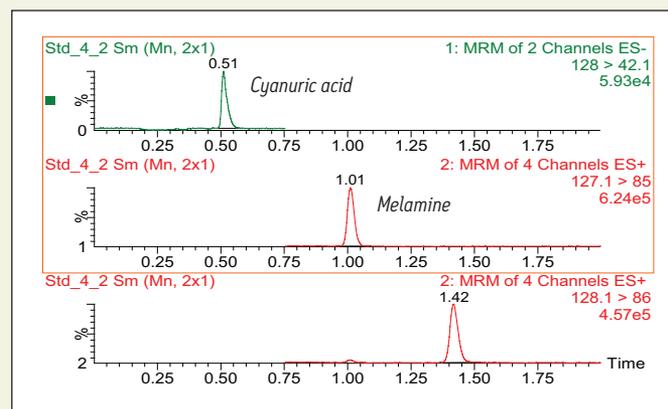


Figure 3. UPLC/MS/MS chromatograms showing rapid, selective detection of critical pet food contaminants.

This example highlights the need for rapid, sensitive, and effective testing strategies. The use of Waters Oasis MCX and MAX SPE cartridges provide efficient sample extraction and clean-up and UPLC/MS/MS analysis with an ACQUITY TQD system provided rapid separation, quantitation, and confirmation of the critical impurities.

### Profiling of raw materials

Variability in raw materials can be monitored using the speed and improved resolution of UPLC. The chromatograms in Figure 4 demonstrate the ability of UPLC to rapidly profile the composition of complex oils.

Differences in composition due to seasonal variation, degradation during storage or processing, or adulteration can easily be identified and action taken to prevent poor quality material entering the process.

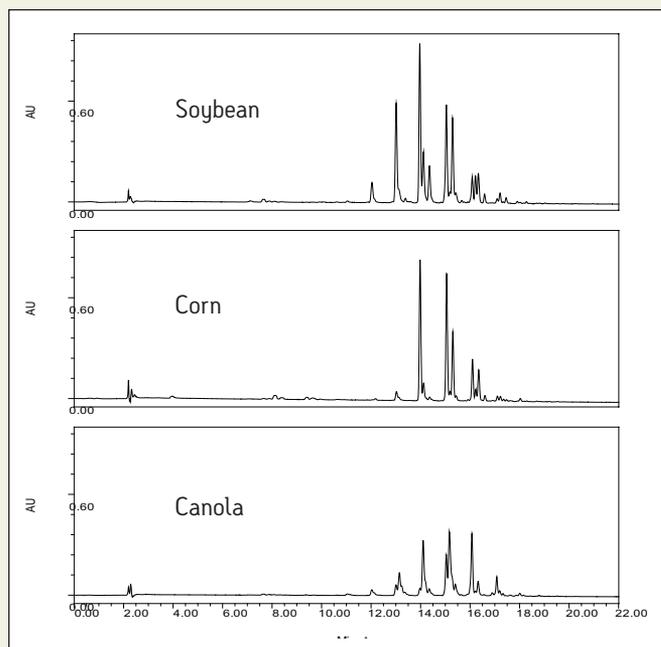


Figure 4. UPLC/UV chromatograms of food oils showing the rapid, high resolution separations achieved by UPLC and ideally suited to profiling and detection of variations and adulteration.

## IN PROCESS / PRODUCTION

### Reaction Monitoring

Monitoring specific chemical composition, such as amino acid levels during fermentation, can provide important information regarding production control, recognizing the need for adjustment of conditions and, ultimately, product quality. For example, Waters UPLC Amino Acid Analysis Solution provides a rapid, reproducible, and accurate way of monitoring amino acid levels of in-process samples. Also, the Waters PATROL UPLC Process Analyzer provides an online testing solution.

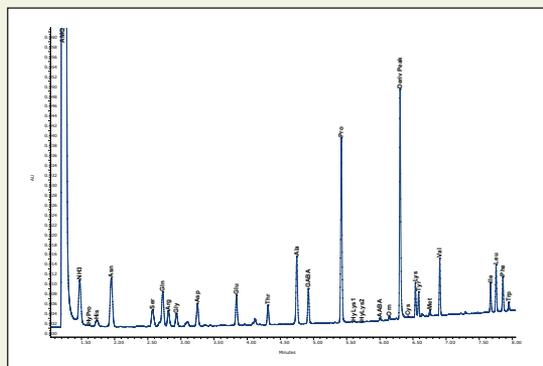


Figure 5. Free amino acid profile of starting malt for brewing process.

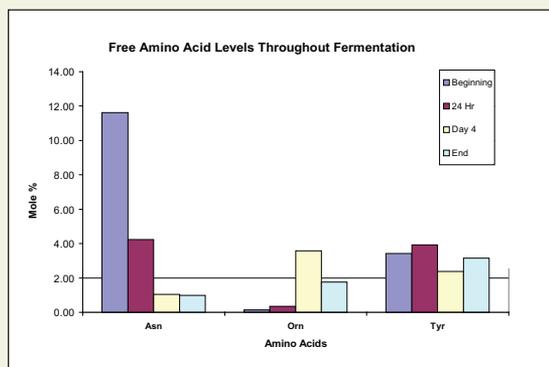


Figure 6. Quantitative changes in amino acid composition monitored during fermentation.

### Use of statistical profiling

In addition to monitoring specific chemical species, processes may be monitored using statistical models to identify non-conforming products or reaction conditions.

The power and speed of UPLC enables complex, information-rich datasets to be collected and tested against models developed using well-established chemometric processes.

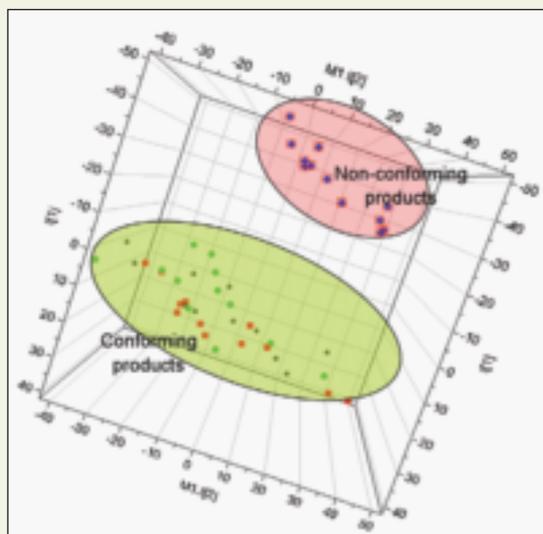


Figure 7. Differentiation of good and bad products based on Principal Component Analysis of a number of UPLC/MS datasets acquired with an ACQUITY SQD System.

## PATROL UPLC PROCESS ANALYZER SYSTEM

**PATROL**<sup>™</sup>  
UPLC® PROCESS ANALYZER

The PATROL UPLC Process Analyzer is a real-time Process Analytical Technology (PAT) System that detects and quantifies complex multiple component manufacturing samples and final product directly on the production floor.

Designed with the same enabling technology that drives the ACQUITY UPLC System, PATROL moves existing liquid chromatography (LC) analysis from off-line Quality Control (QC) laboratories directly to the manufacturing process, resulting in significant improvements in production efficiency.

- Delivers real-time analysis in step with manufacturing processes
- Provides the selectivity, sensitivity, and dynamic range of LC analysis
- UPLC's fast resolving power quickly quantifies related and unrelated compounds
- Reduces process cycle times, so that more product can be produced with existing resources
- Increases production while allowing manufacturers to consistently meet or exceed product specifications, potentially eliminating variability, failed batches, or the need to rework materials
- Assures end product quality, including final release testing

## EMPOWER 2 CHROMATOGRAPHY DATA SOFTWARE

The powerful features of Empower 2 Software make it easy to focus on running samples and producing meaningful results without experiencing downtime for training, re-engineering workflow, or adding new software to support instruments such as mass spectrometry detectors or advanced chromatographic techniques.

### Empower 2:

- Advanced data management
- An intuitive online answer wizard to help maximize laboratory efficiency
- Flexible configuration to support a single workstation, a small group, or a network for the entire lab
- 21 CFR Part 11 compliance capabilities to manage data integrity

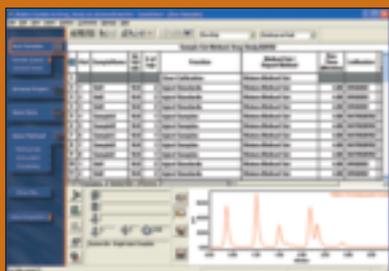


Figure 11. Empower 2 Software's user-friendly interface is designed to maximize productivity and improve data collection, processing, and printing of chromatography data.

## FINAL PRODUCT

### Final product QC testing

In many QC environments, there is a demand for rapid, reliable testing of batches prior to release. UPLC brings substantial benefits to QC laboratories as illustrated in Figure 8. Here the analysis of additives in soft drinks is shown with a cycle time of 1.3 minutes compared to a standard HPLC method taking 17 minutes.

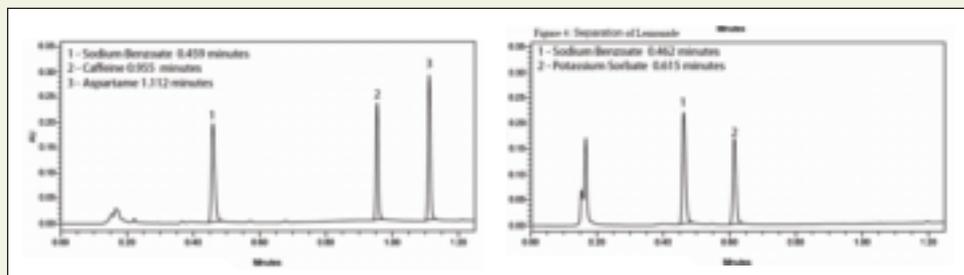


Figure 8. Rapid separation of soft drink additives in (a) diet cola and (b) lemonade.

### Analysis of 'functional' ingredients

It is important to ensure the final product contains the expected levels of 'functional' ingredient. As with Anthocyanins, plant pigments associated with rich antioxidant properties. Over 600 such compounds occur naturally and for analysis they are converted to a group of six anthocyanidins by acid hydrolysis. The UPLC separation below shows baseline resolution of key anthocyanidins in under two minutes compared to over 20 minutes for a typical HPLC run.

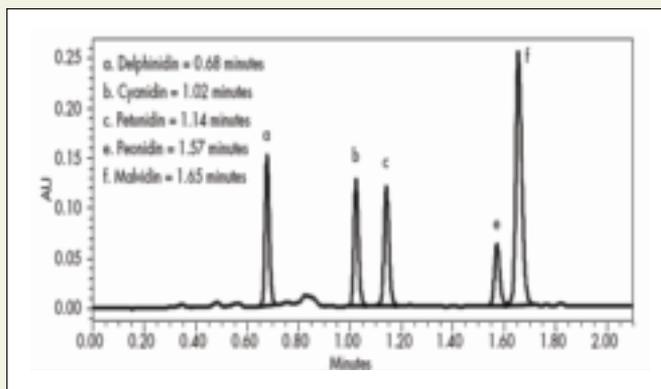


Figure 9. Analysis of anthocyanidins in wild low bush blueberries.

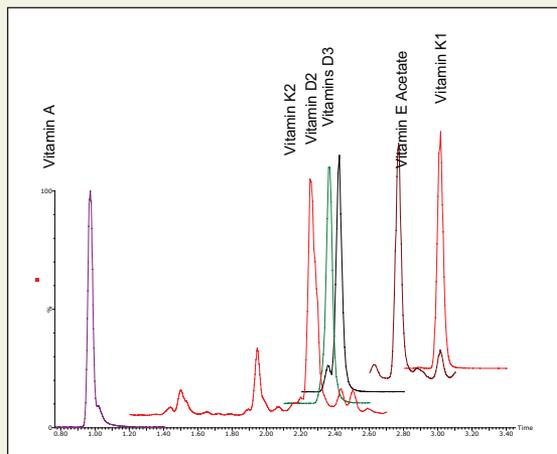


Figure 10. Rapid separation of fat soluble vitamins using UPLC/MS.

### Labeling requirements

Accurate statement of product composition and levels of active ingredients is a key requirement for QC laboratories. The use of ACQUITY UPLC SQD combines the power of rapid, high resolution UPLC separations with the sensitive and selective capability of mass spectrometry for component quantitation and confident confirmation of identity.



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