

Fast LC Analysis of Soft Drinks

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Introduction

Some commercial sub-2µm columns have been introduced to reduce analytical time and increase sample throughput. The smaller particle columns produce higher pressures while demonstrating high separation efficiency, but require specialized systems that can operate under high pressure.

Recently, a 2.2 µm column was introduced that allows performance similar to sub-2µm particles, but at much lower pressures. This column, combined with a conventional HPLC, easily allows high-throughput analysis.

There has been a growing demand for alternatives to high-sugar-content soft drinks, based on several links to obesity. Sugars stimulate the production of insulin, which triggers the body to store fat. "Diet" soft drinks are sweetened with chemicals, such as Acesulfame potassium and Aspartame, which are perceived as sweet by most people, yet do not stimulate the production of insulin or have any other nutritional value. The need to analyze increasing numbers of available beverages suggests the use of Ultra-Fast LC as an appropriate technology for this industry.

Approach to Ultra-Fast LC

- Key: Increase of throughput and the amount of samples
 - Shortening run time while maintaining column resolution efficiency

- Shorter column length
- Higher linear velocity of mobile phase
- These can't sufficiently maintain column efficiency

- Approach to High Speed and High Resolution:

- Smaller particle size
 - Smaller particle size enables use of a shorter column for high-resolution analysis.
 - Separation efficiency is maintained at a high flow rate.
- High temperature
 - Separation efficiency is maintained at a high flow rate.

Smaller Particle Size Column 1

- Sub-2µm particle sizes easily provide shorter run times compared to ordinary 5µm particle sizes

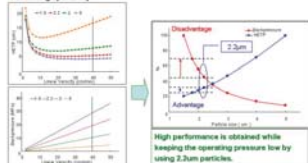
Disadvantages of sub-2µm particles

- Column pressure increases in inverse proportion to square of particle size
- High pressure prevents use of longer columns for additional resolution
- Specialized hardware and connections are required for high pressures
- Higher pressures reduce consumable parts life, and lead to poor reproducibility of retention time

Optimization of particle size

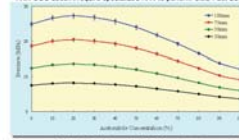
Smaller Particle Size Column 2

- What are the considerations for obtaining high speed and high resolution while maintaining operability?

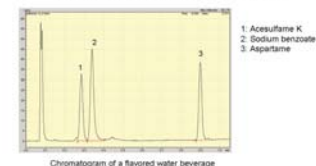


Shim-pack XR-ODS

- Operation under less than 30MPa
- XR-ODS doesn't require specialized HW to perform Ultra-Fast LC.

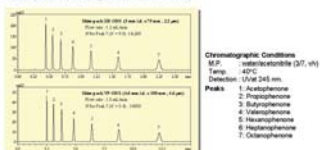


Chromatogram of Beverage Sample



Shim-pack XR-ODS

- Shim-pack XR-ODS (particle size: 2.2µm)



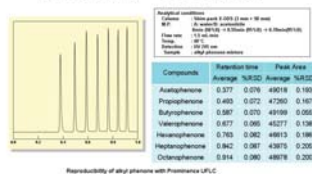
Method Conditions for Soft Drinks

- Column: Shim-pack XR-ODS (3.0mm ID, x 30 mm, 2.2µm)
- Mobile Phase: A=22 mM KH₂PO₄/3 mM K₂HPO₄ (pH 6.0), B=ACN
- Gradient: 2-25% B over 1.0 min
- Flow Rate: 2.25 mL/min
- Column Temp: 40°C
- Detection: 214 nm
- Injection Vol: 1 µL

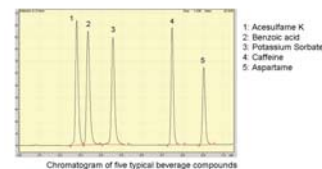
Prominence UFLC

- Higher speed and uncompromised separation
 - Outstanding speed and separation even at conventional pressure levels by combination with Shim-pack XR-ODS column
 - Analyzing at even higher speeds
 - High-speed injection (about 10 seconds)
 - Shortening the overall analysis time with automated features such as automatic purging
 - Maximizing data reliability
 - Excellent reproducibility of retention time and peak area
 - Autosampler 6-port valve achieves 0.3% or less area reproducibility in 100,000 cycle endurance test
 - Reduced carryover by inhibiting the adsorption of samples
 - Making ultra-fast analysis more accessible
- Prominence UFLC can also be used for conventional HPLC or column switching analysis

Reproducibility – UFLC Gradient



Chromatogram of Beverage Standards



Conclusions

The five beverage components were separated within a one-minute analysis time. UFLC can be a valuable tool for increasing sample throughput for high-volume applications such as the beverage industry; however, the entire cycle time of the analysis must be considered. As analysis times become shorter, column equilibration times and autosampler injection speeds become of increasing importance in determining cycle times that can be achieved. Shimadzu's high-speed autosampler features a 10-second injection cycle time that allows the full benefits of UFLC to be realized. The use of 2.2 µm particles with a narrow distribution range allows ultra-fast separations to be performed with a conventional HPLC system.