

Application News

Liquid Chromatography Mass Spectrometry

No.C66

Ultra Fast Analysis of Combination Cold Remedy Using LCMS-2020

Now that ultra fast LC can provide analyses with peak widths in fractions of a second, it is necessary to have detectors that can keep pace with the sharp peaks generated by this technique. The LCMS-2020 is a mass spectrometer with "UFswitching" that allows high speed positive / negative ionization mode switching (15 msec positive / negative ionization switching), and

"UFscanning" that allows high-speed scan measurement (max. 15,000 u/sec scan speed) to fully support the progression to LC ultra fast analysis.

Here we present an example of ultra fast analysis of a combination pharmaceutical cold remedy using the Prominence UFLCxR ultra fast, high-resolution LC system and the LCMS-2020.

■ Analysis of Combination Cold Remedy

Sample preparation consists of crushing a tablet of combination cold remedy A and dissolving 10 mg of the sample in 10 mL of purified water. The solution was filtered and then analyzed by LC/MS. An electrospray source (ESI) was used for ionization, and simultaneous positive / negative ion analysis was used to maximize detection of different compounds.

The total ion chromatogram (TIC) and mass chromatograms of combination cold remedy A are shown in Fig. 1, with the structural formulas of the 7 confirmed ingredients shown in Fig. 2. Using a mobile phase flow rate of 1.8 mL/min, analysis was performed in just 1.5 minutes, including the time required for column equilibration. The entire mobile phase flow was directed into the MS without splitting.

Fig. 3 shows the mass spectra of all ingredients. A protonated molecule was found for every ingredient in positive ion mode. In addition, a deprotonated molecule was also confirmed for 2. acetaminophen in the negative ion mode.

Table 1 Analytical Conditions

Column	: Advanced Materials Technology HALO C18 (3.0 mm I.D. × 50 mm L, 2.7 μm)
Mobile Phase A	: 5 mM Ammonium formate and 5 mM Formic acid - water
Mobile Phase B	: Acetonitrile
Time Program	: 7 %B (0 min) → 45 %B (1 min) → 7 %B (1.01-1.5 min)
Flow Rate	: 1.8 mL/min
Injection Volume	: 1 μL
Column Temp.	: 60 °C
Probe Voltage	: 4.5 kV /-3.5 kV (ESI-Positive mode/Negative mode)
DL Temperature	: 250 °C
BH Temperature	: 200 °C
Nebulizing Gas Flow	: 1.5 L/min
Drying Gas Flow	: 20 L/min
DL,Q-array Voltage	: Default values
Scan Range	: m/z 100 - 700 (60 msec/Scan)

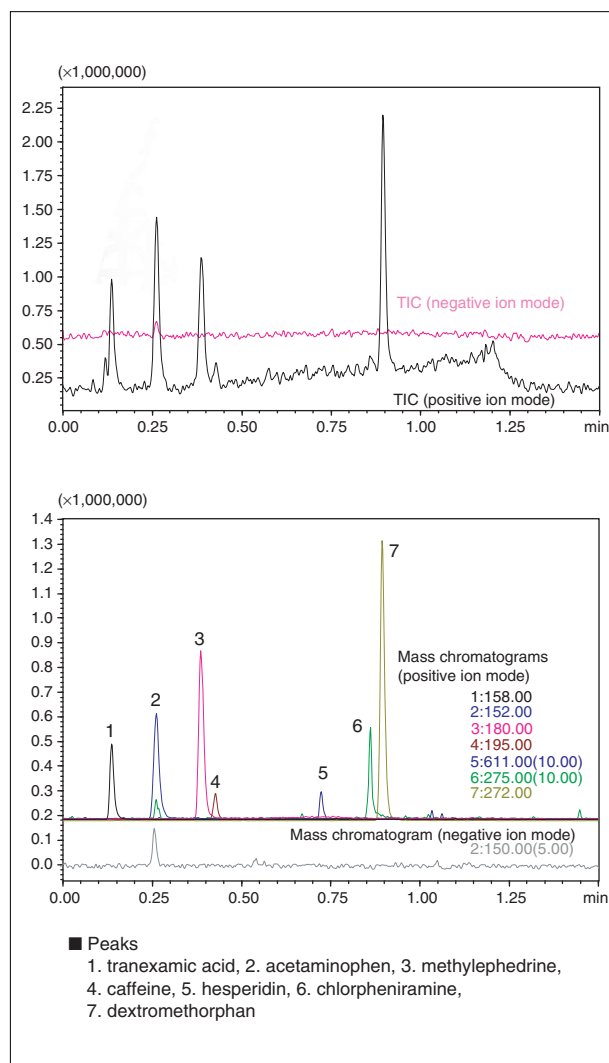


Fig. 1 TIC (upper) and Mass Chromatograms (lower) of Combination Cold Remedy A

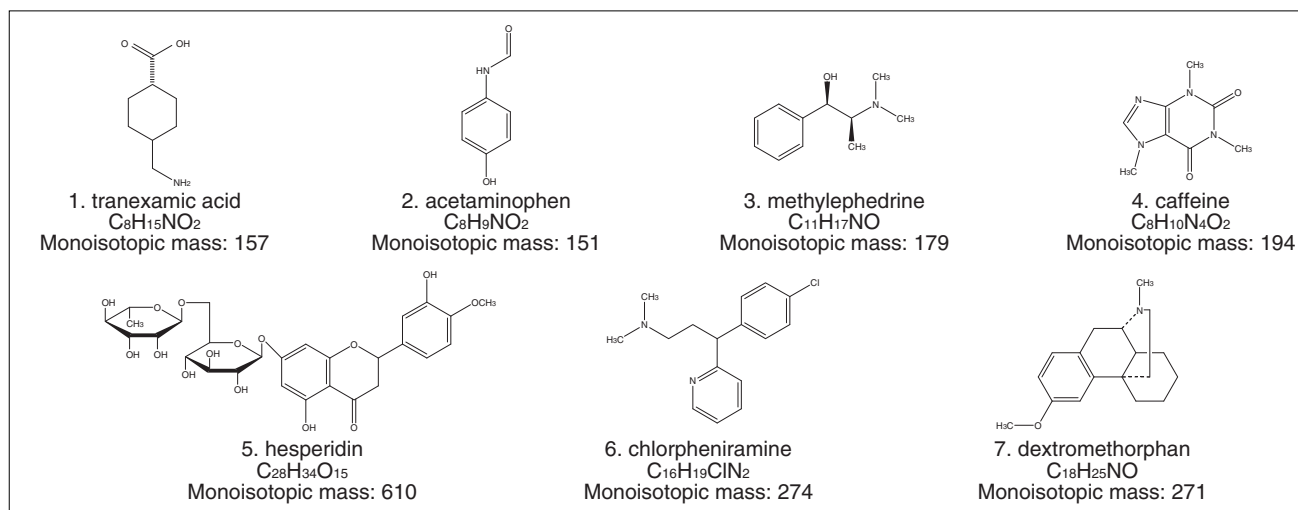


Fig. 2 Structural Formulas of Ingredients in Combination Cold Remedy A

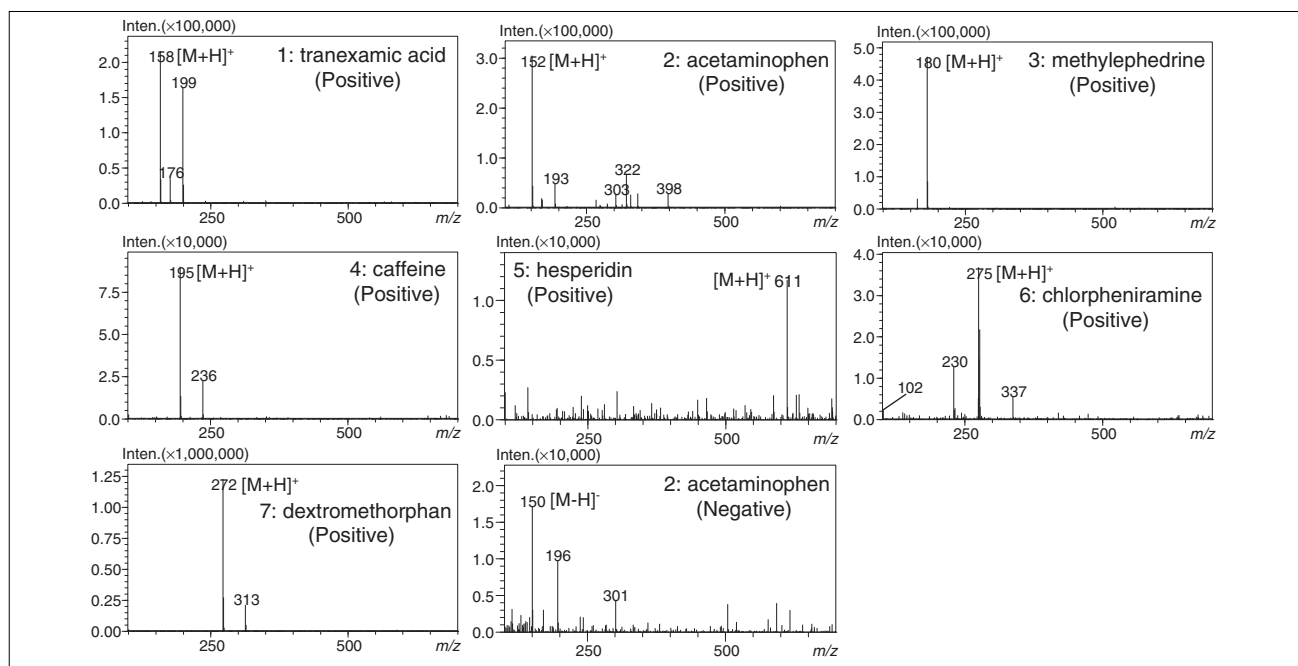


Fig. 3 Mass Spectra of Ingredients in Combination Cold Remedy A

■ Ultra Fast Mass Analysis

In this analysis, measurement was conducted with a scan speed of 15,000 u/sec. With the mass range of m/z 100 - 700 for the acquisition, each scan required just 60 msec (Fig. 4). This fast sampling time assures that a sufficient number of scans are acquired for the sharp peaks generated using the Prominence UFLC_{XR}.

The LCMS-2020 clearly supports ultra fast analysis together with the Prominence UFLC_{XR} ultra fast, high-resolution LC system. This combination provides excellent, reproducible results for 1.5-min fast analysis of combination cold remedy. Even for the fastest

peaks, spectral quality is maintained and reliable for correct confirmation of each constituent.

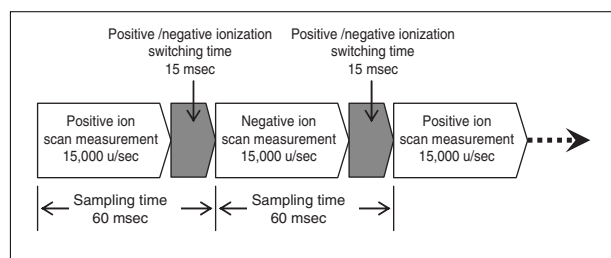


Fig. 4 Sampling Time and Positive / Negative Ionization Switching Time

NOTES:

*This Application News has been produced and edited using information that was available when the data was acquired for each article. This Application News is subject to revision without prior notice.



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