

Application News

Analytical Strategy for Food Safety Control Using i-Series HPLC—High-Speed Determination of Additives Associated with Ultra-Processed Food Consumption—

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User Benefits

- ◆ Preservatives, acidulants, and other compounds with differing polarities can be quickly analyzed using a general-purpose HPLC system, even without using a dedicated instrument.
- ◆ The photodiode array (PDA) detector built into the integrated HPLC system enables acquisition of chromatograms at any desired wavelength from a single analysis.

Introduction

Foods can contain various ingredients, including preservatives that inhibit microbial growth. In January 2026, the U.S. federal government recommended limiting intake of ultra-processed foods (UPF), identifying 11 target compounds including propylparaben¹⁾. Around the same time in Japan, there was a reported recall of confections containing methylparaben, which is not approved domestically. Given the circumstances, more rapid and effective quality-control systems are required to ensure product safety.

Organic acids, including those employed as acidulants, are typically analyzed using dedicated systems based on ion-exclusion chromatography. However, the application of reversed-phase chromatography enables the simultaneous determination of preservatives and other additives within a single analytical run. This approach enhances analytical throughput and improves workflow efficiency for compounds with diverse physicochemical properties.

In this study, an integrated HPLC system (Fig. 1) is utilized for the rapid and simultaneous analysis of food additives, including preservatives, acidulants, and a bittering agent.



Fig. 1 Integrated LC System "i-Series" (LC-2080C 3D)

Analysis of Mixed Standard Solution

Twelve food-additive compounds were targeted for analysis: methylparaben, ethylparaben, isopropylparaben, propylparaben, isobutylparaben, butylparaben, benzoic acid, sorbic acid, dehydroacetic acid, citric acid, propionic acid, and caffeine. Table 1 shows the analytical conditions and Fig. 2 shows the chromatograms from analyzing the mixed standard solution (with citric acid and propionic acid at 100 mg/L each and the other ten compounds at 1 mg/L each).

The PDA detector wavelength was set near the maximum absorption wavelength for each target compound. Using a simultaneous analysis method, all 12 target compounds were eluted within 10 minutes.

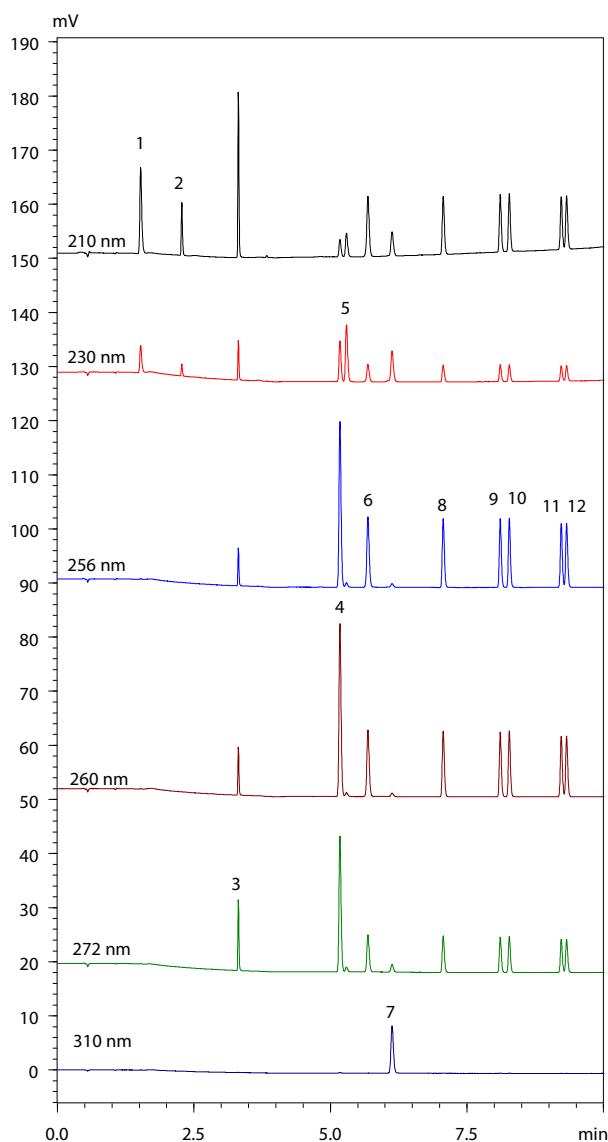


Fig. 2 Chromatograms for Mixed Standard Solution
1. Citric acid, 2. Propionic acid, 3. Caffeine, 4. Sorbic acid, 5. Benzoic acid, 6. Methylparaben, 7. Dehydroacetic acid, 8. Ethylparaben, 9. Isopropylparaben, 10. Propylparaben, 11. Isobutylparaben, 12. Butylparaben

Table 1 Analytical Conditions

System:	i-Series LC-2080C 3D
Column:	Shim-pack Scepter C18-120 ¹ (100 mm × 3.0 mm I.D., 1.9 μm)
Flowrate:	0.8 mL/min
Mobile Phase:	A) 20 mmol/L (sodium) phosphate buffer (pH 2.4) B) Acetonitrile
Time Program:	2 % B (0 min) → 25 % B (2.50 min) → 32 % B (4.50 min) → 60 % B (9.00 - 10.00 min) → 2 % B (10.01 - 15.00 min)
Column Temp.:	30 °C
Injection Volume:	5 μL
Vial:	Shim-vial H glass ²
Detection:	Ch 1: 210 nm, Ch 2: 230 nm, Ch 3: 256 nm, Ch 4: 260 nm, Ch 5: 272 nm, Ch 6: 310 nm

*1 P/N: 227-31013-03, *2 P/N: 227-34500-01

■ Repeatability

Table 2 shows the repeatability (%RSD) of retention time and peak area values in six repeated analyses of the mixed standard solution (with citric acid and propionic acid at 100 mg/L each and the other ten compounds at 1 mg/L each). Repeatability of both retention time and peak area values was satisfactory for all compounds.

Table 2 Repeatability (%RSD) in Six Repeated Analyses

Compound	Retention time	Peak area
Citric acid	0.08	0.08
Propionic acid	0.04	0.34
Caffeine	0.03	0.15
Sorbic acid	0.03	0.08
Benzoic acid	0.02	0.22
Methylparaben	0.02	0.08
Dehydroacetic acid	0.02	0.10
Ethylparaben	0.02	0.11
Isopropylparaben	0.03	0.06
Propylparaben	0.03	0.13
Isobutylparaben	0.03	0.31
Butylparaben	0.03	0.14

■ Calibration Curves

The calibration curves for the 12 target compounds were highly linear, with coefficients of determination (r^2) of 0.9999 or greater. Fig. 3 shows the calibration curves of citric acid and sorbic acid. Table 3 shows the concentration ranges of the calibration curves and coefficients of determination for the target compounds.

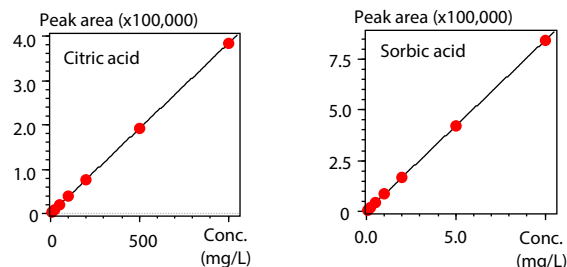


Fig. 3 Calibration Curves (Citric Acid and Sorbic Acid)

Table 3 Concentration Ranges of Calibration Curves and Coefficients of Determination (r^2)

Compound	Conc. Range (mg/L)	r^2
Citric acid	10 to 1000	0.9999
Propionic acid	10 to 1000	0.9999
Caffeine	0.1 to 10	0.9999
Sorbic acid	0.1 to 10	0.9999
Benzoic acid	0.1 to 10	0.9999
Methylparaben	0.1 to 10	0.9999
Dehydroacetic acid	0.1 to 10	0.9999
Ethylparaben	0.1 to 10	0.9999
Isopropylparaben	0.1 to 10	0.9999
Propylparaben	0.1 to 10	0.9999
Isobutylparaben	0.1 to 10	0.9999
Butylparaben	0.1 to 10	0.9999

■ Analysis of Energy Drink

Chromatograms of a commercial energy drink are shown in Fig. 4. The sample was diluted 100-fold with ultrapure water and filtered through a 0.2 μm membrane filter before HPLC analysis.

Citric acid, caffeine, benzoic acid and ethylparaben were detected from the energy drink. Table 4 (1) shows the concentrations of each compound in the energy drink. Note that the concentrations shown in Table 4 are average concentrations obtained from six repeated analyses of samples after pretreatment.

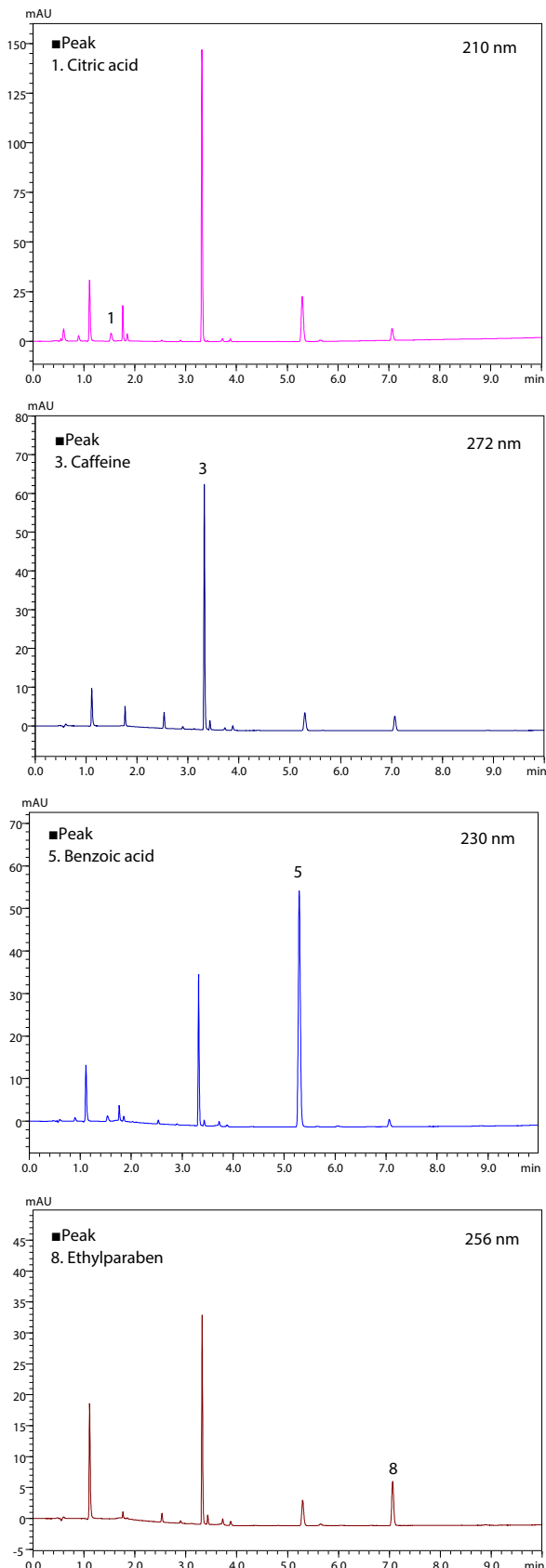


Fig. 4 Chromatograms of Energy Drink

■ Analysis of Wine

A chromatogram of a commercial wine is shown in Fig. 5. The sample was diluted 100-fold with ultrapure water and filtered through a 0.2 μm membrane filter before HPLC analysis.

Sorbic acid was detected from the wine. Table 4 (2) shows the concentrations of each compound in the wine. Note that the concentrations shown in Table 4 are average concentrations obtained from six repeated analyses of samples after pretreatment.

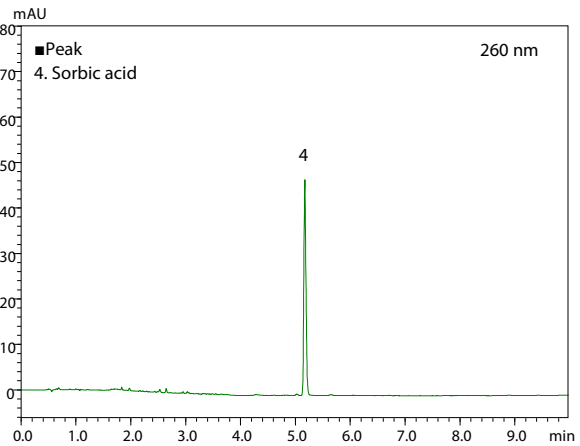


Fig. 5 Chromatogram of wine

Table 4 Analysis Results (n = 6)

Compound	(1) Energy drink		(2) Wine	
	Conc. (mg/L)	%RSD	Conc. (mg/L)	%RSD
Citric acid	25.5	0.61	N.D.	
Caffeine	4.89	0.17	N.D.	
Sorbic acid	N.D.		1.50	0.07
Benzoic acid	5.31	0.11	N.D.	
Ethylparaben	0.56	0.18	N.D.	

■ Conclusion

A rapid reversed-phase chromatographic method for the analysis of multiple food additives was developed using the i-Series LC-2080C 3D. While organic acids such as citric acid are conventionally determined using dedicated ion-exclusion chromatography systems, the proposed approach enables their simultaneous analysis together with preservatives and a bittering agent within a single run. By consolidating the analysis of compounds with diverse physicochemical properties, the i-Series system significantly enhances analytical throughput and minimizes overall analysis time, thereby providing an efficient and streamlined solution for high-speed quality control workflows.

References

- 1) Dietary Guidelines for Americans, 2025–2030 (<https://cdn.realfood.gov/DGA.pdf>)

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