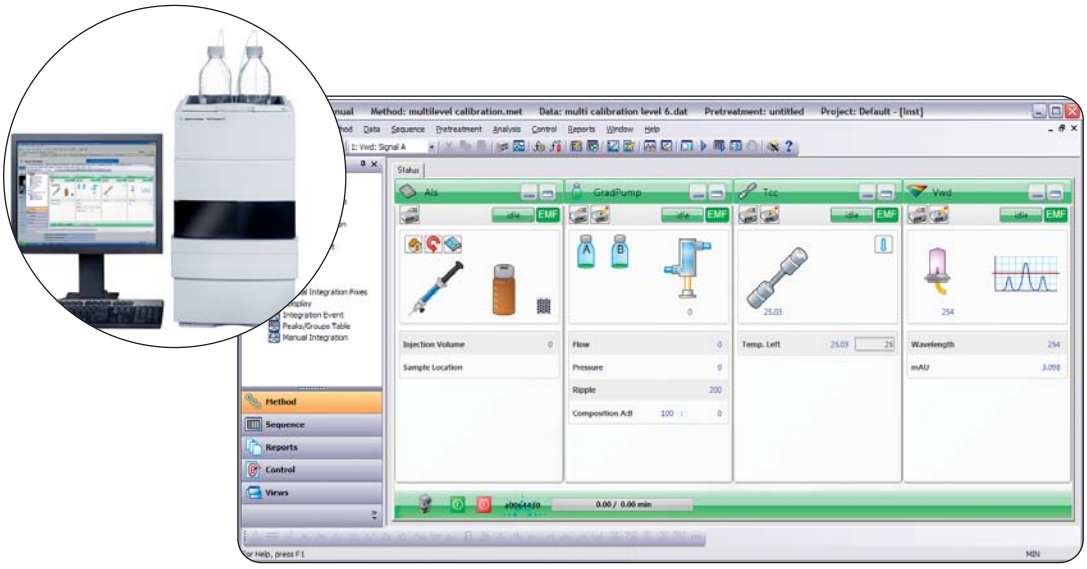
Analysis of preservatives in food and cosmetics with the Agilent 1120 Compact LC system

Application Note

Rongjie Fu,
Zhixiu Xu



Abstract

An HPLC method was developed for simultaneous determination of the nine preservatives most often used in food and cosmetics. The system suitability results showed that the Agilent 1120 Compact LC is the system of choice for conventional, analytical scale liquid chromatography. This integrated HPLC system was designed for ease of use, performance, and reliability. The quantitative analysis of typical samples is demonstrated in this Application Note.

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Introduction

Preservatives are very popular in the food and cosmetics industries because they prevent these products from degrading within the warranty time. But preservatives are strictly regulated because their overuse can cause some health problems in humans. For example, some preservatives can accumulate in the human body and negatively influence the metabolism process. Today's trends in food and cosmetics increasingly emphasize the concepts of healthy and green. That means use of safer raw materials, as well as fewer preservatives and control of preservatives within a safe limit.

In developing countries like China, the regulation of preservatives in food and cosmetics is approaching the international standards, such as the commonly used regulations adopted by the Codex Alimentarius Commission (CAC). In general, the regulations set the concentration limits on the preservatives in cosmetics and food. With increased research on safety of food and cosmetics, it might be necessary to analyze more preservatives in the future.

Some popular preservatives were analyzed in this application. A face conditioner and glace fruit were selected as typical samples that contain certain kinds of preservatives. The manufacturers of these products need to control the quality of their products before they go to market. The regulatory agencies check these products in the market very carefully to see if the amount of preservative is within the limit.

The experiments in this Application Note were performed with the Agilent 1120 Compact LC, which is the system of choice for conventional, analytical scale liquid chromatography. It is an integrated HPLC system designed for ease of use, performance, and reliability. It is ideally suited for routine analyses in the food and fine chemical industries because of its capability to achieve very precise retention times and peak areas, as well as low detection limits for the analyzed compounds.

Experimental

Equipment

- Agilent 1120 Compact LC system with gradient pump (degasser inside), autosampler, column compartment, and variable wavelength detector (VWD)
- EZChrom Elite Compact software

Chemicals and reagents

- Reference standards were purchased from Sinopharm Chemical Reagent Co. Ltd., Shanghai, China.
- Water was obtained from a Millipore water purifier.
- Acetonitrile (Fisher Scientific) was HPLC purity. All other reagents were analytical purity.

Sample preparation

Glace fruit: The fruit was cut into pieces and 1.5 g was weighed and diluted to 10 mL with water. The mixture was treated with an ultrasonic for 15 minutes, and was filtered with a 0.45 µm filter prior to injection.

Face conditioner: 1 mL was diluted to 10 mL with water, and the sample was filtered with a 0.45 µm filter prior to injection.

Chromatographic conditions

- Column: Agilent HC-C18(2), 4.6 x 250 mm, 5 µm
- Mobile phase: A = 20 mM acetate buffer, pH 4.2; B = acetonitrile
- Gradient: 0 – 25 min, 30 %B – 45 %B
- Flow rate: 1 mL/min
- Wavelength:

0 min	260 nm
5 min	230 nm
5.6 min	260 nm
9.2 min	230 nm
10.2 min	260 nm

- Injection volume: 5 µL
- Temperature: 30 °C

Results and discussion

Development of a method for these preservatives in food and cosmetics must consider the run time and the separation of the nine compounds. The other fact that must be considered is that the matrix of the real samples may influence the separation. The HPLC system needs a column with good efficiency, and for quantitative analysis of the preservatives, it must deliver good precision for retention times and peak areas.

Because the compounds used as preservatives have different ultraviolet (UV) spectra, the wavelength program of the variable wavelength detector was used in this study to detect all the compounds at their most sensitive wavelength.
The overlaid chromatograms of

two real samples and the preservative standards are shown in figure 1. The corresponding peak names are listed in table 1. In glace fruit, the benzoic acid and sorbic acid were found. In the face conditioner sample, only methylparaben was found.

The system reproducibility was tested with the nine compounds (table 1). The high precision of the retention times gives high confidence when comparing the standards and real samples.

The linear range of the standards was tested with this Agilent 1120 Compact LC system. The results are listed in table 2. The data shows that very good regression factors (values of r^2) were achieved for each compound.

The quantitative results from the two samples are shown in table 3.

Conclusion

The Agilent 1120 Compact LC is ideal for the routine analysis of preservatives in food and cosmetics. Excellent resolution and good separation were achieved, and system suitability experiments showed the robustness and high precision for this kind of application. The high precision of the retention times and peak areas ensures reliable results when quantitation is needed for quality control. The variable wavelength detector can be used with programmed wavelength to adjust to the maximum absorbance for all the compounds.

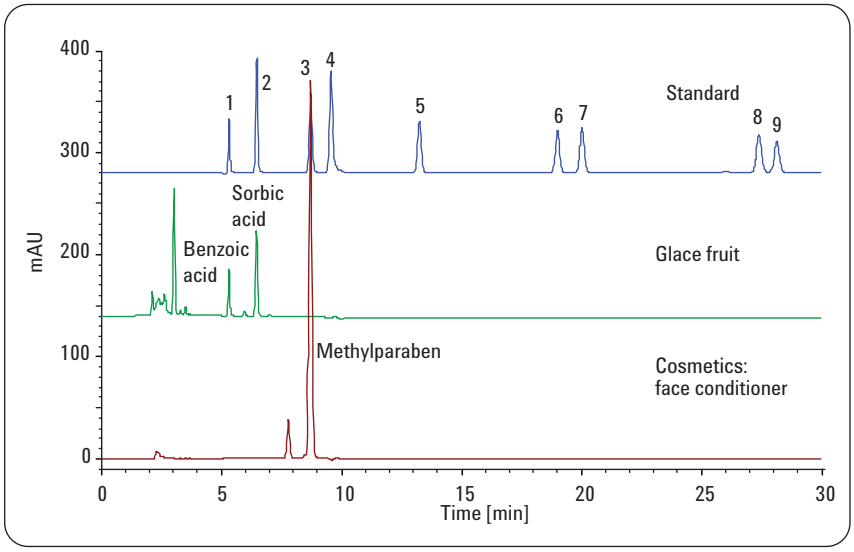

Figure 1
Overlaid chromatograms of preservative standards and real samples.

Peak	Compound	% RSD retention times	% RSD areas
1	Benzoic acid	0.03	1.11
2	Sorbic acid	0.03	0.16
3	Methylparaben	0.03	0.11
4	Dehydroacetic acid (DHA)	0.04	1.05
5	Ethylparaben	0.03	0.10
6	Isopropylparaben	0.03	0.11
7	n-Propylparaben	0.02	0.07
8	Isobutylparaben	0.02	0.10
9	n-Butylparaben	0.02	0.11

Table 1
Reproducibility of six injections of nine preservative standards.

Peak	Compound	Range (ng)	r^2
1	Benzoic acid	45.5 - 455	0.9998
2	Sorbic acid	35 - 350	0.9998
3	Methylparaben	66.5 - 665	0.9998
4	Dehydroacetic acid (DHA)	135 - 1350	0.9991
5	Ethylparaben	64.5 - 645	0.9998
6	Isopropylparaben	67 - 670	0.9998
7	n-Propylparaben	71.5 - 715	0.9998
8	Isobutylparaben	76.5 - 765	0.9998
9	n-Butylparaben	63.5 - 635	0.9998

Table 2
Linearity of the nine preservative standards.

	Benzoic acid	Sorbic acid	Methylparaben
Glace fruit	101.63 mg/Kg	70.59 mg/kg	ND*
Face conditioner	ND	ND	1.07 mg/mL

*ND = not detected

Table 3
The amount of preservatives in the real samples.

References

1.
X. Ping, “Analysis of preservatives in beverage using Agilent TC C18 column”, *Agilent Technologies Application Note, publication number 5989-3638CHCN*, **2005.**

2.
B. Huang, “Analysis of 8 preservatives and 3 sweeteners in sauce and beverage by HPLC”, *Chinese Journal of Health Laboratory Technology, 15(10), 1208*, **2005.**

Rongjie Fu and Zhixiu Xu are Application Chemists at Agilent Technologies in Shanghai, China.

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