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Application News

No. L540

High Performance Liquid Chromatography

# High-Resolution and High-Speed Simultaneous Analysis of Preservatives in Cosmetics Using SPP Column

Cosmetics usually have a high water and oil content. For this reason, both bacteria and molds can proliferate in cosmetics stored at ambient temperatures, resulting in a change in the physical properties and safety of the product. The Pharmaceutical Affairs Law stipulates that cosmetics must have consistent properties and quality over more than three years, and those that may deteriorate within three years must display an "expiration date". To increase stability, many cosmetics contain antiseptic compounds such as parabens and 2-phenoxyethanol. However, these preservatives are not only bactericidal but can also cause allergic symptoms in humans, mainly skin conditions such as eczema and dermatitis. Strict regulations are therefore imposed in Japan and Europe on the amount of these ingredients that can be used in cosmetics.

This article introduces a method for the analysis of 24 types of cosmetic preservatives, either specified in the cosmetics standards established by the Japanese Ministry of Health, Labour and Welfare or regulated by the European Commission, using ultra-high performance liquid chromatography (UHPLC). Analyses were performed using the Nexera™ series UHPLC system equipped with a Shim-pack Velox™ C18 SPP (superficially porous particles, core-shell) column, which enabled both high resolution and reduced assay time.

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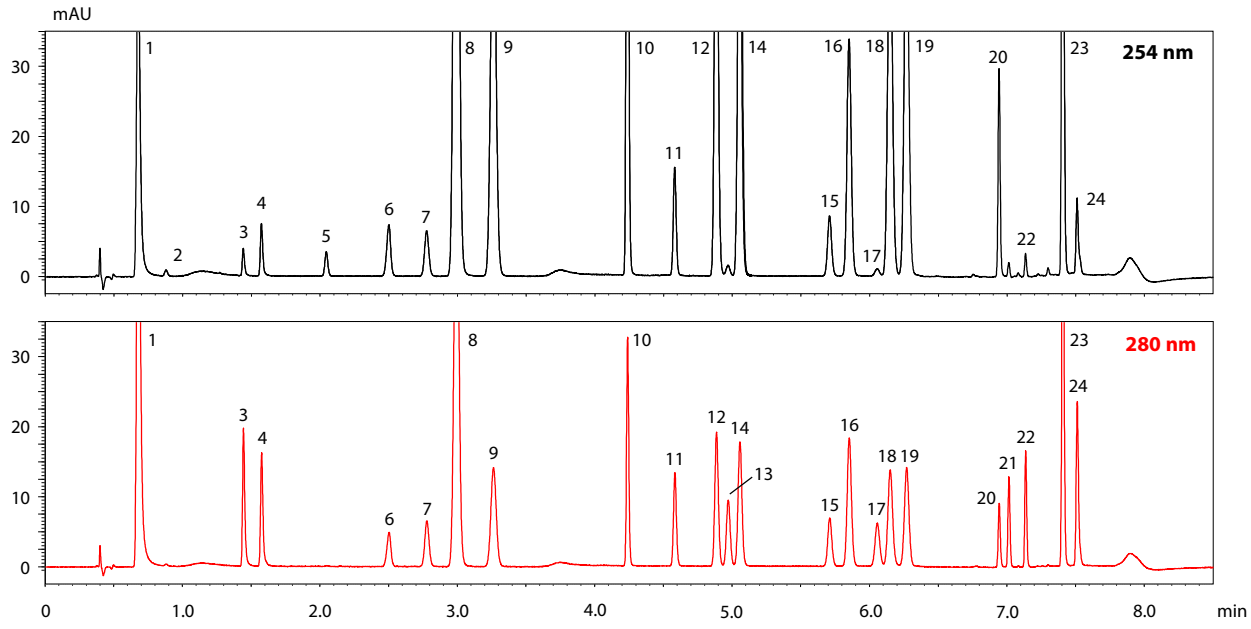
## Analysis of Standard Solutions

We used the Shim-pack Velox C18 SPP column to analyze 24 types of compounds regulated by the Pharmaceutical Affairs Law of the Ministry of Health, Labour and Welfare and the European Commission.

All standard solutions were prepared at concentration 50 mg/L. Simultaneous chromatographic separation is shown in Fig. 1, and assay conditions are reported in Table 1. Separation and detection of all 24 components were possible in about 9 minutes (at 45 MPa).

**Table 1 HPLC Analytical Conditions**

| | |
|---|---|
| Column | : Shim-pack Velox C18 (100 mm L × 3.0 mm I.D., 2.7 µm) |
| Mobile phase | : A) 25 mmol/L $NaH_2PO_4$ aq. (pH 3.8)<br>B) MeOH/$CH_3CN$=9/1 |
| Time program | : B Conc. 8 % (0 min) → 30 % (0.31 min-3.00 min) → 49 % (3.01 min-4.50 min) → 53 % (5.00 min-5.50 min) → 80 % (6.50 min-7.20 min) → 8 % (7.21 min-9.00 min) |
| Flow rate | : 1.0 mL/min |
| Column temperature | : 45 °C |
| Injection volume | : 1 µL |
| Detector | : SPD-M40 Photo diode array detector |
| Cell | : Semi-micro cell |
| Wavelength | : 190 to 800 nm (Monitor 254 nm and 280 nm) |




Peaks:
1. 2-methyl-4-isothiazolin-3-one, 2. 2-bromo-2-nitro-1,3-propanediol, 3. salicylic acid, 4. isothiazolinones, 5. benzyl alcohol, 6. benzoic acid, 7. 2-phenoxyethanol, 8. sorbic acid, 9. methyl paraben, 10. ethyl paraben, 11. methyl benzoate, 12. isopropyl paraben, 13. 4-chloro-3-methylphenol, 14. propyl paraben, 15. ethyl benzoate, 16. 2-phenylphenol, 17. chloroxylenol, 18. isobutyl 4-hydroxybenzoate, 19. butyl paraben, 20. phenyl benzoate, 21. 2,4-dichloro-3,5-dimethylphenol, 22. clorofene, 23. triclocarban, 24. triclosan

**Fig. 1 Chromatograms of Cosmetic Preservative Standard Solutions (50 mg/L Each)**

## Shim-Pack Velox Column

Shim-pack Velox columns are the first columns equipped with Shimadzu Corporation's core-shell technology, which enables high-speed separation while keeping back pressure low through the use of superficially porous particles (SPP, core-shell). The SPP particles have a central, non-porous cores, and a porous layer on the surface containing the sample components. As a result, the average molecular migration distance within the particle is shortened, and the mass transfer diffusion (as a distribution of the migration distance of each molecule) is smaller than for fully porous particles. This structure gives sharp peak shapes compared to full-porous particles of the same particle size, resulting in a higher number of theoretical plates.

Fig. 2 shows a diagram of the stationary phase of the Shim-pack Velox C18 used in this study. This column is suitable for analysis in a wide range of fields, including pharmaceuticals, foods, and environmental science, due to its high end-capping rate and high hydrophobic retention.

**Fig. 2 Representation of the Shim-pack Velox C18 Stationary Phase**

## Analysis of Commercially Available Lotions

Chromatograms of lotions A, B, and C are shown in Fig. 3, 4, and 5, respectively. Approximately 0.1 g of each sample was diluted with methanol in a 50 mL volumetric flask, filtered through a membrane filter, and then analyzed (1 µL injection).

Analytical conditions are shown in Table 1.

2-Phenoxyethanol, methylparaben and ethylparaben were detected in all samples analyzed (A, B and C). Propylparaben was also detected in lotion C. The amounts of each component present are shown in Table 2. All the compounds were below the maximum allowable content of 1000 mg/100 g specified in the Cosmetic Standards based on the Pharmaceutical Affairs Law.

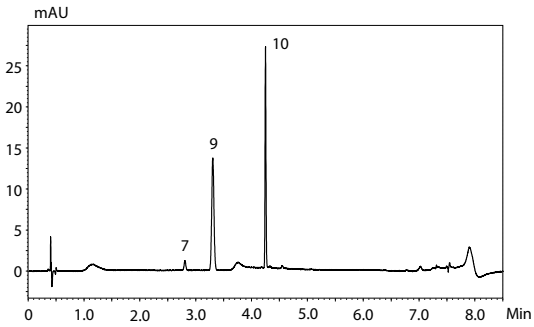



**Fig. 3 Chromatogram of Lotion A**

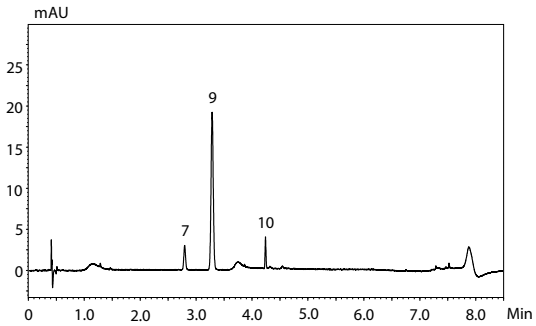



**Fig. 4 Chromatogram of Lotion B**

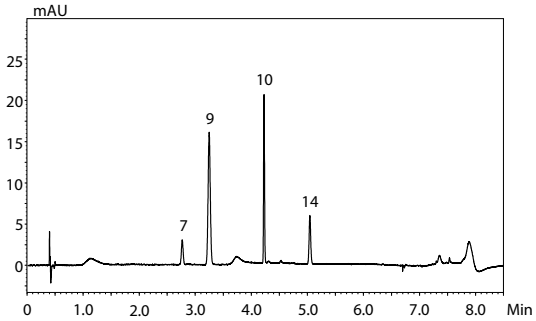



**Fig. 5 Chromatogram of Lotion C**

**Table 2 Preservative Content Results for All Lotion Samples**

| Preservative | Amount present/ mg/100 g | | |
|---|---|---|---|
| | A | B | C |
| Phenoxyethanol | 99.0 | 224.5 | 261.6 |
| Methylparaben | 112.9 | 152.6 | 142.1 |
| Ethylparaben | 97.3 | 11.2 | 79.2 |
| Propylparaben | - | - | 41.5 |



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Nexera series
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