SHIMADZU
Excellence in Science

Application News

No.L473

High Performance Liquid Chromatography

Analysis of Quasi-Drug by Nexera-i (Part 1) High-Speed, High-Resolution Analysis of Active Ingredient and Preservatives

Quasi-drugs (cosmetics, etc.), in many cases, consist of an active ingredient together with various components such as preservatives. Typically, a UV detector or photodiode array detector (PDA) is used to conduct qualitative and quantitative analysis of these substances. In particular, a PDA permits qualitative analysis of a UV absorption spectrum and quantitative analysis based on a chromatogram.
Following are examples of analysis of the active ingredient in a quasi-drug (cosmetic), in addition to analysis of the coexisting preservatives, using the new Nexera-i integrated ultra-high-performance liquid chromatograph.

Table 1 Analytical Conditions

Column	:Shim-pack XR-ODS Ⅱ (75 mm L × 3.0 mm I.D., 2.2 µm)
Mobile Phase	:A) 1 % Acetic Acid in Water B) 1 % Acetic Acid in Acetonitrile
Time Program	:B. Conc. 25 % (0 min) → 50 % (3.0 - 3.5 min) → 25 % (3.51 - 6 min)
Flowrate	:1.2 mL/min
Column Temp.	:40 °C
Injection Volume	:1 µL
Detection	:LC-2040C 3D at 260 nm
Flow Cell	:High-Speed High-Sensitivity Cell

Analysis of Standard of Active Ingredient and Preservatives

Glycyrrhizic acid, a commonly used active ingredient in quasi-drugs (cosmetics) added to provide an anti-inflammatory effect, in addition to ten preservative components (methylparaben, ethylparaben, isopropylparaben, propylparaben, isobutylparaben, butylparaben, phenoxyethanol, salicylic acid, benzoic acid, dehydroacetic acid), were prepared in solution as the analytical targets.
Fig. 1 shows the chromatogram obtained from analysis of a standard mixed solution using a 1 µL injection (each 100 mg/L, prepared with methanol), and Table 1 shows the analytical conditions used. For the analytical column, the high-speed Shim-pack XR-ODS Ⅱ analytical column was used. By adding acetic acid to the mobile phase, neither salicylic acid nor dehydroacetic acid displayed any tailing even using an ODS column, demonstrating that all the components can be separated.

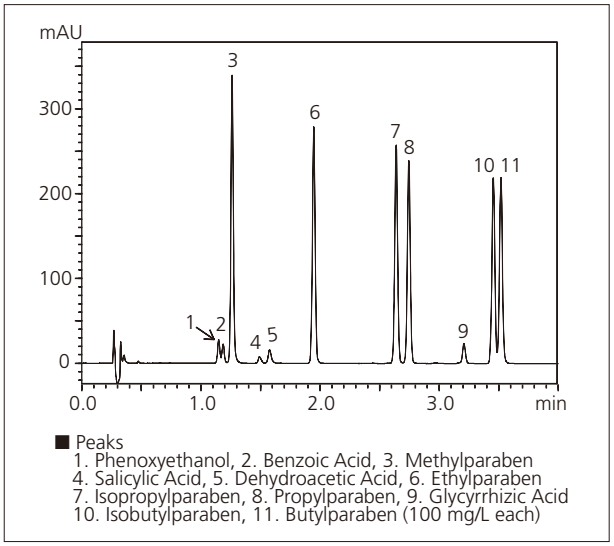

Fig. 1 Chromatogram of Standard Mixture of Active Ingredient (Glycyrrhizic Acid) and Ten Preservatives (100 mg/L each, 1 µL Injected)

Analysis of Quasi-Drug

Fig. 2 shows an example of analysis of a commercially available quasi-drug (medicated toothpaste). After weighing out 1 g of sample and adding 5 mL of methanol, ultrasonic extraction was conducted, and the supernatant was then filtered through a 0.22 µm pore-diameter membrane filter. The injection volume was 1 µL, while the other analytical conditions were the same as those of Table 1.
Salicylic acid, ethylparaben, propylparaben, and glycyrrhizic acid were separated and detected in a commercially available medicated toothpaste.

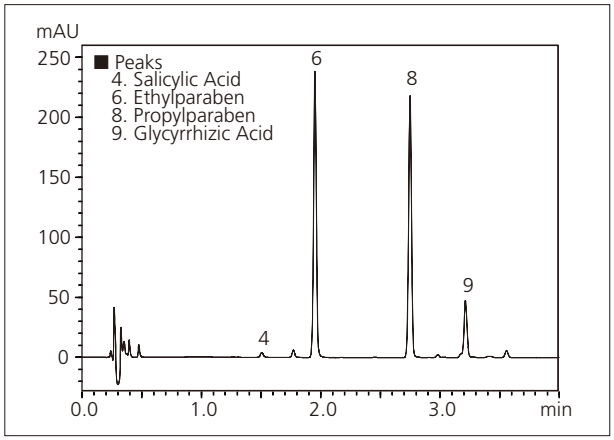

Fig. 2 Chromatogram of Medicated Toothpaste (1 µL Injected)

Analysis of a Cosmetic Product

Fig. 3 shows an example of analysis of a commercially available cosmetic (lotion mist). The supernatant of the sample was diluted two-fold with methanol, and then filtered through a 0.22 µm membrane filter. The injection volume was 1 µL, with the other analytical conditions the same as those of Table 1.
Methylparaben, ethylparaben, propylparaben, and glycyrrhizic acid were separated and detected in the commercially available lotion mist.

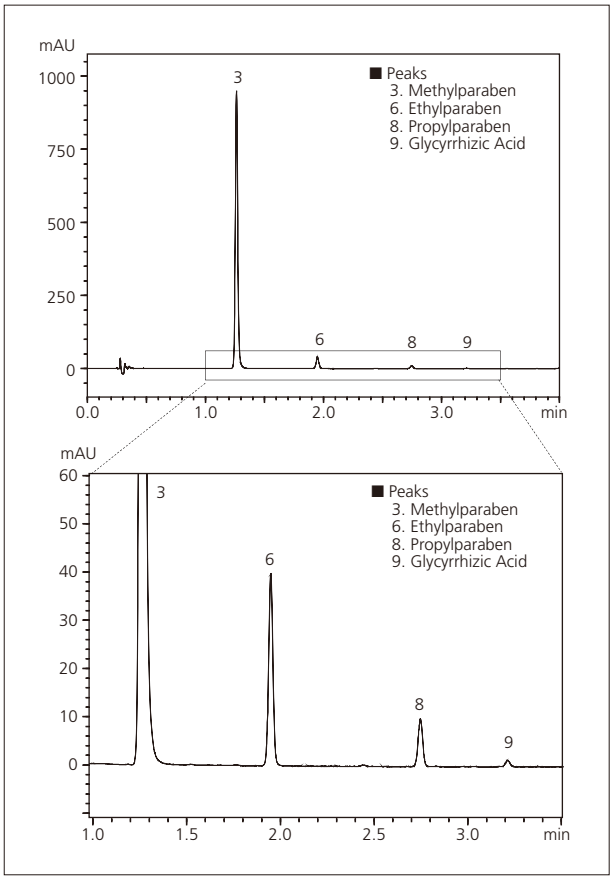

Fig. 3 Chromatogram of Lotion Mist (1 µL Injected)

UV-VIS Spectra of Target Compounds

Qualitative analysis of the various target compounds was conducted using the peak retention times obtained according to Fig. 2 and 3, and by then comparing the spectra obtained from those peaks with the spectra of respective standard substances (Fig. 1).
Fig. 4 shows the spectra of glycyrrhizic acid and salicylic acid contained in a quasi-drug (cosmetic product) acquired using the LC-2040C 3D (PDA model) with the spectra normalized to facilitate their comparison. Maximum absorption peaks associated with glycyrrhizic acid in the vicinity of 251 nm, and with salicylic acid at 237 and 301 nm, respectively, were detected.

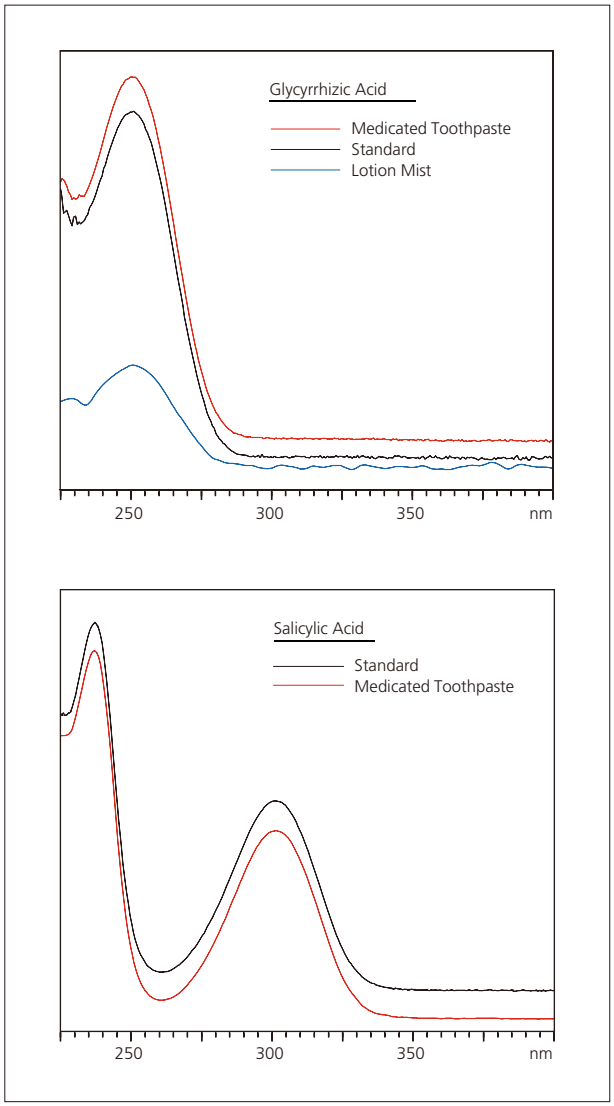

Fig. 4 Spectra of Active Ingredients and Preservative
Upper: Glycyrrhizic Acid
Lower: Salicylic Acid

First Edition: Jan. 2015

Shimadzu Corporation
www.shimadzu.com/an/