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Application News

No. L541

High Performance Liquid Chromatography

High-Resolution and High-Speed Simultaneous Analysis of Regulated UV-Adsorbents in Cosmetics using SPP Column

Many cosmetics contain ultraviolet light absorbents (UV-absorbents) to protect the skin from ultraviolet light. In Japan, the standards for cosmetics based on the Pharmaceutical Affairs Law (Ministry of Health and Welfare Notice No. 331, 2000) regulate the types and allowable quantities of UV-absorbents that may be used in cosmetic products. Since regulations can vary significantly between different countries and regions, quantification with HPLC is used to determine compliance with import/export regulations.

This report presents an example of high-throughput analysis of 23 kinds of UV-absorbents using the Nexera™ LC system and Shim-pack Velox™ C18 SPP (superficially porous particles, core-shell) column. This is a larger number of compounds than covered in a previous report (Application News No. L381), as it also includes some residues regulated by EU agencies.

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Analysis of Standards

Fig. 1 shows a 1 µL injection of a mixed solution of 23 UV-absorbents (100 mg/L each). The analytical conditions are shown in Tables 1 and 2. The 23 components of UV-absorbents that may be incorporated into cosmetics were analyzed (see Table 3 on the next page for details). All components showed good linearity with an R^2 value of 0.999 or higher within the range 1-100 mg/L.

Shim-pack Velox column enables high-speed separation while keeping back pressure low through the use of SPP technology. The system pressure in this analysis did not exceed 30 MPa.

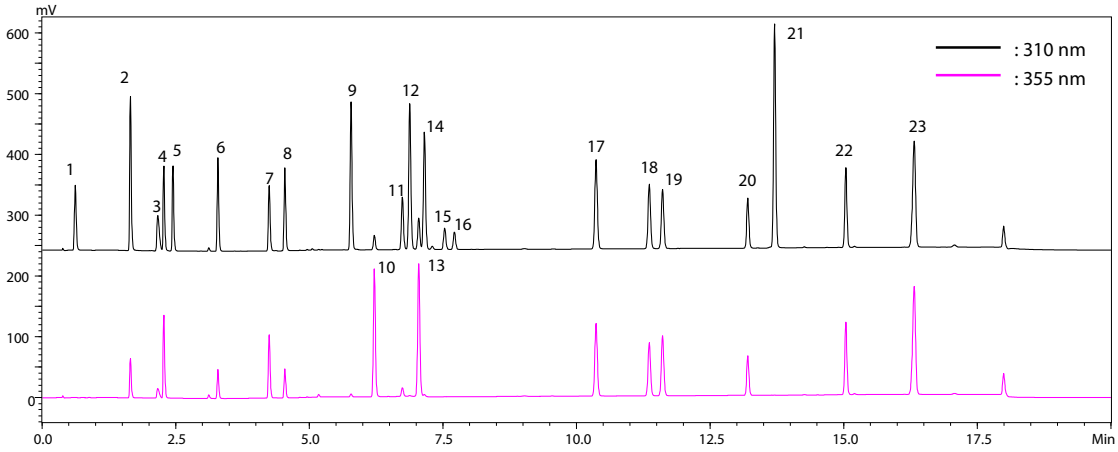
The flow rate of 1 mL/min enabled the accurate quantitation of 2,2'-methylenebis[6-(benzotriazol-2-yl)-4-tert-octylphenol) (peak 22) and bis-ethylhexyloxyphenol methoxyphenyl triazine (peak 23) within 20 minutes. This is a large reduction compared to the previous analytical time required for these two compounds.

Table 1 Analytical Conditions

Column	: Shim-pack Velox C18 (100 mm L. × 3.0 mm I.D., 2.7 µm)
Mode	: Low pressure gradient
Mobile Phase	: A) 0.1 % Formic acid in water B) Acetonitrile C) Methanol
Flow Rate	: 1 mL/min
Column Temp.	: 60 °C
Injection volume	: 1 µL
Detection	: 310 nm, 355 nm

Table 2 Time Program

Time (min)	A. Conc	B. Conc	C. Conc
0	90	10	0
2	60	40	0
4	25	0	75
14	0	0	100
17	0	0	100
17.01	90	10	0
20	90	10	0



1, 4-aminobenzoic acid
2, ferulic acid
3, 2-hydroxy-4-methoxybenzophenone-5-sulfonic acid
4, 2,2',4,4'-tetrahydroxybenzophenone
5, ethyl 4-aminobenzoate
6, 2,4-dihydroxybenzophenone
7, 2,2'-dihydroxy-4,4'-dimethoxybenzophenone
8, 2-hydroxy-4-methoxybenzophenone
9, 4-methylbenzylidene camphor
10, hexyl 2-[4-(diethylamino)- 2-hydroxybenzoyl]benzoate
11, 2-ethylhexyl 2-cyano-3,3-diphenylacrylate
12, 2-ethylhexyl-4-dimethylaminobenzoate
13, 4-tert-butyl-4'-methoxydibenzoylmethane
14, 2-ethylhexyl 4-methoxycinnamate
15, 2-ethylhexyl salicylate
16, 3,3,5-trimethylcyclohexyl salicylate
17, 2-benzotriazol-2-yl- 4,6-di-tert-butylphenol
18, 2-(2H-benzotriazol-2-yl)- 4,6-di-tert-pentylphenol
19, 2,4-di-tert-butyl-6- (5-chloro-2H-benzotriazol-2-yl)phenol
20, drometrizole trisiloxane
21, 2,4,6- tris[4-(2-ethylhexyloxycarbonyl)- anilino]-1,3,5-triazine
22, 2,2'-methylenebis[6-(benzotriazol-2-yl)-4-tert-octylphenol]
23, bis-ethylhexyloxyphenol methoxyphenyl triazine

Fig. 1 Standard Chromatograms of 23 UV-Adsorbents (100 mg/L Each).

Reproducibility

Table 3 shows the reproducibility of the retention times and peak area values over six repeated injections of a standard mixed solution (10 mg/L each) containing 23 UV-absorbents.

Table 3 Reproducibility of 23 UV-Absorbents (10 mg/L Each)

No.	Compound Name	Retention Time (%RSD)	Area (%RSD)
1	4-aminobenzoic acid	0.33	1.93
2	ferulic acid	0.25	1.48
3	2-hydroxy-4-methoxybenzophenone-5-sulfonic acid	0.41	1.98
4	2,2',4,4'-tetrahydroxybenzophenone	0.21	1.27
5	Ethyl4-aminobenzoate	0.23	1.06
6	2,4-dihydroxybenzophenone	0.13	1.05
7	2,2'-dihydroxy-4,4'-dimethoxybenzophenone	0.12	1.47
8	2-hydroxy-4-methoxybenzophenone	0.08	1.47
9	4-methylbenzylidene camphor	0.11	0.90
10	Hexyl 2-[4-(diethylamino)-2-hydroxybenzoyl] benzoate	0.10	0.98
11	2-ethylhexyl 2-cyano-3,3-diphenylacrylate	0.10	0.92
12	2-ethylhexyl-4-dimethylaminobenzoate	0.12	1.04
13	4-tert-butyl-4'-methoxydibenzoylmethane	0.12	1.31
14	2-ethylhexyl 4-methoxycinnamate	0.10	1.10
15	2-ethylhexyl salicylate	0.13	1.15
16	3,3,5-trimethylcyclohexyl salicylate	0.11	1.56
17	2-benzotriazol-2-yl-4,6-di-tert-butylphenol	0.11	0.90
18	2-(2H-benzotriazol-2-yl)-4,6-di-tert-pentylphenol	0.09	0.89
19	2,4-di-tert-butyl-6-(5-chloro-2H-benzotriazol-2-yl) phenol	0.10	1.19
20	drometrizole trisiloxane	0.08	0.94
21	2,4,6-tris [4-(2-ethylhexyloxycarbonyl)-anilino]-1,3,5-triazine	0.07	0.95
22	2,2'-methylenebis [6-(benzotriazol-2-yl)-4-tert-octylphenol]	0.05	0.90
23	bis-ethylhexyloxyphenol methoxyphenyl triazine	0.11	0.93

Analysis of Commercial Samples

Fig. 2 shows the analysis of a commercially available cosmetic cream. 100 mg of the sample was weighed out, and 5 mL of tetrahydrofuran added. An ultrasonic bath was used for extraction. After centrifugation, 0.5 mL of the supernatant was diluted to 10 mL with acetonitrile and filtered through a membrane filter with 0.22 µm pore size for analysis. The injection volume was 1 µL. All the components include with sample were below the maximum allowable content specified in the regulation.

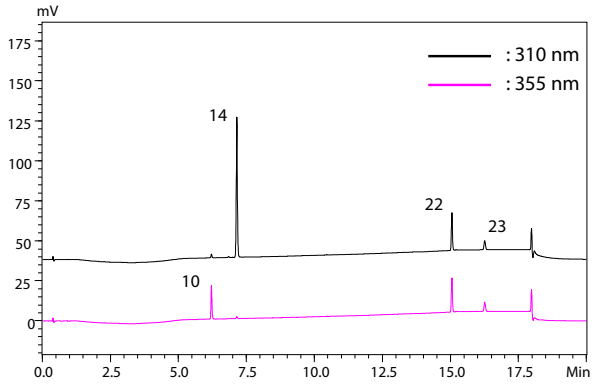


Fig. 2 Chromatograms of a Cosmetic Cream

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