APPLICATION NOTE

Detection of UV filters in cosmetic products (sunscreen) by HPTLC and confirmation by HPTLC-MS

A-103.1

Keywords

UVA, UVB, sunscreen, cream, UV spectrum, mass detection

Introduction

Sunscreen products contain UV filters that absorb or reflect a part of the sun's ultraviolet (UV) radiation. The identification of UV filters in cream can be a challenging task because of the multiple ingredients used in the cosmetic formulation. After simple sample preparation followed by HPTLC separation the presence of the target compounds can be confirmed by mass detection.

Scope

This method is suitable for the detection of octocrylene, avobenzone, octisalate, and enzulizol in finished products.
The TLC-MS interface 2 is used to elute target zones from HPTLC plates to confirm the presence of those UV filters by MS.

Required or recommended devices

Automatic TLC Sampler 4, Automatic Developing Chamber ADC 2, TLC Visualizer, TLC Scanner 4, visionCATS software, TLC-MS Interface 2, Waters ACQUITY QDa Detector (Performance), Empower® or MassLynx® software.

Sample and Standards

A sunscreen sample as well as octocrylene, avobenzone, octisalate, and enzulizol reference materials were provided by DSM Nutritional Products (Kaiseraugst, Switzerland).
To 100 mg of the cream or 10 mg of each individual standard, 5 mL of THF are added. The mixture is homogenized for 30 s by vortex mixing and extracted in an ultrasonic bath for 10 min at room temperature. 2 mL of H_2O and 3 mL of methanol were added to the mixture, which is then homogenized for 30 s by vortex mixing. After centrifugation for 10 min at 25°C, the supernatant is collected and used as test solution.

Chromatography

Stationary phase:	HPTLC Si 60 F_{254}, 20 x 10 cm (Merck)
Sample application:	Bandwise application, 15 tracks, band length 8 mm, track distance 11.4 mm, distance from left edge 20 mm, distance from lower edge 8 mm, application volume 2 µL.

Development 1	
Developing solvent 1	Heptane, ethyl acetate 8:2 (*v/v*)
Development 1	In the ADC 2 with chamber saturation (with filter paper) 20 min and after conditioning at 33% relative humidity for 10 min using a saturated solution of magnesium chloride
Developing distance 1	70 mm (from the lower edge)
Plate drying	Drying 5 min in the ADC 2
Development 2	
Developing solvent 2	Isopropanol
Development 2	In the ADC 2 with no saturation and no humidity control
Developing distance 2	28 mm (from the lower edge)
Plate drying	Drying 5 min in the ADC 2
Documentation	With the TLC Visualizer under UV 254 nm
Densitometry	Densitometric analyses are performed at 254 nm in absorption mode, at a scanning speed of 20 mm/s using a slit of 5.0 × 0.3 mm.
MS confirmation	Localizing the zones of the UV filters on the HPTLC plate: Marking zones to be eluted by using TLC Visualizer under UV 254 nm or based on R_F values of reference substances obtained by densitometry. Target zones are directly eluted using the TLC-MS Interface 2 with oval elution head into the ACQUITY QDa Detector at a flow rate of 0.5 mL/min with acetonitrile / water 95:5 (*v/v*) + 0.1% formic acid. For a full scan spectrum it is recommended to first elute a blank, which can be subtracted from the spectra of the target zones. There is no need for Single Ion Recording (SIR).
MS parameter	The ACQUITY QDa Detector is operated in ESI (+ / -) mode using default settings. The ESI capillary is set to 0.8 kV, cone voltage to 15 V, and desolvation temperature at 600 °C. A full scan mass spectrum between m/z 50 - 500 is acquired at a sampling rate of 10.0 points/sec (continuum). Data processing and evaluation of mass spectra are performed with Empower. For routine use in quality control Single Ion Recording (SIR) can be performed.

Results

System Suitability Test (SST) under UV at 254 nm:
Octocrylene zone at R_F ~ 0.46
Avobenzone zone at R_F ~ 0.41

NOTE: The presented results are to be regarded as examples only!

Please contact CAMAG for more application notes and products!

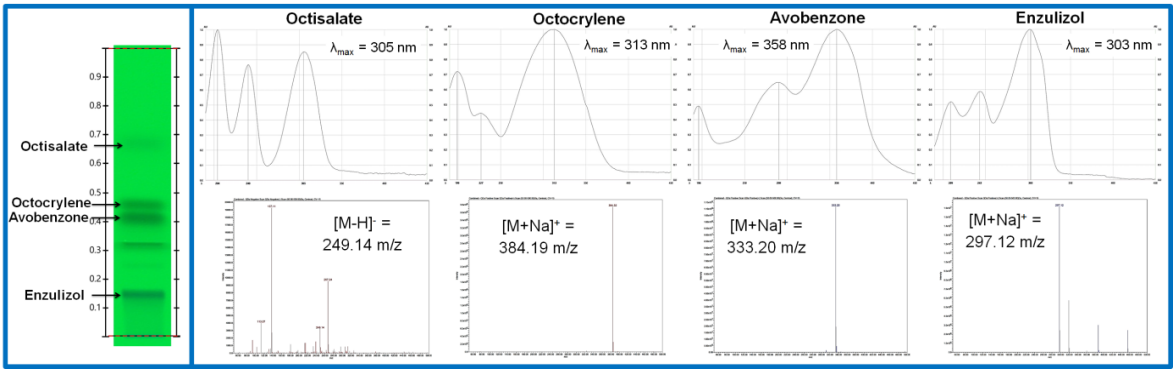

Fig. 1 Left: developed plate under UV 254 nm; right: UV spectra (displayed range from 190 to 450 nm) and mass spectra (displayed range m/z 50 to 500) of the standards

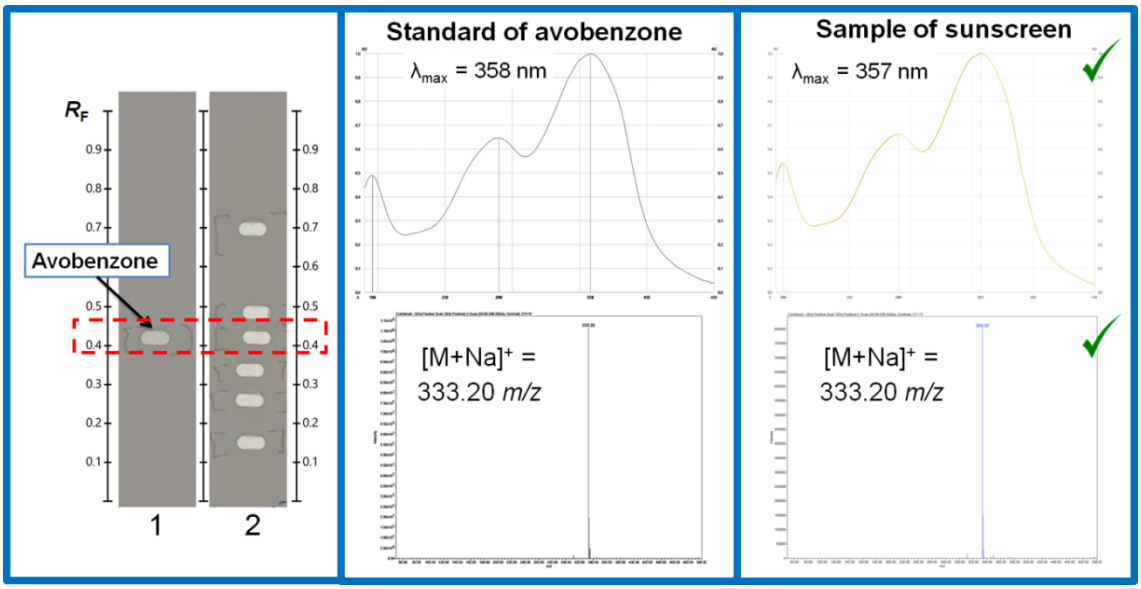

Fig. 2 Left: developed plate under white light; right: UV spectra (displayed range from190 to 450 nm) and HPTLC-MS spectra of Avobenzone (standard; track 1), and target zone (sample; track 2), displayed range m/z 50 to 500.

Conclusion

The safety of a cosmetic product being based on the safety of its ingredients comes from the fact that many different cosmetic products on the market are all derived from a limited number of substances. Hence toxicity testing has been concentrated on ingredients, and particularly on those that are intended to react with biological matrices and therefore are of most concern for human health such as UV filters. To safeguard the consumer health, a general method has been developed to detect UV filters by HPTLC and to confirm their identity by MS, before toxicity testing.

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Literature

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