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Fast Separation of Twelve Active Ingredients Used in Products Containing Sunscreen

INTRODUCTION

Dionex AN 223 shows separation of ten active ingredients found in sunscreen products and application of the separation to determine some of those ingredients in three products containing sunscreen.¹ In the work shown here, two additional active sunscreen ingredients are added to the standard mixture: benzophenone-4 and ethylhexyl triazone. In addition to developing a separation of the twelve sunscreen compounds, the authors transferred the method to the Dionex UltiMate® 3000 Rapid Separation Liquid Chromatography (RSLC) system.

The original Acclaim® 120 C18, 3 μm , 4.6 \times 100 mm column was replaced with a 2.2 μm Acclaim RSLC 120 C18, 2.1 \times 100 mm column. The authors adjusted the gradient, and used the bypass mode feature of the WPS-3000 split-loop sampler to reduce the gradient delay volume. The twelve sunscreen compounds were separated in ten min (Figure 1) with acceptable resolution and peak purity results (Table 1). This separation of twelve compounds was faster than the separation of ten in AN 223 and required less than half the eluent.

Table 1. Resolution and Peak Purity of Twelve Sunscreen Standards in an Injection of a Mixed Standard*

Compound	Resolution* (USP)	Match	RSD Match	PPI (nm)	RSD PPI
2-Phenylbenzimidazole-5-sulfonic acid (PHS)	16.31	999	0.38	235.6	0.16
Benzophenone-4 (B-4)	19.62	976	4.65	239.9	0.82
Benzophenone-3 (B-3)	6.80	992	3.34	237.9	1.23
Diethylamino hydroxybenzoyl hexyl benzoate (DHHB)	2.02	1000	0.23	293.8	0.08
4-Methylbenzylidene-camphor (4-MBC)	3.15	999	0.38	239.5	0.14
Octocrylene (OCR)	3.92	998	0.93	240.5	0.36
Methyl anthranilate (MA)	2.84	995	1.48	251.0	0.42
Octyl methoxycinnamate (OMC)	2.09	1000	0.15	243.7	0.06
Butyl methoxydibenzoylmethane (BMDM)	2.86	1000	0.20	291.6	0.07
Octyl salicylate (OS)	2.97	998	0.60	243.4	0.18
Homosalate (HMS)	22.22	1000	0.17	242.1	0.07
Ethylhexyl triazone (EHT)	n.a.	1000	0.04	245.3	0.01

*Detection at 310 nm (wavelength scanning 250 to 600 nm for peak purity)

EQUIPMENT

Dionex UltiMate 3000 RSLC system including:

SRD-3600 Integrated Vacuum Degasser

HPG-3400RS Binary pump with 200 μ L mixer kit
(P/N 6040.5150)

WPS-3000RS Split-loop sampler with 100 μ L
sample loop

TCC-3000RS Thermostatted Column Compartment

DAD-3000RS Diode Array Detector with semi-micro
(2.5 μ L, 7 mm) SST flow cell (P/N 6082.0300)

CONDITIONS

Column: Acclaim 120 RSLC C18, 2.2 μ m,
2.1 \times 100 mm (P/N 068982)

Mobile Phase: A: 0.8% Acetic acid
B: Ethanol

Gradient: Table 2

Maximum Pressure: 600 bar (8700 psi)

Flow Rate: 0.35 mL/min

Inj. Volume: 0.5 μ L

Column Temp.: 25 $^{\circ}$ C

Detection: 310 and 354 nm

Data collection rate, 25 Hz

Response time, 0.50 s

Table 2. Gradient Table

Time (min)	%A	%B	Remark
-4.00	75	25	
0.00	75	25	Start injection
0.10	75	25	Bypass sample loop and sample needle
0.11	75	25	
0.80	55	45	
1.50	55	45	
1.51	20	80	
5.00	20	80	
5.01	10	90	
10.00	10	90	

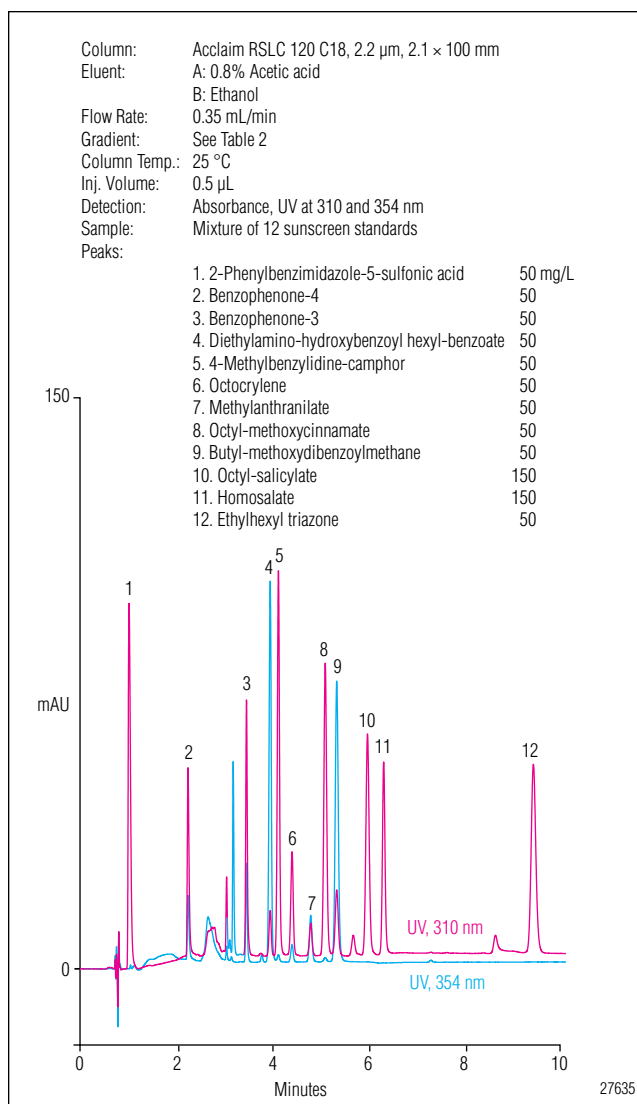


Figure 1. Separation of twelve sunscreen compounds.

REFERENCES

1. Dionex Corporation, *Determination of Ten Active Ingredients in Sunscreen-Containing Products in a Single Injection*. Application Note 223, LPN 2183, 2009, Sunnyvale, CA.

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