TECHNICAL NOTE

Liquid Chromatography

Flexar PDA Plus Detector

A Definitive Solution to Determine Peaks Co-elution and Product Adulteration

The PerkinElmer® Flexar™ PDA Plus™ detector with its impressive spectral sensitivity allows the comparison of the spectra on the upslope and downslope of peaks to determine compound purity. Thus, the peak purity is the ratio of the peak upslope and downslope spectra. A purity of less than 1.5 indicates similar spectrum along the peak and a purity of 1.5 or more indicates dissimilar spectrum along the peak that is typical of adulterated or co-eluting compounds.

Among the three peaks (A, B, C) shown in Figures 1-a, the compound represented by peak A has a peak purity of 6.6 (Figure 1-b). Such an impurity level calls into question the integrity of the compound. It is possible that peak A is a co-elution of two or more compounds. In Figures 2-a and 2-b, chromatographic conditions are changed. Injecting the same sample, four peaks instead of three are separated and all four peaks have a purity of less than 1.5. Peak A has been resolved into two peaks: A1 and A2, confirming the prior suspicion of co-elution.

Chromatographic conditions:

Mobile phase: 20% A (1.25% acetic acid in water)
80% B (acetonitrile)
Flush solvent: 75:25 methanol/water
Flow rate/injection volume: 1 mL/min; 2 uL
Column: Brownlee™ SPP C18, 150 x 3.0 mm, 2.7 µm
at ambient temperature (Cat# N9308411)
Analytical wavelength: 320 nm
Chromera® version 4.0. Sampling rate: 5 pts/sec

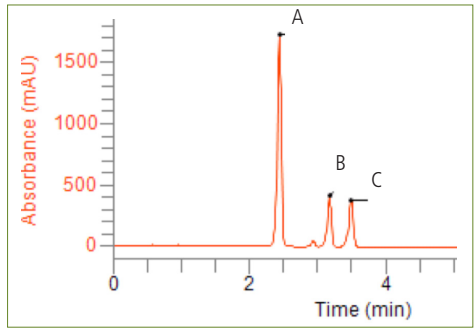

Figure 1-a: Chromatogram of a mixture of four sunscreen compounds with a presumed co-elution in peak A. A: octocrylene/avobenzone B: octisalate, C: monosalate

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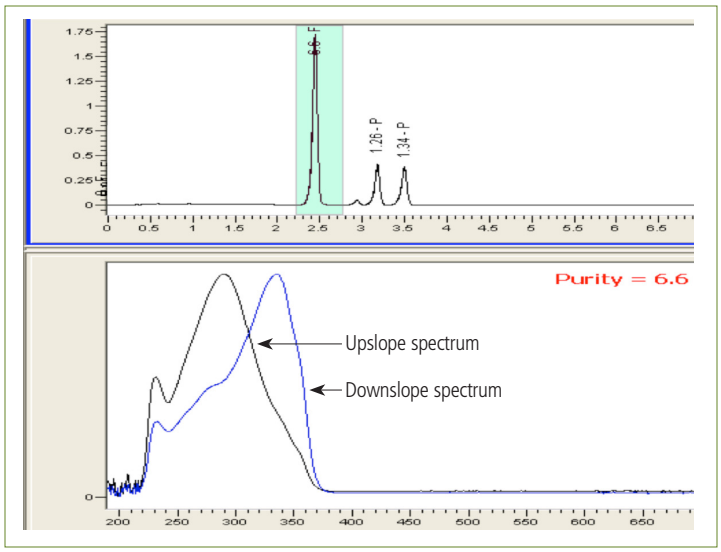

Figure 1-b: Purity evaluation of peak A, suggesting a co-elution in peak A.

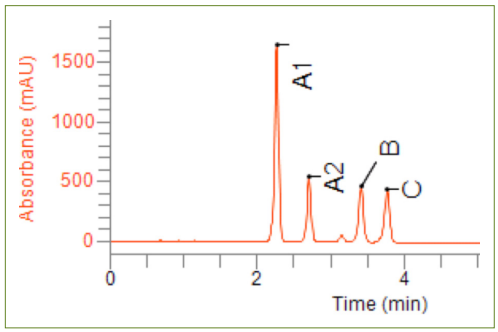

Figure 2-a: Chromatogram of a mixture of four sunscreen compounds with a presumed co-elution in peak A.

New chromatographic conditions:

Mobile phase: 15% A (1.25% acetic acid in water)
85% B (1:1 acetonitrile/methanol)
Flush solvent: 75:25 methanol/water
Flow rate/injection volume: 0.8 mL/min; 2 uL
Column: Brownlee™ SPP C18, 150 x 3.0 mm, 2.7 µm
Cat# N9308411 at ambient temperature
Analytical wavelength: 320 nm
Chromera® version 4.0. Sampling rate: 5 pts/sec

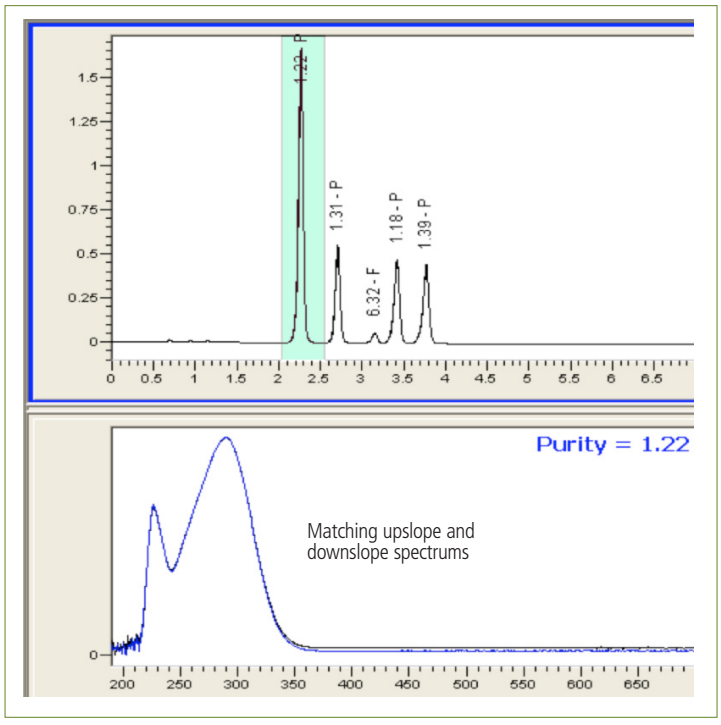

Figure 2-b: Peak purity evaluation of peak A1 showing no co-elution.
A1: octocrylene ; A2: avobenzone B: octisalate, C: monosalate.

Application of the purity feature of the Flexar PDA plus detector through Chromera or a third party chromatography data system helps to quickly detect coeluting peaks, thereby simplifying method development in R&D labs. For QC labs, it also helps provide more effective screening in detecting adulteration.

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