

Polymer Additive Analysis Study Using Tetrahydrofuran and Advanced Polymer Chromatography with Gradient Elution

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This is an Application Brief and does not contain a detailed Experimental section.

Abstract

This application brief familiarizes the scientists with the aggressive solvent compatible APC System using a gradient elution for the analysis of non-polar and polar polymer additives in one chromatographic result.

Benefits

The APC System, with a THF-compatible p-QSM, enables a gradient elution separation of polar and non-polar polymer additives in a mixture.

Introduction

Polymer manufacturers rely on the timely analytical characterization of their raw materials. One

aspect of polymer analysis is the qualification and quantification of polymer additives, such as Irganox 1010.¹ Analysis using legacy chromatographic methods and instrumentation can be time-consuming and tell only part of the story. However, transferring legacy high performance liquid chromatography (HPLC) methods to a Waters ACQUITY Advanced Polymer Chromatography (APC) System, with a Polymer Quaternary Solvent Manager (p-QSM) and an ACQUITY UPLC PDA (photodiode array) Detector, provides gradient elution of polymer additives using aggressive solvents like tetrahydrofuran (THF) quickly and without system modifications during installation. The addition of gradient elution capability to the APC System allows the baseline separation of polymer additives, such as Tinuvin 327 and Tinuvin 328, previously known to co-elute using traditional separation methods (Figure 1).²

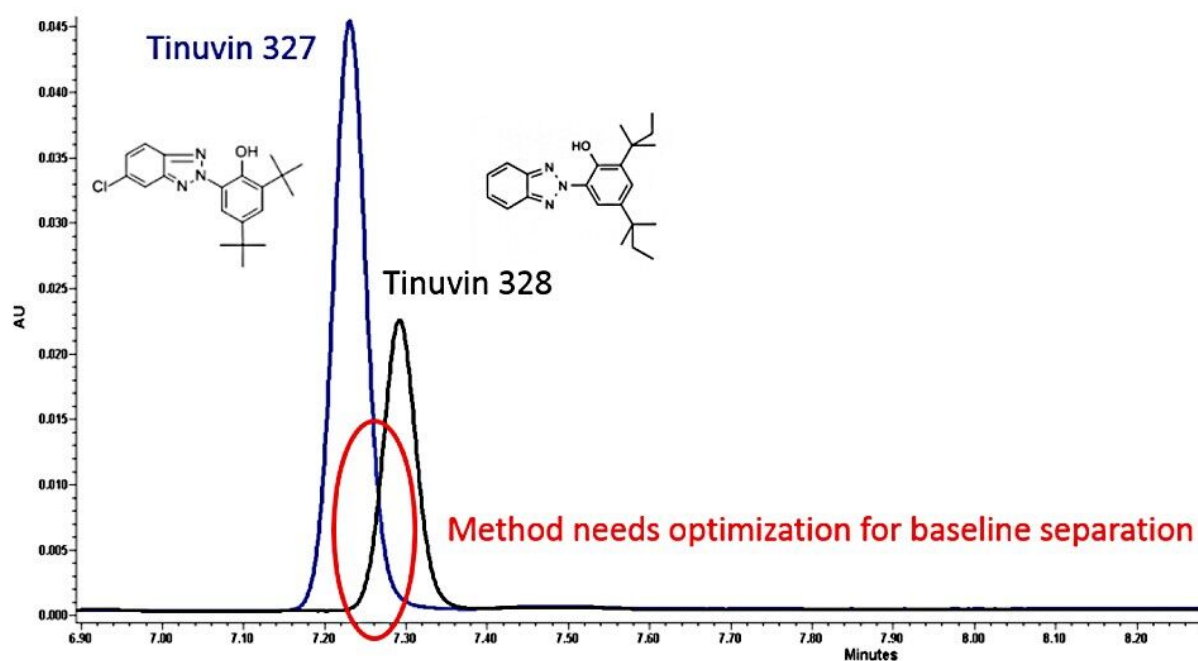


Figure 1. APC with p-QSM chromatographic overlay of Tinuvin 327 and 328.

Results and Discussion

The term polymer additives covers a wide variety of chemistries, and this experiment concentrates on two commonly used groups: polar and non-polar. A legacy polymer additive method is optimized

using the column calculator to accommodate a change in column chemistry with scalability and wide chemical compatibility for this analysis (Figure 2).³ The first experiment is separating the known co-eluting Tinuvin 327 and 328 using the THF-compatible APC System with a p-QSM elution gradient pump (Figure 3). The second experiment is separating a mixture containing non-polar Irganox samples 1330, 1010, and 1076 and the comparatively polar Tinuvin samples 328, 327, and 360 (Figure 4).

Transferring a legacy polymer additive separation method to a new column is easily done using a column calculator tool. For the analysis of polymer additives, the legacy method is transferred to the APC System with p-QSM for gradient elution using aggressive solvents, like THF. When entering the column choices, the ratio of column length to packing particle size (L/d_p) is kept constant between methods. According to United States Pharmacopeia (USP) guidelines, using a constant L/d_p allows the new method to be used without revalidation.⁴ The column calculator tool enables planning of the experiment, ordering columns, and consideration of system capabilities before entering the lab. Instrument capabilities, such as system pressures and solvent consumption, have an impact on safely performing analytical procedures.

Columns Calculator

Column 1
XBridge C18, 5 μ m, 4.6 x 150 mm

Column 2
XBridge C18, 2.5 μ m, 3 x 75 mm

From...
Describe your original method.

Column

Diameter (D): 4.600 mm
 Length (L): 150 mm
 Particle Size (dp): 5.0 μ m
 L/dp: 30,000

System

Dwell volume: 0.000 mL

Method

Injection volume: 20.0 μ L
 Temperature: 40 $^{\circ}$ C
 Run time: 18.00 min

Time (min)	Flow Rate (mL/min)	%A Water	%B Acetonitr	%C Methano	%D Water	Colu Value
0.00	1.000	50.0	0.0	50.0	0.0	0.00
1.00	1.000	50.0	0.0	50.0	0.0	0.61
10.00	1.000	1.0	0.0	99.0	0.0	5.47

1,100 psi
Maximum pressure

To...
Describe your target method.

Column

Diameter (D): 3.000 mm
 Length (L): 75 mm
 Particle Size (dp): 2.5 μ m
 L/dp: 30,000

System

Dwell volume: 0.000 mL
 High pressure limit: 15,000 psi

Method

Flow rate: Scaled (0.851 mL/min) Custom: 0.350 mL/min

Time (min)	Flow Rate (mL/min)	%A Water	%B Acetonitr	%C Methano	%D Water	Colu Value
3.50	0.851	1.0	0.0	99.0	0.0	2.43
1.63	0.851	50.0	0.0	50.0	0.0	0.30
4.50	0.851	50.0	0.0	50.0	0.0	2.13

4,399 psi Maximum pressure **4.3 μ L** Injection volume **4.50 min** Run time

Figure 2. Column calculator tool for transferring HPLC methods while maintaining L/dp.

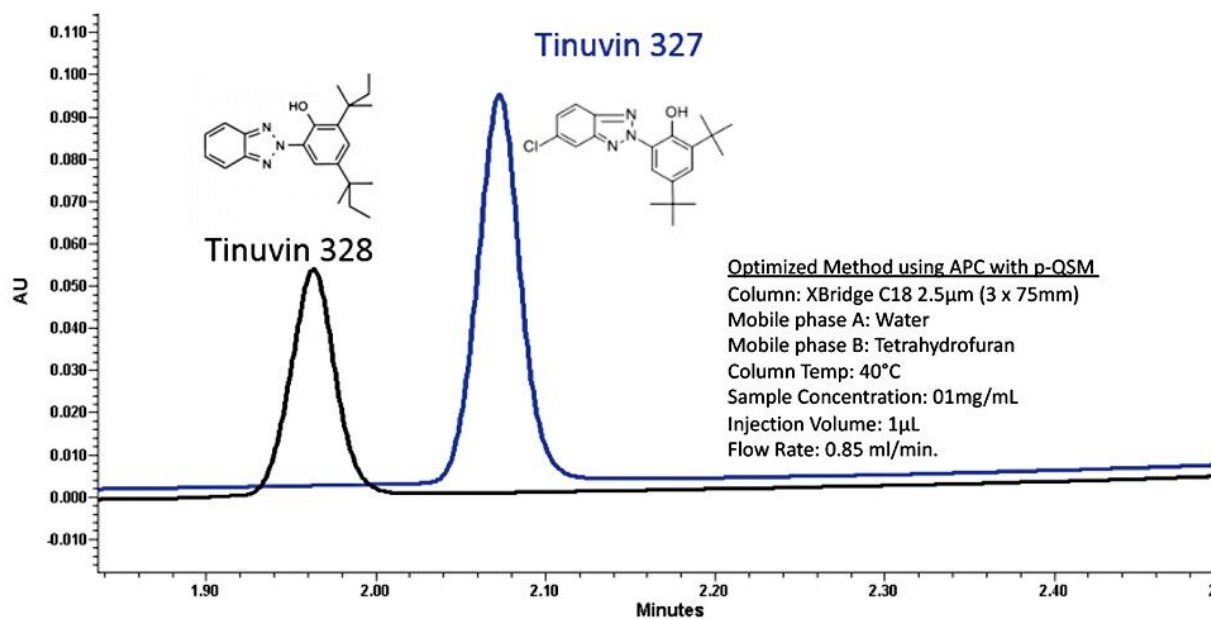


Figure 3. XBridge Column chemistry reversing the peak order of Tinuvin 327 and 328.

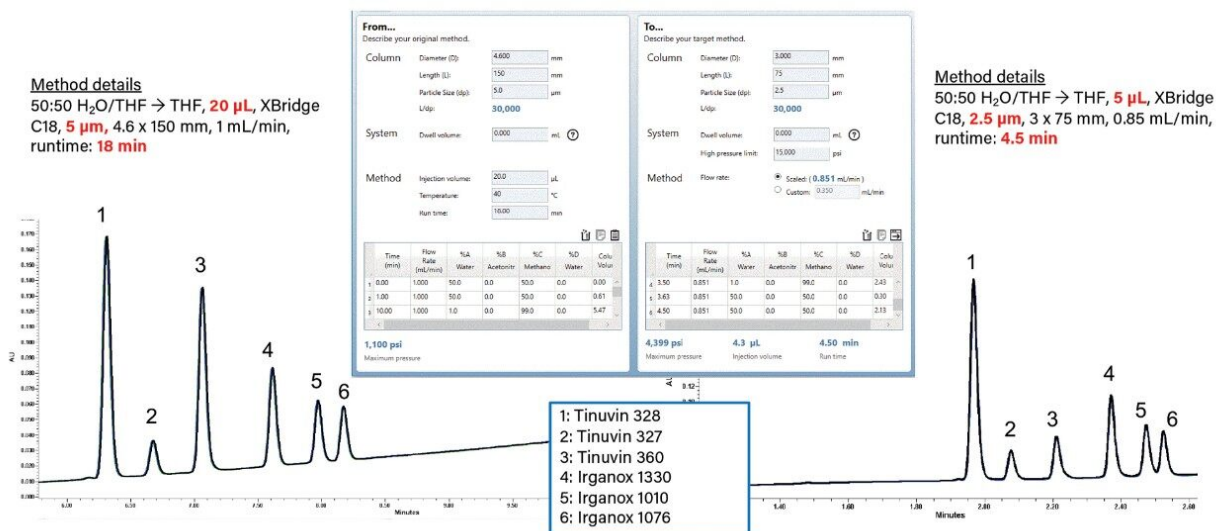


Figure 4. Separating a mixture of polar and non-polar polymer additives in one method.

Conclusion

The differing chemistries of polymer additives can affect their solubility in traditional mobile phases. By using the APC and p-QSM with aggressive solvent compatibility, the separation of mixtures with a wide variety of solubilities is less of a challenge. The separation of co-eluting Tinuvin samples, using a gradient with 100% THF capability, allows for more flexibility of method optimization. Also, the separation method for non-polar and polar polymer additives in one mixture is optimized and scalable using the robust XBridge BEH Column technology.

References

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<https://www.waters.com/waters/promotionDetail.htm?id=134694974&locale=101>.
4. United States Pharmacopeia (USP) general regulation <621>, <https://www.usp.org/frequently-asked-questions/chromatography>.

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