

응용 자료

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

Claudia Lohmann, Damian Morrison, Jennifer Gough

Waters Corporation

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

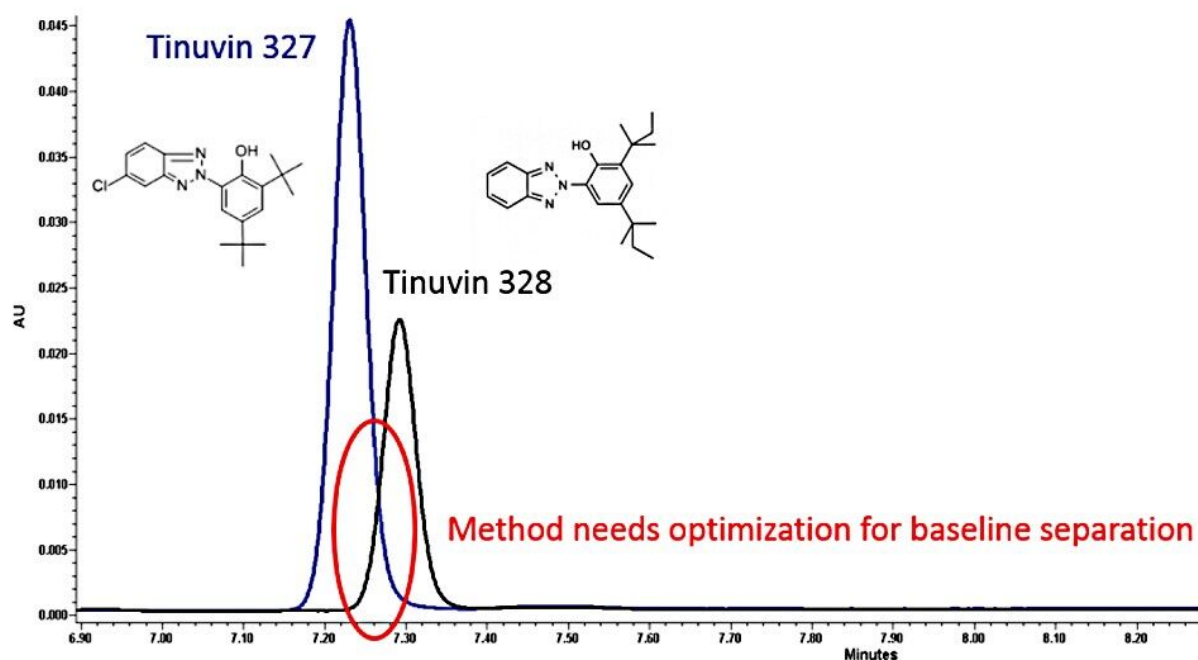


Figure 1. APC with p-QSM chromatographic overlay of Tinuvin 327 and 328.

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/dp) is kept constant between methods. According to United States Pharmacopeia (USP) guidelines, using a constant L/dp 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.

The screenshot shows the 'Columns Calculator' software interface. It is divided into two main sections: 'Column 1' and 'Column 2'. Both sections have a 'From...' and 'To...' area for describing the original and target methods, respectively. The 'From...' section for Column 1 includes: Diameter (D): 4.600 mm, Length (L): 150 mm, Particle Size (dp): 5.0 μm, L/dp: 30,000, System Dwell volume: 0.000 mL, Method Injection volume: 20.0 μL, Temperature: 40 °C, and Run time: 18.00 min. The 'To...' section for Column 2 includes: Diameter (D): 3.000 mm, Length (L): 75 mm, Particle Size (dp): 2.5 μm, L/dp: 30,000, System Dwell volume: 0.000 mL, High pressure limit: 15,000 psi, and Method Flow rate: Scaled (0.851 mL/min). Below these input fields are two tables showing the resulting HPLC method parameters. The first table for Column 1 shows a maximum pressure of 1,100 psi. The second table for Column 2 shows a maximum pressure of 4,399 psi, an injection volume of 4.3 μL, and a run time of 4.50 min.

| Time (min) | Flow Rate (mL/min) | %A Water | %B Acetonitr | %C Methano | %D Water | Colu Volur |
|------------|--------------------|----------|--------------|------------|----------|------------|
| 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 |

| Time (min) | Flow Rate (mL/min) | %A Water | %B Acetonitr | %C Methano | %D Water | Colu Volur |
|------------|--------------------|----------|--------------|------------|----------|------------|
| 3.50 | 0.851 | 1.0 | 0.0 | 99.0 | 0.0 | 2.43 |
| 3.63 | 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 |

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

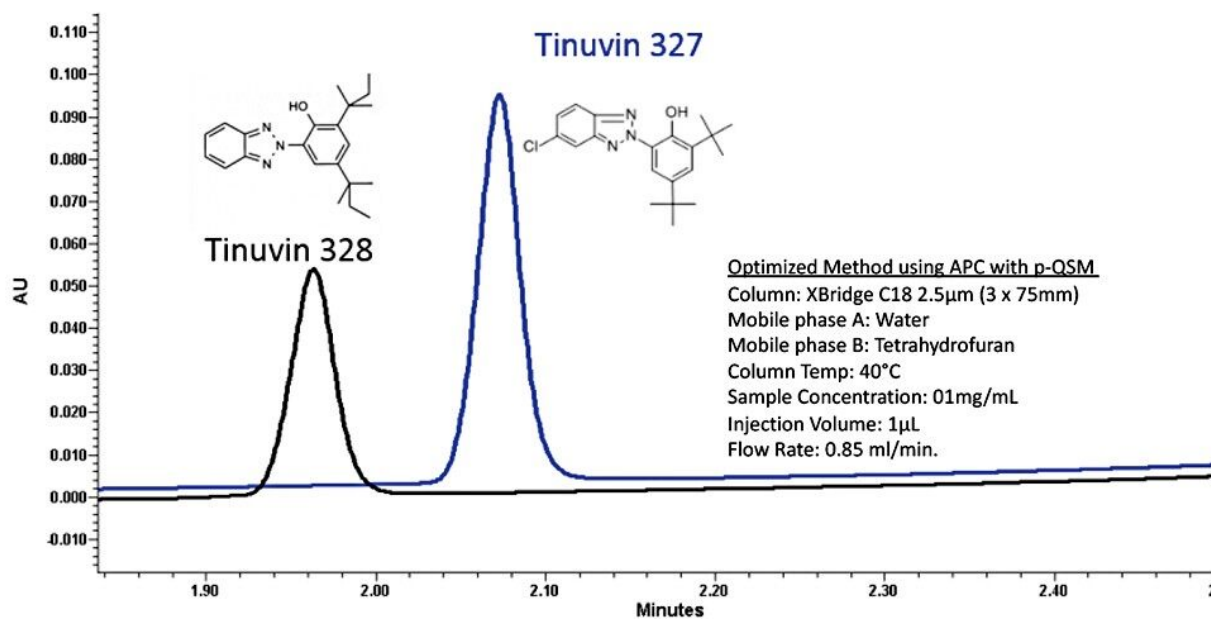


Figure 3. XBridge Column chemistry reversing the peak order of Tinuvin 327 and 328.

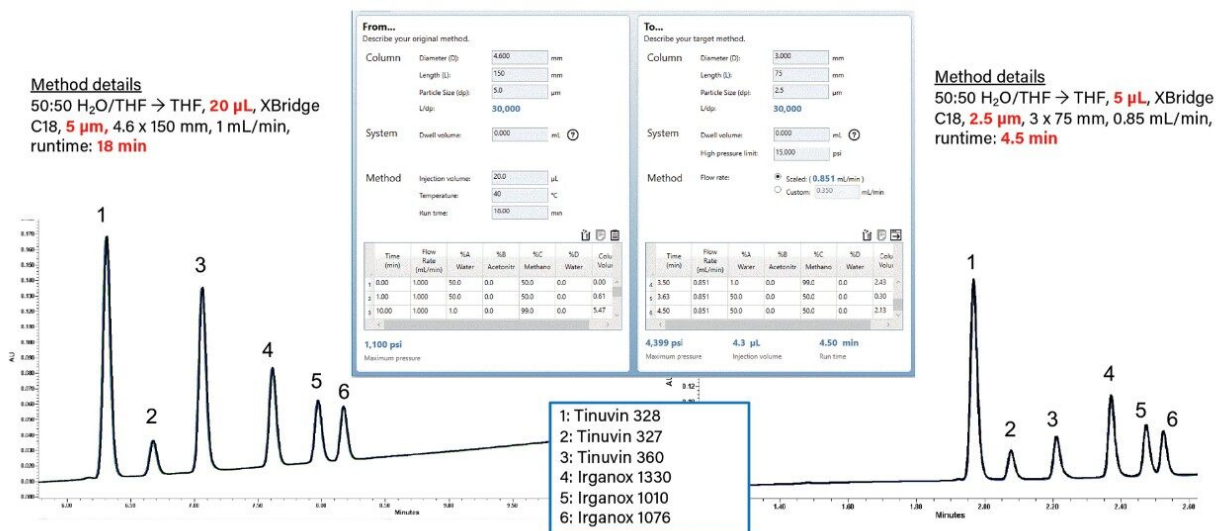


Figure 4. Separating a mixture of polar and non-polar polymer additives in one method.

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

1. Bolgar, Michael; Hubball, Jack; Groeger, Joseph; Meronek, Susan; Handbook for the Chemical Analysis of Plastic and Polymer Additives, CRC Press, Boca Raton, U.S.A., 2016.
2. "Comparison of HPLC and UHPLC Analysis of Polymer Additives with Elucidation of Mass Detection", Waters Corp. U.S.A., 2017. <https://www.waters.com/waters/library.htm?lid=134956804&cid=511436>.
3. Waters On-Line Tool Box, "Column Calculator Tool", <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>.

Featured Products

[ACQUITY UPLC PDA Detector <https://www.waters.com/514225>](https://www.waters.com/514225)

[ACQUITY Advanced Polymer Chromatography System <https://www.waters.com/134724426>](https://www.waters.com/134724426)

720006707, October 2019

©2019 Waters Corporation. All Rights Reserved.