This technical bulletin details the separation of mono and di-esters from BPA(EO)30DMA methacrylated polyol. The current method for Surface Specialties UCB’s analysis of the mono- to di-ester ratio in a final polymer product, specifies an HPLC method which is 30 minutes in length. However, their current method required two injections per process sample, thereby bringing the total analysis cycle time to approximately 60 minutes. Here we report a new method using ZirChrom®-PBD at a column temperature of 80°C with a total analysis time of only 10 minutes.

Introduction

Surface Specialties UCB is a company located in Smyrna, Georgia that manufactures specialty chemicals, coating resins, additives, and technical resins. Surface Specialties UCB approached ZirChrom method developers with the challenge of shortening the run time of an analysis of mono- to di-ester ratio in a final polymer product. Their current method for the analysis of the mono- to di-ester ratio in a final polymer product specifies an HPLC method less than 30 minutes in length. In practice, however, two injections are required for each process sample bringing the total analysis time to approximately 60 minutes. Since the HPLC analysis is used for online monitoring of the reaction, a 60 minute analysis time meant it was too late to stop a reaction that had gone too far. ZirChrom Solution: Use a ZirChrom®-PBD column and elevated temperature to speed up the analysis!

Experimental

A mixture of mono and di-esters from BPA(EO)30DMA methacrylated polyol was separated at 80 °C using a ZirChrom®-PBD column. The separation conditions were as follows:

- **Column:** ZirChrom®-PBD, 150 mm x 4.6 mm i.d. (Part Number: ZR03-1546)
- **Mobile Phase:**
  - A: water
  - B: acetonitrile
- **Time %A %B**
<table>
<thead>
<tr>
<th>Time (min.)</th>
<th>%A</th>
<th>%B</th>
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<tbody>
<tr>
<td>0</td>
<td>80</td>
<td>20</td>
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<tr>
<td>5</td>
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- **Temperature:** 80 °C with Metalox® 200-C column heater
- **Flow Rate:** 1.5 ml/min.
- **Injection Vol.:** 5 µl of 1 mg/ml sample
- **Pressure Drop:** 155 bar
- **Detection:** UV at 240 nm

**Figure 1:** Separation of Mono and Di-ester at 80 °C using ZirChrom®-PBD and the Metalox® 200-C.

Customer Allyson Norman, Analytical Scientist with Surface Specialties UCB had this to say about the ZirChrom method; “ZirChrom method developers reduced our analysis turn around time from 1 hour to 10 minutes!”

The ZirChrom value assessment program: (available on the ZirChrom website: [http://www.zirchrom.com/documents/value.xls](http://www.zirchrom.com/documents/value.xls)) predicts reducing the total analysis time from 60 minutes (30 minutes/injection) to 10 minutes (5 minute/injection) will save UCB roughly $20.00/sample in total analysis costs. This translates to roughly $158,600/year in savings (savings based on possible cycles/instrument/year using the ZirChrom column). This savings calculation takes into account the cost of purchasing a Metalox® 200-C high temperature column heater and a ZirChrom®-PBD column. Although the cost savings alone are impressive, having the analysis completed before the reaction has gone too far is priceless.

This method can be tailored to your specific application needs. ZirChrom technical support can help to optimize and transfer this technology to your site. Please contact ZirChrom technical support at 1-866-STABLE-1 or support@zirchrom.com for details. For more information on the Metalox® 200-C high temperature column heater visit [www.metalox.com](http://www.metalox.com).

ZirChrom phases offer unique selectivity, high efficiency, and excellent chemical and thermal stability.

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Visit [www.zirchrom.com](http://www.zirchrom.com) for more application notes using ultra-stable, high efficiency ZirChrom columns.