COMPARISON OF HPLC AND UHPLC ANALYSIS OF POLYMER ADDITIVES WITH ELUCIDATION OF MASS DETECTION

INTRODUCTION
Polymer manufacturers rely on their timely analytical and quality control services for the characterization of their raw materials. One aspect of polymer analysis is the qualification and quantification of polymer additives such as Irganox 1010. Legacy instrumentation methods lead to long analysis times and tell only part of the story. However, transferring legacy high performance liquid chromatography (HPLC) methods to an ultra-high pressure liquid chromatography (UHPLC) system method, with photodiode array (PDA) and added mass detection, can provide the information in one quarter of the time with an assigned mass to charge ratio for unique peak and impurity identification. The addition of mass detection can identify Irganox 1010 and its unknown components in order to see subtle changes that may affect product quality, yet enable control charts and monitoring specification limits.

METHODS

For the HPLC and UHPLC sample analyses, polymer additives were dissolved in isopropanol. A mobile phase consisting of 10mM ammonium formate in water [A] and in acetonitrile [B] was used with a phenyl column, 4.6 x 150 mm, 5.0 µm (HPLC) and 4.6 x 75 mm, 2.5 µm (UHPLC). The column temperature was maintained at 40 °C. PDA was collected at 254nm wavelength and mass detection with default settings were used.

Chromatographic System Control
Waters Empower 3 FR3 Software for instrument control, data acquisition and chromatographic data processing.

DISCUSSION & RESULTS

Using a legacy HPLC method for the analysis of the polymer additive Irganox 10/10, the method is transferred from the Waters Alliance HPLC system to the Waters ACQUITY Arc UHPLC system were calculated by entering the method parameters into the column calculator tool (Figure 1 & 2). The column choices are based on keeping the ratio of column length to particle size constant between methods. The column calculator tool enables planning of the experiment, ordering columns, and consideration of system capabilities before entering the lab. System capabilities such as pressure limits and solvent consumption for waste reservoirs have an impact on safely performing analytical procedures.

The HPLC and UHPLC chromatograms are compared in Figure 3 with the MS spectra from the ACQUITY Arc/QDa analysis shown below. With the assigned mass to charge ratio (m/z) as further characterization, the QDa mass detection can be helpful in the quality control (QC) assurance of a polymer additive raw material. This mass to charge ratio could be quantified in a concentration series, and then control charted for internal QC. These chromatographic peaks can be monitored over time to alert the users of batch to batch changes or manufacturing plant changes.

Once the polymer additives have passed internal QC, the additives are blended with proprietary formulations. These blends can be quickly confirmed with the ACQUITY Arc UHPLC analysis, and the assigned m/z (Figure 4 & 5). From the previously created concentration curves, the proprietary blends can be checked for process control of blending procedures and formulations.

CONCLUSIONS

- Transferring methods from HPLC to UHPLC can significantly shorten analysis time.
- LC method development is easily calculated and transferred to a ultra pressure system using the family of Waters columns and the column calculator tool.
- Bringing higher sensitivity to chromatographic detection by adding mass detection in these LC experiments brings expanded polymer additive characterization while maintaining a robust separation, lower solvent consumption, and shorter analysis times.

References

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