

Nota applicativa

Rapid Polymer Analysis with Atmospheric Solids Analysis Probe/Mass Spectrometer (ASAP/MS)

Waters Corporation

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

Abstract

This technology brief describes how to successfully and rapidly analyze short chain natural and synthetic specialty polymer, surfactant, and oligomeric materials in order to provide absolute molecular weight profiles, data, and valuable material architecture in five minutes or less using desorption mass spectrometry with Waters ASAP sample inlet and ACQUITY SQ Detector (SQD).

Benefits

ASAP/MS provides data including absolute molecular weight profiles for polymer materials in less than five minutes.

Introduction

Analysis of specialty polymers and surfactants is often limited to size-based analysis, such as Size Exclusion

Chromatography (SEC) with an appropriate detection method. Inherent in this technique is the requirement for a suitable calibration protocol that takes into account detector bias and chromatographic stability. Further, as product space expands to include multi-functional materials, the SEC approach is limited when addressing compositional variation in the material.

The goal of matching the SEC analysis with mass spectrometry can provide necessary compositional analysis, but it is fraught with significant challenges. Typical SEC solvents, such as THF, DMF, and toluene, do not allow for a suitable environment for mass spectral analysis. Infusion of polymeric material for mass spectral analysis has been explored, but this approach is limited due to ionization suppression effects caused by the competing ionization of many infusion solvents. Use of ASAP as a sample inlet provides for direct mass spectral analysis.

As a sample inlet, ASAP eliminates the solvent impact since the sample is loaded directly onto the capillary tip of the probe, vaporized using heated gas, and then ionized using a corona discharge.

Results and Discussion

ASAP coupled to ACQUITY SQD has proven to be a powerful laboratory tool for polymeric analysis. The utility of the solids probe provides a simple, direct, and rapid mode of sample introduction. Due to sample desorption from the probe tip, the analyte is introduced without interference from solvents, allowing consistent ionization of the analyte. The resulting thermally desorbed molecular chains are ionized across their molecular weight distribution.

The analysis is completed in a few steps:

- The sample is dissolved in solvent and applied to the tip of a melting point capillary tube. The solvent is flashed off of the tip in the first seconds of the analysis due to the controlled desolvation gas flow and temperature. The analyte is left on the capillary tip free of background solvent and related ionization and suppression effects.
- The polymeric material thermally desorbs or volatilize from the tip under controlled desolvation gas temperature and flow.
- As the analyte molecules volatilize they are ionized.

- The ionized molecules are detected using the ACQUITY SQD.

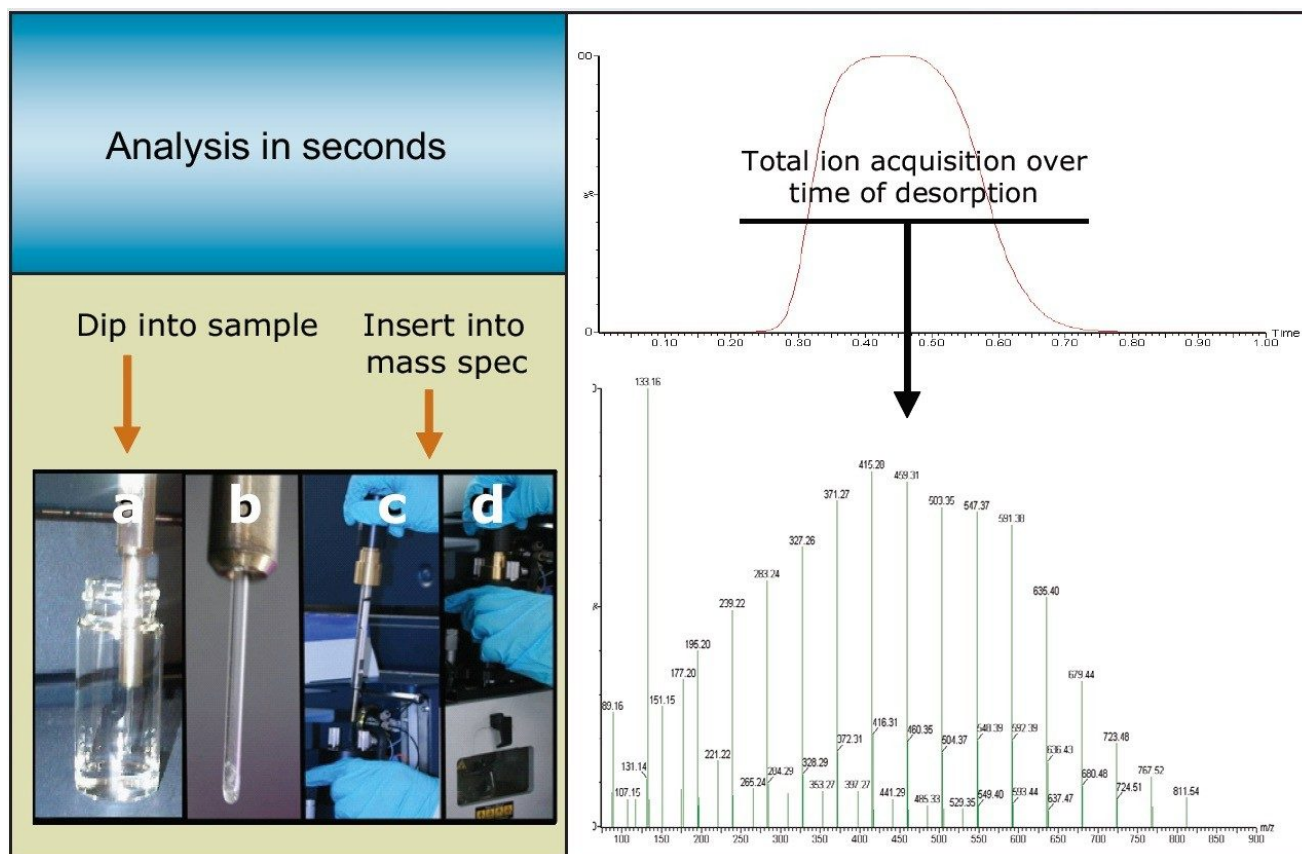


Figure 1. Using ASAP as a sample inlet, a sample is loaded directly onto the capillary tip of the probe, vaporized using heated gas, and then ionized using a corona discharge.

The resulting thermal desorption data is tabulated based on the m/z (equal to mass for singly charged molecules) and abundance of each polymer chain length. The mass is adjusted for proton inclusion and the adjusted mass and abundance data is combined and summed over the weight distribution.

Sample: $\text{CH}_3(\text{CH}_2)_m\text{O}(\text{CH}_2\text{CH}_2\text{O})_n\text{OH}$ Where $m = 10-14$ and $n = 1-14$

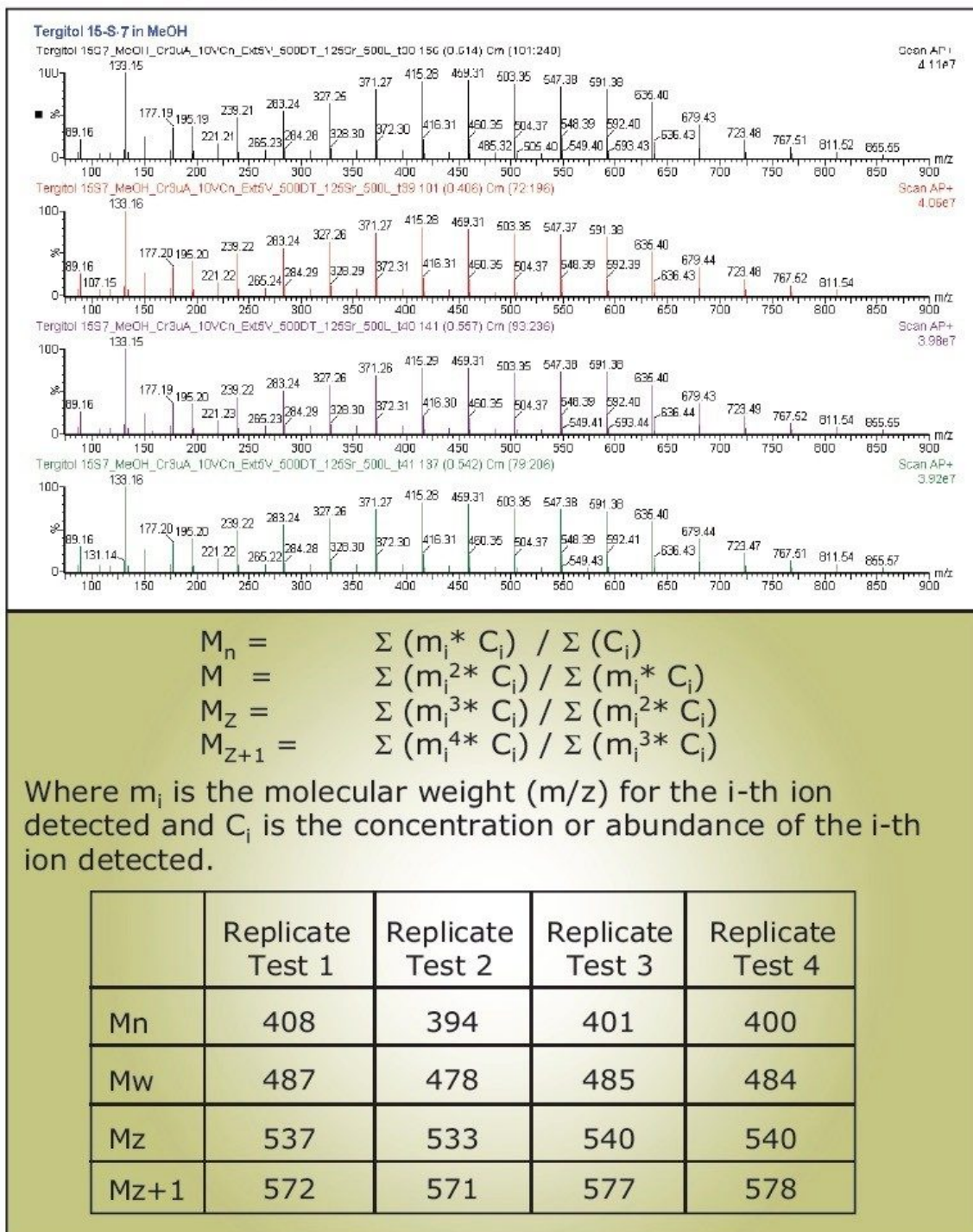


Figure 2. The summed data is computed as the number average, weight average, Z average, and

Z+1 average molecular weights (M_n , M_w , M_z , and M_z+1).

Conclusion

The versatility and advantages of Waters ASAP/SQD approach has shown that a broad array of samples can be evaluated in one or two minutes, depending on the sample type and its volatility. Reproducible data can be easily obtained without sample specific method development. Further, the unique mass spectral signature of the sample allows for the analysis of compositional 'fingerprint' variations not seen with conventional size exclusion separation analysis. The approach offers a reduction in the time required for analysis and operator training, as well as elimination of costs associated with solvent consumption and waste treatment.

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SQ Detector 2 <<https://www.waters.com/134631584>>

720003840, February 2011

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