Waters Integrity System Applications

Detection and Identification of Polymer Additives in Surgical Sponge Material

Highlights: Extracted surgical sponge material was analyzed for the presence of polymer additives; advantages of MS and PDA dual detection capabilities are featured for compound identification

Positive compound identification using combined PDA and MS detection is a powerful analytical tool. This technique, featuring the Integrity LC/MS System, has wide utility for industrial applications. In the following example, confirmation and identification of polymer additives in surgical sponges using the Integrity System is discussed. Urethane based sponge material was extracted by the included method. The chromatographic conditions also follow:

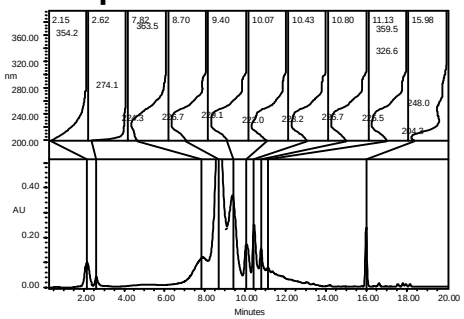
►Extraction Procedure:

1. Weigh 5.0 grams of sponge material in a beaker.
2. Cover with 50 ml of Ethyl Acetate.
3. Sonicate for 30 min. in a warm water bath.
4. Concentrate extract to 2.0 ml.

►Chromatographic Conditions:

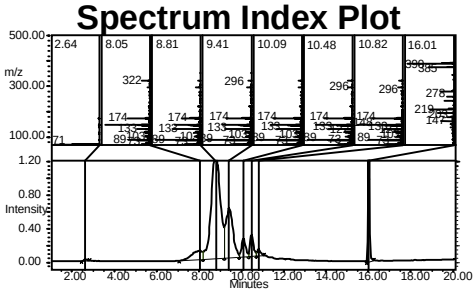
Column: Symmetry C -18 (3.0 mm x 150.0 mm)
Conditions: 30% Acetonitrile to 100% Acetonitrile in 15 min. Hold for 15 min. at 100 % Acetonitrile.
Flow Rate: 0.4 ml/min.
Injection Volume: 20 ul

Urethane Sponge Extract PDA Spectrum Index Plot



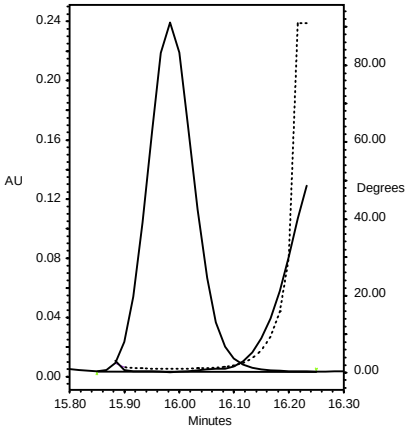
The power of the combined detection capabilities of the Integrity system allows for simultaneous acquisition of PDA and MS data. Consequently, PDA and MS spectra can be displayed above each chromatographic peak as in the Spectrum Index Plots for the urethane sponge extract shown at left. The UV Spectrum Index Plot also has the ability to display the point of maximum impurity for each chromatographic peak. Similar retention times for the PDA and MS chromatograms shows minimal loss of chromatographic resolution from the PDA detector through the ThermaBeam interface

Urethane Sponge Extract MS Spectrum Index Plot



The peaks eluting from approximately 8 minutes to 11 minutes are urethane oligomers. The early eluting peaks in this series probably have molecular ions below 1000 m/z, while the later eluting ones have molecular weights well in excess of 1000 amu. The peak of interest is the one eluting at 16.01 minutes in the MS chromatogram and at 15.98 min in the PDA chromatogram. The mass spectrum clearly shows the spectral characteristics of a hindered phenol.

Unknown Antioxidant Purity Plot



Peak Purity Passes Table

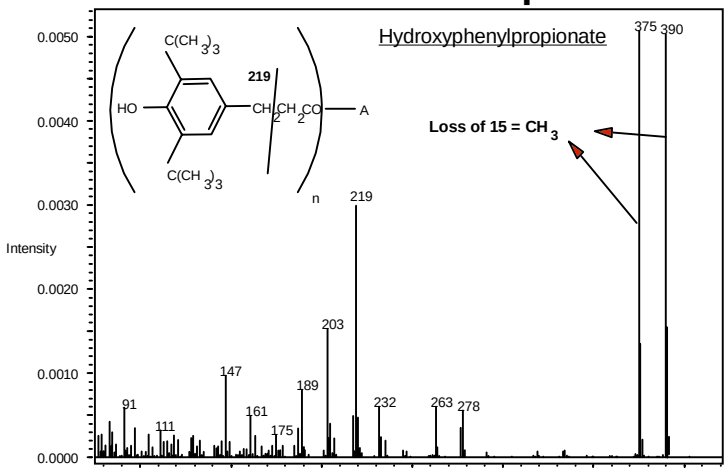
#	Purity Angle	Purity Threshold	Purity Flag	Maximum Impurity
1	0.321	1.074		16.033

Purity Threshold .....

Purity Angle ——

PDA data yields information on peak homogeneity, linearity and diagnostics as well as the ability to search UV spectra against a user built library. When focusing on the peak of interest from page one, the illustration at left shows the Peak Purity Plot for this "unknown" material. From these data, this peak is determined to be homogeneous.

Unknown Antioxidant Spectrum



At right is the mass spectrum for the unknown peak of interest. The spectrum contains ions which are characteristic of hindered phenols. The spectrum is similar to the spectrum for Irganox 1076. The molecular ion for this particular additive is 390 Da. This assumption can be made by examining logical losses from the molecular ion. For example, m/z 390 minus 375 gives a difference of 15 mass units indicating a loss of a methyl group. The ion produced at m/z 219 is formed by breaking the bond between the adjacent CH2 groups off of the ring.
We can further calculate the structure of this hindered phenol by looking at the isotopic contribution from C 13 at m/z 391. The approximate C 13 contribution from the fragment ion at m/z 390 indicates there are 25 carbons in this molecule. We know that the structure of a hindered phenol will contain three oxygen atoms. The calculated mass is therefore 348 and the difference would be made up by hydrogen atoms. The chemical formula for this molecule would thus be C25O3H42.

Positive compound identification of polymer additives is easily achieved using the Integrity LC/MS System. Simultaneous acquisition of PDA and MS data on a single injection and seamless processing and reporting of both channels of data as well as automated library search capabilities offer confidence in peak identification.