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Agilent Technologies

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Introduction

The alkyl amine ethoxylate emulsifier Ethomeen T/20H is a multi-compound, polymeric emulsifier used in agricultural and oil additive formulations. It is composed of polymers with the general structure $C_xH_yNH(CH_2CH_2O)_{14}$ -H. When these polymers fragment, they repeatedly lose (CH₂CH₂O) groups, causing mass spectra with peaks 44 *m/z* apart (see Figure 1).



Figure 1. Full scan mass spectrum of polymer m/z 698.6 with 44 m/z monomeric losses representing C₂H₄O.

In this method, the components of Ethomeen T/20H are separated and analyzed by LC/MSⁿ using TurboDDS, a datadependent scanning and software capability of the Varian 500-MS Ion Trap Mass Spectrometer. In TurboDDS analyses, a full scan "survey" is performed to search for precursor ions of interest. Once a "trigger" ion is found, MSⁿ is performed to the desired depth (n=2, 3, 4, etc.). These data-dependent analyses make it possible to gain the maximum amount of information for a mixture of unknown components.

Instrumentation

- Varian 500-MS Ion Trap LC/MS with ESI source
- Varian 212-LC Binary Solvent Delivery Modules
- Varian ProStar[™] 430 AutoSampler

Application Note 01507

Characterization of Alkyl Amine Ethoxylate Emulsifier (Ethomeen® T/20H) Using TurboDDS™ on the Varian 500-MS Ion Trap LC/MS

HPLC	Conditions
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Column:	Pursuit [™] XRs C18 3 µm, 150 x 3.0 mm ID				
	(Varian Part	No. A3001	1150X030)	
Solvent A:	0.1% formic acid in water				
Solvent B:	0.1% formic acid in methanol				
LC Program:	Time	06	0/6 B	Flow	
_	(min:sec)	90 A	90 A 90 D		
	00:00	100	0	200	
	02:00	100	0	200	
	12:00	0	100	200	
	17:00	0	100	200	
	17:01	100	0	200	
	20:00	100	0	200	
	100 1				

Injection Volume: 100 µL

MS Parameters

Ionization Mode:	ESI (positive)	
Needle:	5000 V	
Shield:	600 V	
Nebulizing Gas:	20 psi	
Drying Gas:	25 psi at 350 °C	

TurboDDS Parameters

Survey Scan Range:	<i>m/z</i> 50-1000
Capillary Voltage:	50 V
RF Loading:	100%
MS ⁿ Depth:	n=4
MS ² Breadth:	n=3
MS ³ Breadth:	n=3
MS ^₄ Breadth:	n=1
Trigger Threshold:	4000 counts

Results & Discussion

This method was set up to detect the components of the emulsifier Ethomeen T/20H. Figure 2 shows the total ion chromatogram (TIC) and extracted ion chromatograms from a full scan analysis. The TIC is pictured on top, and then the extracted ion chromatograms of the five most abundant components are shown below the TIC.

After the full scan run was performed, a TurboDDS run was carried out to identify and analyze the components of Ethomeen T/20H. The survey scan showed several large chromatographic peaks, the largest of which was m/z 726.6. In the mass spectrum for each of these components, the mass spectral peaks spaced 44 m/z apart indicate that these are the polymers of interest (see Figure 3).



Figure 2. TIC and extracted ion chromatograms of five polymeric components of Ethomeen® T/20H emulsifier.



Figure 3. Full scan spectrum of m/z 726.6 with 44 m/z spacing for monomeric losses of C₂H₄O.

TurboDDS[™] was set to perform MS⁴ on the most abundant mass in a particular survey scan, so m/z 726.6 trigged a MS⁴ analysis. The software easily displays a product ion stacked chromatogram, shown for m/z 726.6 in Figure 4.



Figure 4. Product ion stacked chromatogram showing one fragmentation pathway of m/z 726 with MS⁴ fragmentation.

Conclusion

The Varian 500-MS Ion Trap LC/MS and TurboDDS are powerful tools for the analysis of mixtures with unknown components. In this analysis, a mixture of polymeric compounds was separated and analyzed using automatic data-dependent scanning software.

This type of unknown analysis allows for the maximum amount of information in the minimum amount of time. The analysis is quick, simple, and results are easily synthesized and exported for analysis and presentation.

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