

NOTICE: This document contains references to Varian. Please note that Varian, Inc. is now part of Agilent Technologies. For more information, go to www.agilent.com/chem.



Application Note 01507

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

Joe Stork and Tiffany Payne
Varian, Inc.

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 (CH_2CH_2O) groups, causing mass spectra with peaks 44 m/z apart (see Figure 1).

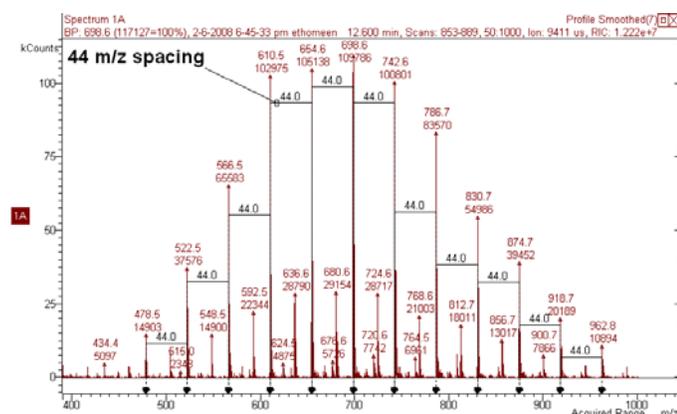


Figure 1. Full scan mass spectrum of polymer m/z 698.6 with 44 m/z monomeric losses representing C_2H_4O .

In this method, the components of Ethomeen T/20H are separated and analyzed by LC/MSⁿ using TurboDDS, a data-dependent 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

HPLC Conditions

Column: Pursuit™ XR C18 3 μ m, 150 x 3.0 mm ID (Varian Part No. A3001150X030)

Solvent A: 0.1% formic acid in water

Solvent B: 0.1% formic acid in methanol

| LC Program: | Time (min:sec) | %A | %B | Flow (μ L/min) |
|-------------|----------------|-----|-----|---------------------|
| | 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 |

Injection Volume: 100 μ L

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: m/z 50-1000

Capillary Voltage: 50 V

RF Loading: 100%

MSⁿ Depth: $n=4$

MS² Breadth: $n=3$

MS³ Breadth: $n=3$

MS⁴ 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).

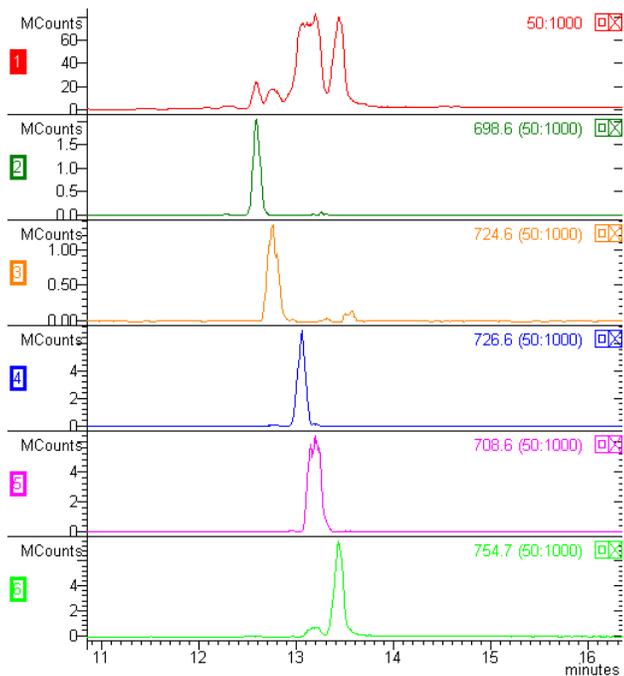


Figure 2. TIC and extracted ion chromatograms of five polymeric components of Ethomeen® T/20H emulsifier.

