Analysis of Surfactants Using the Agilent 500 Ion Trap LC/MS

Application Note

Authors

Joe Stork and Tiffany Payne

Agilent Technologies, Inc.

5301 Stevens Creek Boulevard

Santa Clara, CA 95051

USA

Abstract

Four surfactants were identified in rolling oil used to roll out large sheets of aluminum using the Agilent 500 Ion Trap LC/MS. The full scan sensitivity of the 500 Ion Trap makes it ideal for identification of unknown components in a mixture at an unknown concentration.

Introduction

In this analysis, surfactants were identified in rolling oil used to roll out large sheets of aluminum. It was suspected that surfactant contaminants were causing the oil to foam up over time, and in order to identify the surfactants in the oil, samples were analyzed by LC/MS using the Agilent 500 Ion Trap LC/MS. In addition to using the 500 Ion Trap for initial confirmation of oil contaminants, continuous monitoring of the oil for presence of surfactants maintains optimal operation and efficiency of these machines over time. The full scan sensitivity of the 500 Ion Trap makes it ideal for this type of analysis, where the analysis must identify unknown components in a mixture at an unknown concentration. In addition, the MS/MS capabilities of the instrument make it a powerful tool for structural elucidation and identification of unknown compounds.

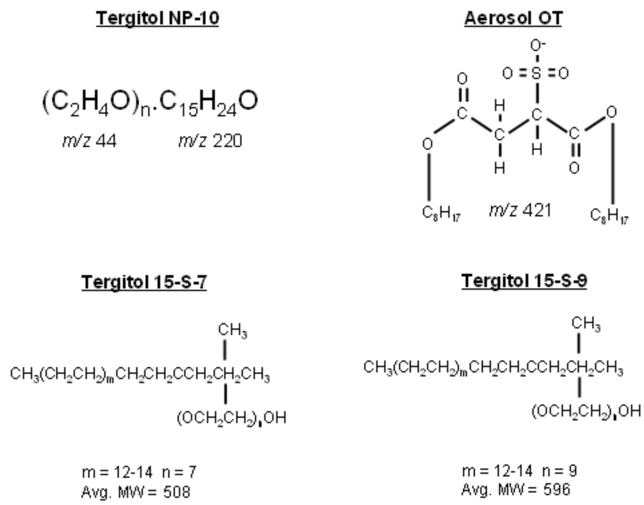

Figure 1. Chemical structures of Tergitol NP-10, Tergitol 15-S-7, Tergitol 15-S-9, and Aerosol OT surfactants.

Instrumentation

The following instruments were used in this study:

- Agilent 500 Ion Trap LC/MS with ESI source
- Agilent 212-LC Binary Solvent Delivery Modules
- Agilent ProStar 430 AutoSampler

Materials and Reagents

A clean sample of Hygold 100 petroleum distillate rolling oil was supplied for analysis, along with neat standards of Tergitol NP-10, Tergitol 15-S-7, Tergitol 15-S-9, and Aerosol OT.

Sample Preparation

ESI positive compounds

- **Neat rolling oil** (blank) was Hygold 100 petroleum distillate, Ergon Refining MSDS, CAS #64742-52-5. The oil was agitated in water (acidified) to release any water soluble components into the aqueous phase. The aqueous layers were injected and analyzed by LC/MS.
- **Neat surfactant materials** included Tergitol NP-10 in Hygold 100, Dow MSDS 1985; Tergitol 15-S-7 surfactant, Union Carbide MSDS 1917; Tergitol 15-S-9 surfactant, Union Carbide MSDS 1912. The neat surfactant materials dissolved in rolling oil were added to acidified aqueous (0.2% acetic acid) and agitated to release surfactants into the aqueous phase. The aqueous layer was analyzed by LC/MS.

ESI negative compounds

- **Neat rolling oil** (blank) was Hygold 100 petroleum distillate, Ergon Refining MSDS, CAS #64742-52-5 and was agitated in water (alkaline) to release surfactants into the aqueous phase. The aqueous layer was injected and analyzed by LC/MS.
- **Surfactant material** was Aerosol OT surfactant in Hygold 100, Alcoa MSDS #120778. The surfactant dissolved in rolling oil was added to alkaline aqueous (0.2% ammonium hydroxide) and agitated to release surfactants into the aqueous phase. The aqueous layer was injected and analyzed by LC/MS.

HPLC Conditions

Column:	Inertsil ODS-3,100 × 4.0 mm, 3 µm (Pursuit C18 Recommended Agilent Alternative p/n A3001100X020)
Solvent A:	0.2% acetic acid in water (ESI+) 0.2% ammonium hydroxide in water (ESI-)
Solvent B:	Methanol

LC program:

Time (min:sec)	%A	%B	Flow (µL/min)
00:00	95	5	200*
01:00	95	5	200*
11:00	0	100	200
31:00	0	100	200
31:01	95	5	200*
36:00	95	5	200*

* diverted

Injection volume: 10 µL

MS parameters

Ionization mode:	ESI (positive) and ESI (negative)
API drying gas:	15 psi at 300 °C
API nebulizing gas:	20 psi
Needle:	4500 V –5500 V
Shield:	600 V –600 V

Table 1. MS Segment Parameters

Analyte	Scan range	Retention time (min)	Capillary voltage (V)	RF load
Tergitol NP-10	200–700	13.48	100	85
Tergitol 15-S-7	329 > 280–320	11.12	75	82
Tergitol 15-S-9	315 > 280–320	11.68	75	82
Aerosol OT	313 > 280–320	12.30	75	82

Results and Discussion

The surfactants analyzed in this method were spiked into blank Hygold rolling oil. Figure 2 shows a chromatogram that compares a full scan blank injection with the subsequent spiked injections containing the surfactants of interest. The blank chromatogram is the extracted ion chromatogram for all of the ions of interest for all the surfactants tested. As the chromatogram shows, no background signal for the surfactants was observed in the blank rolling oil.

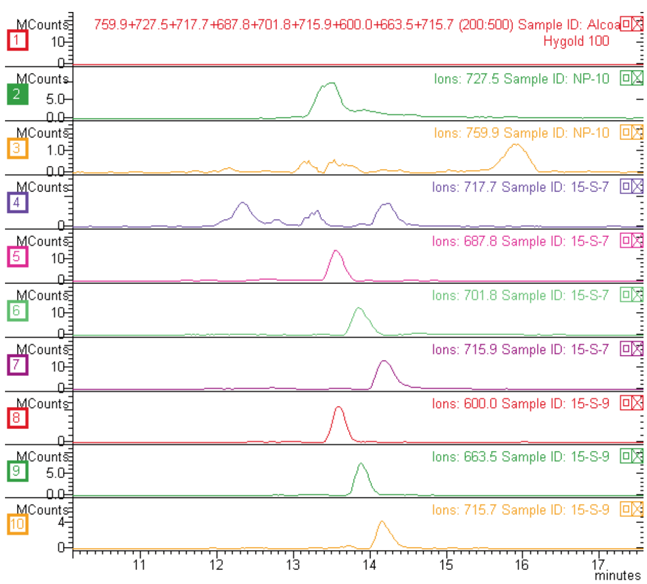

Figure 2. Chromatogram of a blank injection (top) followed by the same blank spiked with Tergitol NP-10, Tergitol 15-S-7, and Tergitol 15-S-9.

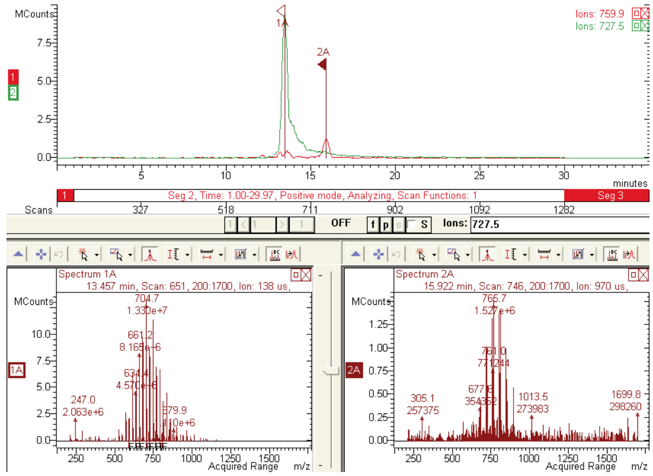

Figure 3. Extracted ion chromatogram and mass spectra for Tergitol NP-10 surfactant in Hygold 100 rolling oil, full scan analysis.

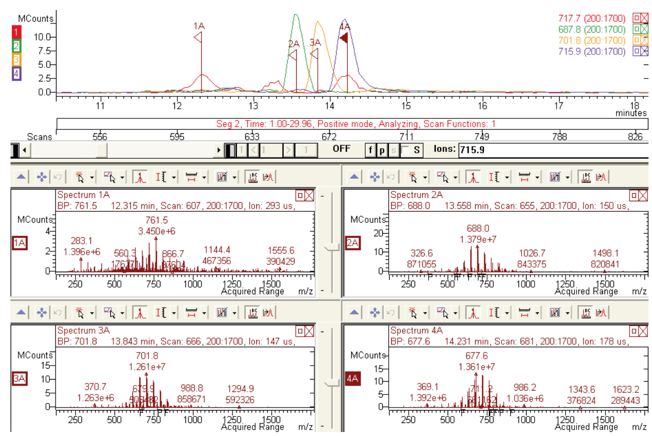

Figure 4. Extracted ion chromatogram and mass spectra for Tergitol 15-S-7 surfactant full scan analysis.

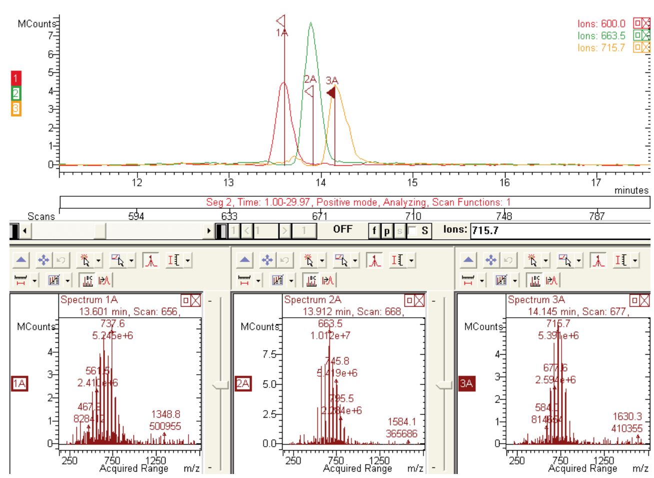

Figure 5. Extracted ion chromatogram mass spectra for Tergitol 15-S-9 surfactant full scan analysis.

The surfactant samples have interesting mass spectra with repeated losses of 44 mass units, representing a loss of C_2H_4O groups from the polymeric chain. Figure 6 shows the mass spectrum of Tergitol NP-10. The fragmentation of NP-10 is observed and a characteristic cluster is seen.

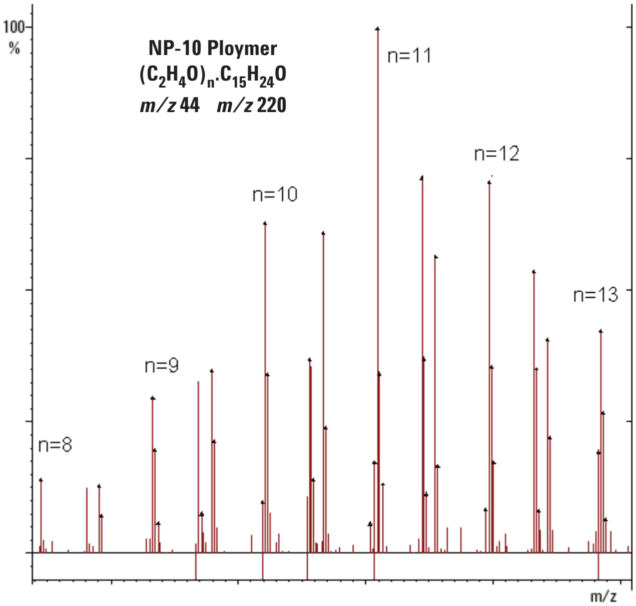

Figure 6. Mass spectrum of Tergitol NP-10.

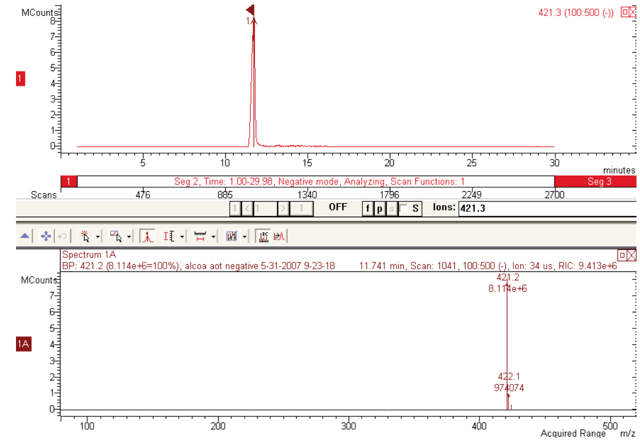

Figure 7. Extracted ion chromatogram of full scan analysis of Aerosol AOT surfactant.

Conclusion

Four surfactants were extracted from rolling oil for machines that roll out large aluminum sheets. The components of the oil were unknown, which made the full scan sensitivity of the Agilent 500 Ion Trap LC/MS ideal for this analysis. By extracting the ions of interest, clear chromatographic peaks are observed and the unique mass spectra are identified.

For More Information

These data represent typical results. For more information on our products and services, visit our Web site at www.agilent.com/chem.

www.agilent.com/chem

Printed in the USA
February 24, 2011
SI-1558

Agilent Technologies