

Application Brief AB-064

IMS DETECTION OF DOBER DETERGENT - CHEMATIC 82AE

Introduction

An IMS method for determining trace quantities of Dober's Chematic® 82AE detergent (a basic detergent) has been developed using the IONSCAN-LS. The method is based on the detection of the surfactant LAE (linear alcohol ethoxylate), a material also found in other Dober detergents – such as the acidic Chematic 91AE and the neutral Chematic 99AE – and variations of this method can readily be developed to analyze for those detergents.



Theory

Cleaning verification methods for detergents are generally based on the detergent component that is the most difficult to rinse, and therefore a good indicator of effective cleaning. The surfactant is that component in detergent formulations: the one that is the “last to leave.” Surfactants typically make up 0.5 – 10 percent of a detergent formulation, and they are often mixtures of oligomers of different chain lengths. Consequently, “total” surfactant gives rise to several peaks in an IMS plasmagram. This is the case with surfactant LAE. The method in this application note is based on the detection of the lowest molecular weight LAE peak.

Experimental

The method uses High Performance Injection (HPI) in cold injection mode with staged temperature ramping. The HPI method developed performs the following:

1. Evaporates the solvent and vents it out the split vent.
2. Ramps the temperature up to sequentially vaporize the different LAE components.
3. Vents the least volatile detergent components using the split vent.

Temperature ramping is a crucial part of the method. It is used to separate the vaporization of various LAE components over time and reduce their interference. By using this temperature-staging approach, methods can be developed for detergents containing other related surfactants.

The HPI method is shown schematically in Figure 1.

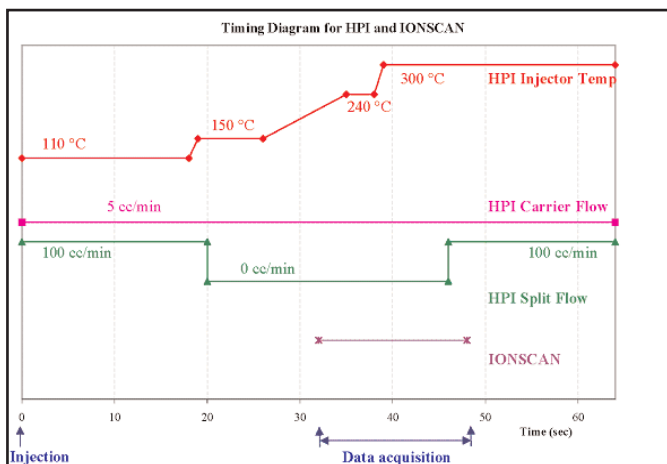


Figure 1. HPI Method Parameters for the Analysis of Chematic 82 AE

Temperature and Flow Staging

In this method, the sample is injected at 110 °C (cold), with the split flow at 100 cc/min. Using a high split flow, the solvent (water) vaporizes and is sent out the split vent rather than through the IMS. The temperature is quickly ramped up to 150 °C and the split flow vent closed. After a few seconds, the temperature is slowly ramped up to 240 °C, during which time the LAE vaporizes. The temperature is kept at 240 °C for only three seconds before the final quick ramp up to 300 °C. Shortly after the final ramp temperature is reached the split flow is opened again.

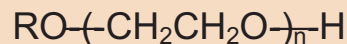
The separation in time of interfering compounds is accomplished by adjusting the temperature-staging parameters. These parameters are used to optimize sensitivity and repeatability.

Results

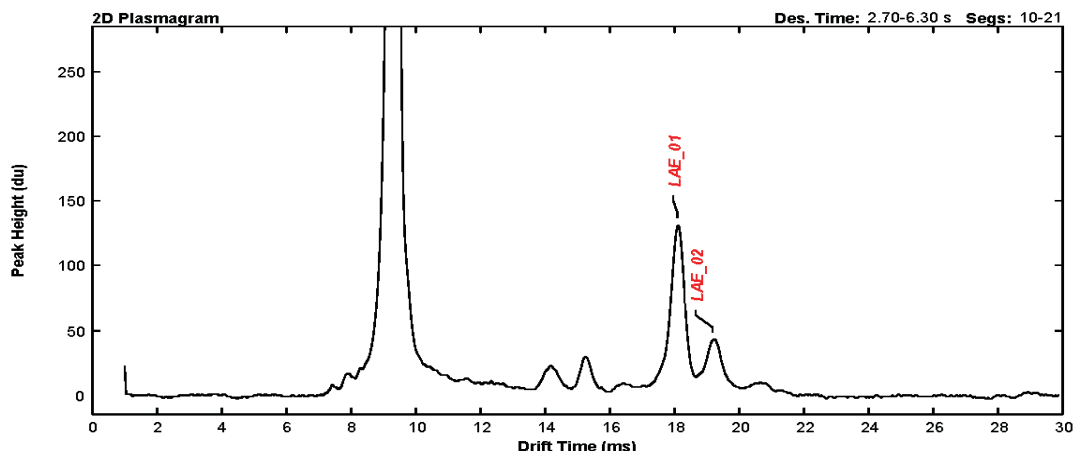
The results are summarized in the following chart. The method yields a limit of detection (LOD) of 1 ng/μL and a limit of quantitation (LOQ) of 2 ng/μL. Based on this experiment, analysts should design their extraction and dilution protocol to target sample concentrations of approximately 8 ng/μL.

CHEMATIC 82AE

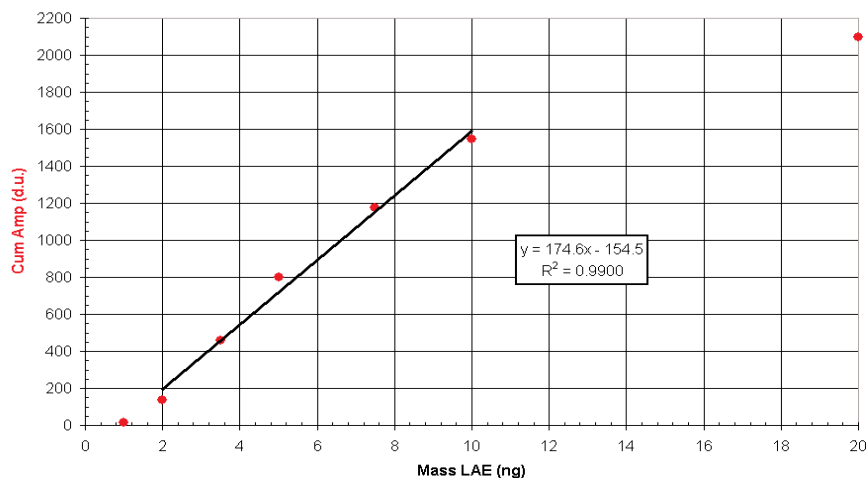
Compound	Chematic 82AE detergent (Dober), contains surfactant LAE
Instrument	IONSCAN-LS, autosampler, HPI
Detection mode	Positive
Ionization reactant	Nicotinamide
Calibrant/ K_0	Nicotinamide/1.8578
Primary std/ K_0	Cocaine/1.1600
Solvent	Water
Sample size	1 μ L
Sample introduction	HPI, cold injection
Analysis time	16 secs
Cycle time	85 sec/sample
Throughput	42 samples/hr
Report number	2005-004-01



Peak ID	K_0	Drift time (ms)	FWHM (μ s)	Operating Temp Drift/Inlet/Desorber
LAE_01	0.9573	18.359	370	229 °C /285°C /285°C



82AE linearity on peak LAE_01



Estimated LOD	1 ng
Estimated LOQ	2 ng
Response function	$y = 175x - 155$ $R^2 = 0.9900$
RSD at 7.5 ng	2-8 %, n = 3

* at concentrations above LOQ