The Analysis of Ethoxylated Surfactants by Supercritical Fluid Chromatography

Introduction

Alcohol ethoxylates are generally made by reacting ethylene oxide with primary alcohols to produce a series of low to high molecular weight, non-ionic surfactants. These polymers can be customized to yield surface-active agents suited to a wide range of applications. The distribution of oligomers having different numbers of ethoxy groups is primarily determined by the relative rates of the initiation and propagation ethoxylation steps. The distribution in the final product impacts the chemical properties and therefore the end-use applications of these surfactants. Consequently, a reliable method for the determination of the distribution of the oligomers is critical for both the producer and the customer. The non-ionic alcohol ethoxylate surfactants are used to emulsify and disperse polymers, waxes and oils and to disperse and suspend solid particles. They are found in formulations of soaps, lubricants, home care products, foods and medications.

The Analysis of the non-ionic surfactant groups has typically been done using HPLC. Because this group of compounds generally lacks chromophores for effective detection by UV, derivatization or other, less sensitive, detectors are required for successful analysis by HPLC techniques. SFC is therefore the method of choice for the ethoxylated alcohols where the universal flame ionization detector (FID) is available. Another added benefit of SFC is the rapid methods development and the reduction in sample preparation time. In this note we demonstrate an analysis of a non-ionic surfactant by SFC without derivitization.

Experimental Conditions

The conditions are shown in Table 1. The chromatographic analysis was performed with a Selerity Technologies Series 3000 SFC equipped with split/splitless injector and a flame ionization detector.

Table 1: SFC Conditions

Oven Program	Isothermal at 125°C
Pump Pressure	125 atm hold, 6 minutes, ramp to 400 atm @ 10 atm/min
Column	SB-Biphenyl-30, 5-m, 50µm ID x 195µm OD
Detector Temperature	FID at 400°C
Restrictor	Frit
Injection	Split/splitless, split opens 1.00 min after injection

Results and Discussion

Figure 1 shows the results of the chromatographic analysis of anon-ionic surfactant, Triton X-100, by supercritical fluid chromatography. As can be seen from the chromatogram, SFC provides a high resolution separation of the oligomers and isomers, and is capable of eluting ethoxylated alcohols, with high resolution, for oligomers which have molecular weights of approximately 5000.

Figure 1. Triton X-100 analyzed by SFC