

THE ANALYSIS OF POLYETHYLENE GLYCOL BY SUPERCRITICAL FLUID CHROMATOGRAPHY

INTRODUCTION

The physical properties of supercritical fluids give supercritical fluid chromatography (SFC) several advantages over other chromatographic techniques including gas chromatography (GC) and high performance liquid chromatography (HPLC) for the analysis of polymers. SFC uses a mobile phase which possesses densities similar to those of a liquid and viscosities and diffusivities closer to those of gases; therefore, is capable of providing rapid, efficient, and low temperature analyses of high molecular weight samples. Another advantage of SFC is the wide range of detector compatibility. This technique can be interfaced to virtually all HPLC and GC detectors. The “universal” flame ionization detector (FID) is among the most popular and is particularly beneficial for polymers due to their lack of chromophores.

Polyglycols are widely used in the chemical industry. They have a diverse range of applications which are dependent upon their molecular weights. Some of their most popular uses are as plasticizers, softeners, paper coating, polishers, binders and bases for cosmetics and pharmaceuticals. They are also used for agricultural and food formulations, plating baths, cutting oils, and starting materials for urethane synthesis. With the large economic investment in this single group of polymers, a reliable means of analysis is essential. These compounds are often difficult to analyze by GC and HPLC due to their high molecular weights and lack of chromophores. In this note we used an underivatized Carbowax PEG 600 to demonstrate the SFC analysis of polyglycols.

EXPERIMENTAL CONDITIONS

The analysis conditions are shown in Table 1. The chromatographic analyses 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 150°C |
| PUMP PRESSURE | 100 ATM HOLD, 10 MINUTES, RAMP TO 400 ATM @ 1.5 ATM/MIN |
| COLUMN | SB-BIPHENYL-30, 5-M, 50µM ID X 1.95µM 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 a polyethylene glycol standard performed with supercritical fluid chromatography. As can be seen from the chromatogram, SFC provides a high resolution separation of all of the oligomers without the added effort of derivatization. This technique is capable of obtaining PEG separation up to a molecular weight of 1500 Daltons.

FIGURE 1. CARBOWAX 600 ANALYZED BY SFC

