



## THE ANALYSIS OF FLUORINATED POLYMERS BY SUPERCRITICAL FLUID CHROMATOGRAPHY (SFC)

### INTRODUCTION

The analysis of fluorinated polymers has proven to be very difficult by all other methods of chromatography other than SFC. The difficulty is due to their insolubility with common solvent for HPLC analysis and their nonvolatility for GC analysis.

Fluorocarbons are available in a wide range of molecular weight ranges to meet the specific lubricating requirements of the computer industry and for many aircraft components that must operate over extreme temperature ranges. To maintain consistent operation in these high performance applications, vendors are increasingly being asked to provide well characterized fractions within a well controlled molecular weight range.

This note provides analyses of fluorinated hydrocarbons on both packed and capillary columns developed specifically for SFC. The columns are optimized for use under supercritical fluid chromatographic conditions where carbon dioxide is used as the mobile phase material.

### EXPERIMENTAL CONDITIONS

The analysis conditions for Polymethyl-333-trifluoropropylsiloxane is in Table 1 and the conditions for the Krytox® analysis is shown in Table 2. All of the chromatographic analyses were performed with a Selerity Technologies' Series 3000 SFC equipped with a flame ionization detector.

### RESULTS

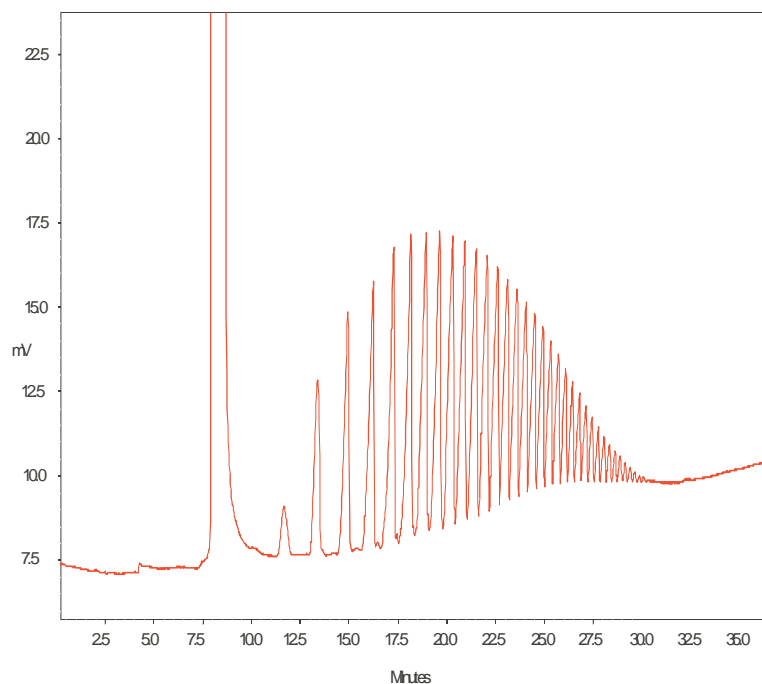
Figure 1 shows a high resolution separation of a fluorinated polymer, Polymethyl-333-trifluoropropylsiloxane analysis on a SB-Biphenyl-30 capillary column. This SFC separation was developed in just a few hours. The separations of two samples from the Krytox® family of fluorocarbons were also performed. Figure 2 shows an overlay of the two compounds and the distinction between the two different molecular weights of the fluorocarbon samples.

TABLE 1: SFC CONDITIONS FOR POLYMETHYL-333-TRIFLUOROPROPYLSILOXANE	
OVEN PROGRAM	100°C HOLD 15 MINUTES, RAMP TO 125°C @ 2°C/MIN
PUMP PRESSURE	100 ATM HOLD 10 MINUTES, RAMP TO 400 ATM @ 7ATM/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

TABLE 2: SFC CONDITIONS FOR KRYTOX®	
OVEN PROGRAM	ISOTHERMAL AT 150°C
PUMP PRESSURE	100 ATM HOLD, 10 MINUTES, RAMP 1 TO 150 ATM @ 7.5ATM/MIN, RAMP2 TO 210 ATM@2.5 ATM/MIN, RAMP3 TO 325 ATM @1.5 ATM/MIN
COLUMN	FLUOROPHASE IMM ID X 50CM OD
DETECTOR TEMPERATURE	FID AT 425°C
RESTRICTOR	FRIT
INJECTION	TIMED SPLIT, 3 SEC



**FIGURE 1: SFC ANALYSIS OF POLYMETHYL-333-TRIFLUOROPROPYLSILOXANE**



**FIGURE 2: THE OVERLAY OF TWO KRYTOX® SAMPLES PERFORMED ON A PACKED FLUOROPHASE COLUMN**

