

SIMULTANEOUS DETERMINATION OF POLYMER MOLECULAR WEIGHT DISTRIBUTION AND ANALYSIS OF ADDITIVES WITH ULTRASTYRAGEL™ COLUMNS

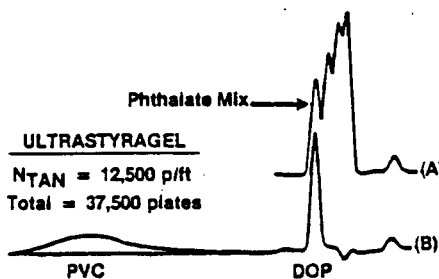
Tygon® tubing, a standard in laboratories throughout the world, consists of a high molecular weight polymer (polyvinyl chloride, PVC) to which has been added a low molecular weight plasticizer (dioctyl phthalate, DOP) which imparts flexibility to the product. Using high efficiency ULTRASTYRAGEL columns (10^3 , 10^4 , 10^5\AA) optimized for the determination of the molecular weight distribution (MWD) of the PVC portion of the Tygon tubing, one can simultaneously obtain enough resolution of a mixture of low molecular weight phthalates to identify the plasticizer by a comparison of retention times. Previously, with lower efficiency columns, resolution of additives was only marginal when high \AA columns were used. Typically, a second analysis was required on a set of low \AA columns to obtain satisfactory resolution in the low molecular weight additive region.

In the example below, a sample of Tygon tubing was analyzed on each of two sets of GPC columns. One set consisted of 10^3 , 10^4 , and 10^5\AA ULTRASTYRAGEL columns (12,500 plates/ft) and the other consisted of the corresponding μ STYRAGEL™ columns (5,200 plates/ft). With both sets of columns, GPC analysis of the Tygon tubing generated the MWD of the high molecular weight PVC as well as a single peak at the end of the run. This single peak represents the phthalate plasticizer. A comparison of Figures #1B and #2B indicates that both phthalate peaks are quite similar except that the peak is significantly narrower with ULTRASTYRAGEL columns (Figure #1B) than with μ STYRAGEL columns (Figure #2B).

A mixture of four phthalates (dioctyl-, dibutyl-, diethyl-, and dimethyl phthalate) was injected in an effort to identify the additive by a comparison of retention times. With the μ STYRAGEL columns (Figure #2A) the phthalate mixture is not resolved. This is not unusual since a set of 10^3 , 10^4 and 10^5\AA columns has only a few small pores. However, since the ULTRASTYRAGEL columns are much more efficient than μ STYRAGEL columns, these few pores are now able to afford partial resolution of all four phthalates (Figure #1A). From a simple comparison of Figures #1A and #1B one can easily see that dioctyl phthalate elutes with the same retention time as the additive in the Tygon sample.

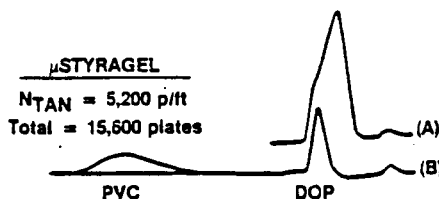
ULTRASTYRAGEL™ vs. μ STYRAGEL™ Columns Tygon® Tubing: MWD and Additive

Figure 1



COLUMNS: 10^3 , 10^4 , 10^5\AA
SAMPLE: A) Phthalate Mix
(DOP, DBP, DEP, DMP)
(0.3% in THF).
B) Tygon tubing
(0.3% in THF)

Figure 2



INJECTION VOLUME: 150 μ l
FLOW RATE: 1 ml/min
MOBILE PHASE: THF
DETECTOR: RI, 8X

Juris Ekmanis