Separation Science and Technology
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713708471

An Observation of a Field-Flow Fractionation Effect with Polystyrene Samples

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To cite this Article Thompson, Gary H. , Myers, Marcus N. and Giddings, J. Calvin(1967) 'An Observation of a Field-Flow Fractionation Effect with Polystyrene Samples', Separation Science and Technology, 2: 6, 797 — 800
To link to this Article: DOI: 10.1080/01496396708049739
URL: http://dx.doi.org/10.1080/01496396708049739

SEPARATION SCIENCE, 2(6), pp. 797–800, Dec., 1967

COMMUNICATION

An Observation of a Field-Flow Fractionation Effect with Polystyrene Samples

Field-flow fractionation (FFF) is a proposed separation method in which some kind of lateral field is coupled with nonuniform axial flow to induce differential migration (*1*). Fractionation is here illustrated by applying a temperature drop laterally across a narrow tube within which a mixture is flowing. In this case thermal diffusion is responsible for lateral enrichment. This variation of the general concept may be termed thermal field-flow fractionation.

The preliminary apparatus consists of two aluminum cylinders, approximately 14 in. in diameter and 2 in. high. One cylinder is solid, the other milled out to permit the flow of cooling water. A length of capillary tubing was placed between the two cylinders and clamped down to provide good contact. In this instance the capillary tube was Teflon of 0.56 mm I.D., 55.5 m long. The solid cylinder, placed on top, is heated by an ordinary electric stove element. The carrier liquid is forced through the capillary by a gravity flow apparatus and the effluent is led to a refractometer detector.

The polystyrene-toluene system was chosen for this study. Polystyrene samples were injected into the capillary tube by means of a microliter syringe. These samples, roughly 2 mg each, consisted of 3525 molecular weight polystyrene, 411,000 molecular weight polystyrene, and a mixture of the two.

The degree of retention of the polystyrene fractions has been recorded in terms of the reduced volume ratio:

$$V_R = (V - V_D)/V_C$$

where V_R = reduced volume, V = volume required to elute sample peak, V_D = end volumes external to the actual separation column, and V_C = separation column volume. A departure from $V_R = 1$ indicates the action of sorptive or FFF effects.

The figures show recorder response (concentration) vs. reduced volume for various polystyrene fractions. Figure 1 shows three experimental curves, two for the component samples (dashed lines) and one for a mixture of the two (solid line). A temperature drop of 55°C was imposed for the three runs (75°C at hot wall, 20°C at cold). Retention of the components as well as partial separation of the mixture is shown.

To show that the separation is not primarily chromatographic (sorptive) in origin, experiments without the thermal gradient were executed. The two single peaks in Fig. 2 are the elution curves for mixtures run without temperature gradient. These individually are near the two extreme temperatures of the thermal gradient system. The higher peak represents $T_H = T_C = 75°C$, the other $T_H = T_C = 25°$. The slight retention of the latter may be due to polystyrene adsorption on the capillary wall. This effect seems to be fairly small and apparently is not able to cause separation without the gradient.

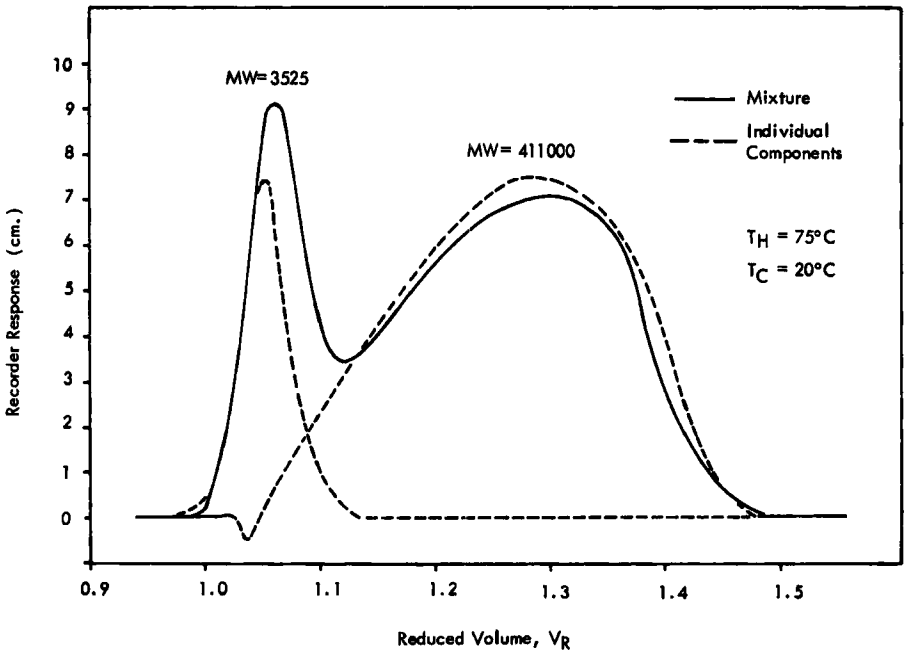

FIG. 1. Comparison of elution peaks for individual components with that for a mixture.

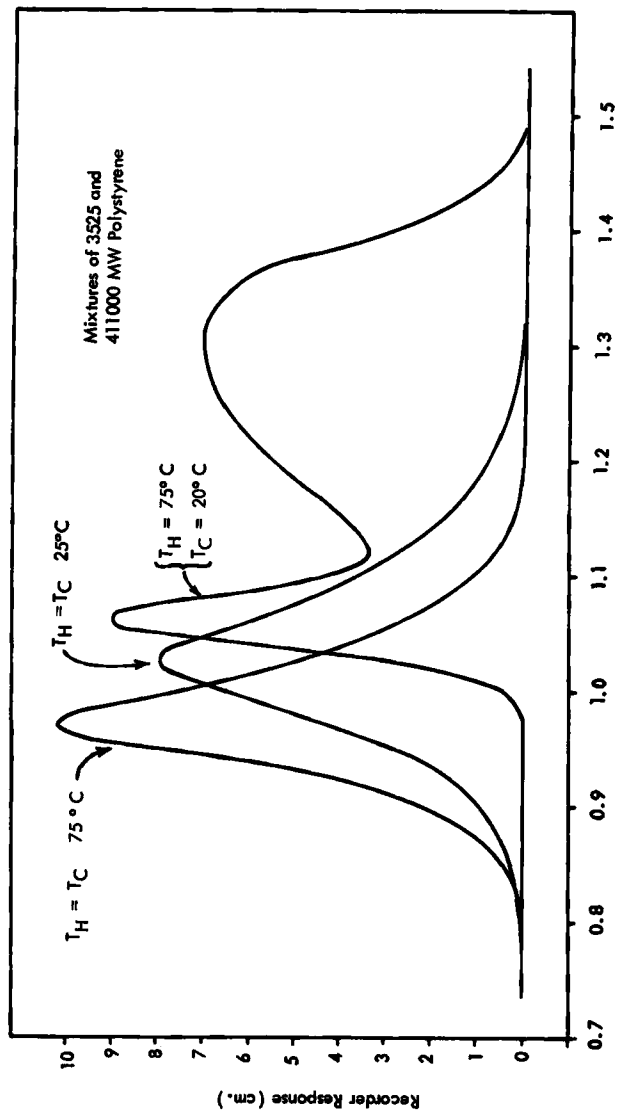

FIG. 2. Comparison of mixture elution peaks at temperature extremes with that produced in a thermal gradient.

Although the components have not been completely resolved, it is apparent that the method is workable. Our most recent results show considerable disturbance in the streamlined flow pattern even at very low flow velocities. This is presumably responsible for the failure to achieve better resolution. We should expect considerably better separation when these disturbances are removed.

Acknowledgment

Financial assistance was received from the University of Utah Research Fund.

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Received by editor November 27, 1967
Submitted for publication November 28, 1967