

CHROM. 17 630

## Note

### Determination of polymer molecular weights by paper chromatography

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The characteristics of synthetic polymers depend to a large extent on their molecular weight and molecular weight distribution (MWD). Much effort, therefore, has been made to determine these important parameters. Classical techniques which have been used extensively for this purpose, however, are time-consuming, tedious and often give unreliable data<sup>1,2</sup>. The advent of gel permeation chromatography has facilitated the rapid and accurate determination of the MWD and molecular weight averages of polymers<sup>3</sup>. Techniques such as thin-layer chromatography and gradient liquid chromatography have also been used effectively for the fractionation and subsequent determination of the MWD and molecular weights of a variety of polymers<sup>4-6</sup>.

I have attempted a novel way of obtaining molecular weights by using paper chromatography in conjunction with a binary mobile phase. The preliminary results indicate that this simple method is capable of giving information on the molecular heterogeneity of synthetic polymers.

#### EXPERIMENTAL

Whatman No. 1 filter-paper was used for the chromatographic separations. Analytical grade methanol and chloroform (Glaxo Labs., India) were distilled prior to use. Iodine (SD Chemicals, India) was used as received. Polystyrene standards were from Waters Assoc. (Milford, MA, U.S.A.) (Table I).

TABLE I  
POLYSTYRENE STANDARDS

<i>Molecular weight</i>	<i>R<sub>F</sub></i>
350 000	0.160
217 600	0.197
111 000	0.308
51 150	0.419
9168	0.716

The polystyrene standards were dissolved in chloroform (1 mg/ml). A polystyrene sample having a broad molecular weight range served as unknown. About 5  $\mu$ l of the solutions were applied on the filter-paper strips (15  $\times$  3 cm) using a microsyringe. Chromatographic development was carried out in a closed chamber containing chloroform-methanol (50:13) as mobile phase. The development was discontinued when the solvent front had ascended to nearly 10 cm. The dried paper strip was then treated with iodine vapour to visualize the spots.

## RESULTS AND DISCUSSION

Fig. 1a shows the paper chromatogram of five polystyrene standards. The dependence of  $R_F$  (defined as the distance travelled by the solute from the starting line divided by the corresponding distance travelled by solvent front) on the molecular weights of the polystyrene standards is illustrated in Fig. 2.

In a good solvent (here chloroform) the polymer standards, irrespective of their molecular weights, migrated together with the solvent front. Methanol (a non-solvent) and mobile phases with a larger methanol fraction prevented the migration of the samples, resulting in  $R_F = 0$ . The strong influence of the mobile phase composition on  $R_F$  indicates that the separation mechanism operative here, as in thin-layer chromatography<sup>4</sup>, is the selective precipitation of the samples as the mobile phase ascends. However, a detailed study of the mechanism has not been attempted.

Fig. 1b illustrates the chromatogram of the unknown polystyrene sample. The concentration of the sample at a distance corresponding to the polymer standard of molecular weight 51 150 indicates the bulk of the unknown sample comprises species having this molecular weight. This value is in good agreement with that of 53 800

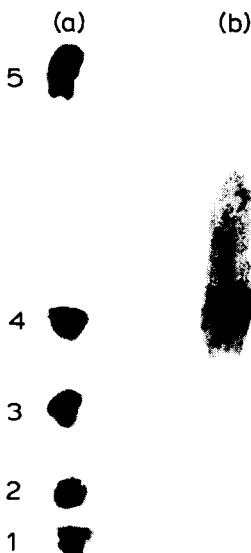


Fig. 1. Paper chromatograms of polystyrene standards (a) and of an unknown polystyrene sample (b). Spots: 1 = molecular weight 350 000; 2 = molecular weight 217 600; 3 = molecular weight 111 000; 4 = molecular weight 51 150; 5 = molecular weight 9168.

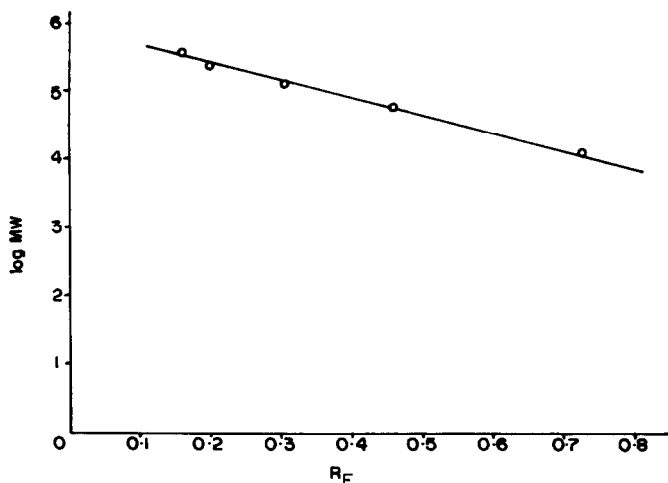


Fig. 2. Plot of  $R_F$  versus log molecular weight (MW) for polystyrene standards.

obtained for this sample in chloroform, by the light scattering method. The tailing of spots towards both the high- and low-molecular-weight regions apparently reveals that the sample has a broad molecular weight range.

The actual distribution curve for this sample could be generated by plotting  $R_F$  or molecular weight versus the intensity of the chromatogram at various points. However, this has not been attempted.

The merits and demerits of the reported method can be assessed only by extending its applicability to other polymers. The immediate outcome is of course the nominal cost of the method in comparison with other chromatographic techniques.

Paper chromatography is acknowledged as a powerful separation technique. Though on the decline, due to the advent of much more sophisticated separation methods, it is simple, cheap and able to separate a wide spectrum of substances. The present communication indicates that paper chromatography can also be extended to provide information on the most fundamental parameters of synthetic macromolecules.

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