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Standard Practice for Use of Liquid Exclusion Chromatography Terms and Relationships[1]

This standard is issued under the fixed designation D3016; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

Liquid exclusion chromatography (LEC) began as "gel filtration chromatography" which is attributed to Porath and Flodin.[2] With the invention of new column packings by Moore[3] for organic-phase work a new form of LEC developed which commonly became known as gel permeation chromatography or GPC. Liquid exclusion chromatography is a form of liquid chromatography (some other forms being partition, ion-exchange, and adsorption) and as such is the preferred name for the technique; however, the reader must be aware that other names are common in the literature, the most prevalent being those cited above. LEC differs from all other chromatographic techniques in that only the exclusion mechanism may be operative if meaningful data are to result. Most other chromatographic mechanisms operate in essentially the opposite way, that is, with small molecules exiting first. Any combination of mechanisms causes confusion and is misleading.

Liquid exclusion chromatography as used for the analysis of polymers has grown and matured since the first issuance of this practice in 1972. Therefore, some infrequently used or "outdated" terms have been deleted and some modern practices (or terms) have been included. Modern developments include the use of constant-volume pumps, use of "microparticle" column packings and much smaller columns, and automated data-handling procedures. In addition, SI units as recommended in ASTM Standard IEEE/ASTM SI 10 for Metric Practice are now used.

1. Scope

1.1 This practice covers the definitions of terms and symbols most often used in liquid exclusion chromatography. Wherever possible, these terms and symbols are consistent with those used in other chromatographic techniques.[4] As additional terms and relationships are developed, they will be incorporated.

NOTE 1—There is no known ISO equivalent to this standard.

2. Referenced Documents

2.1 *ASTM Standards:*

IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI): The Modern Metric System[5]

3. Apparatus Definitions

3.1 *absolute detectors*—devices that sense and measure the absolute concentration or other physical property of solute components contained in the eluate.

3.2 *by-pass or loop injector*—the injector most common in liquid exclusion chromatography and which utilizes a sample chamber that can be filled with sample while it is temporarily external to the flowing liquid stream. It can be manipulated by means of a valving device to sweep the sample with eluent into the column.

3.3 *collection devices*—devices used to collect discrete portions of an eluate according to a preset cycle (for example, times, volume, etc.).

[1] This practice is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods.

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[2] Porath, J., and Flodin, P., *Nature*, NTRWA, Vol 183, 1959, p. 1657.

[3] Moore, J. C., *Journal of Polymer Science*, JPYAA, Part A, Vol 2, 1964, p. 835.

[4] *Journal of Gas Chromatography*, JCHSB, Vol 66, 1968.

[5] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3.4 *column end-fittings*—devices that prevent the column packing from passing through them but which are permeable to the eluent (solvent or solution).

3.5 *columns*—tubes that contain the column packing.

3.6 *detectors*—devices that sense and measure the concentration or other physical property of solute components in the solution (eluate) passing through.

3.7 *differential detectors*—devices that sense and measure the difference in a physical or chemical property between a solution (solvent containing solute components) and a reference liquid (for example, solvent alone).

3.8 *direct injector*—a device for introducing a sample from a source external to the column directly onto the column (for example, septum-syringe injector).

3.9 *pump*—any device that causes mobile phase to flow through the columns.

3.10 *sample inlet system*—a means of introducing samples onto the column.

4. Reagent Definitions

4.1 *column packing*—the stationary phase which consists of microporous material and the stationary liquid phase contained in the pores.

4.2 *eluate*—the liquid emerging from the column.

4.3 *eluent*—the mobile phase or solvent used to sweep or elute the sample (solute) components into, through, and out of the column. Its composition is the same as the stationary liquid phase.

4.4 *solutes*—dissolved substances that, in LEC, are caused to pass through the column and to influence the detector response.

5. Performance in Terms of Resolution, Column Efficiency, and Precision

5.1 Resolution is the resultant of two effects, the separating power of the column packing and the efficiency or peak broadening. The separating power of the column packing is dependent on pore size and pore volume. Peak broadening depends on the nature of the column packings, on how well the columns are packed, and on instrumental components external to the columns. The equations used in LEC (GPC) are similar to those used in other chromatographic techniques. Reference may be made to any standard chromatography text. Resolution for any two samples is defined by the following equation:

$$R_{1,2} = 2[(V_{R_1} - V_{R_2}) / (W_1 + W_2) \quad (1)$$

where subscripts 1 and 2 refer to samples 1 and 2. (See tables for symbols used in this equation.) For complete separation, $R_{1,2}$ must be 1.25 or greater.

5.2 Column efficiency is a measure of peak spreading or the rate of generation of variance with column length. For a monodisperse material, efficiency is the number of theoretical plates, *N*, for the entire system defined as follows: (See tables for symbols used in this equation.)

$$N = 16(V_R/W)^2 \text{ or } \sigma = N/V_R \quad (2)$$

This expression includes all contributions to peak broadening.

5.3 Precision and accuracy are used according to their accepted definitions. Precision is inherent to the system. Both precision and accuracy are dependent on the method of calibration and treatment of the data as well as on the resolving power of the columns. The accuracy must be determined by comparison with other methods. For example, the molecular-weight distribution can be compared with that obtained from equilibrium ultracentrifugation. More commonly the weight- and number-average molecular weights computed from the LEC (GPC) trace are compared with those measured by light scattering, (both static and on-line), osmometry, and on-line viscometry.

6. Readout Definitions

6.1 *chromatogram*—a plot of detector response against volume of eluate emerging from the system. An idealized chromatogram obtained with a detector providing differential response is shown in Fig. 1.

6.2 The definitions that follow apply to chromatograms obtained directly by means of differential detectors:

6.2.1 *baseline*—the portion of the chromatogram recording the detector response when only eluent emerges from the column.

6.2.2 *half width, GH*—the retention volume interval of a line parallel to the peak base, bisecting the peak height, and terminating at the sides of the peak.

6.2.3 *peak*—the portion of the chromatogram recording the detector response while a single component or a single distribution (two or more sample components that emerge together as a single peak) is eluted from the column.

6.2.4 *peak area, BGDHCAB*—the area enclosed between the peak and the peak base.

6.2.5 *peak base, BC*—an interpolation of the baseline between extremities of the peak.

6.2.6 *peak height, AD*—the dimension from the peak maximum to the base measured in the direction of detector response.

6.2.7 *peak width, EF*—the retention volume interval of the segment of peak base intercepted by tangents to the inflection points on either side of the peak.

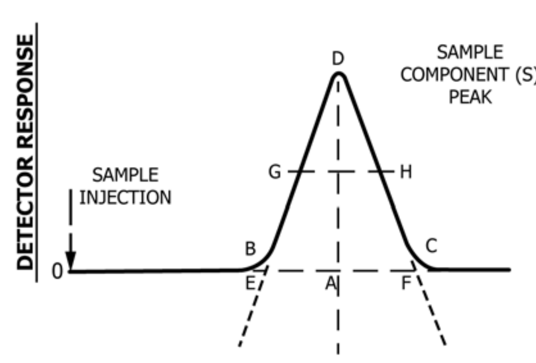

FIG. 1 Typical Chromatogram