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Standard Practice for Ion Chromatography Terms and Relationships[1]

This standard is issued under the fixed designation E 1151; 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.

1. Scope

1.1 This practice deals primarily with identifying the terms and relationships of those techniques that use ion exchange chromatography to separate mixtures and a conductivity detector to detect the separated components. However, most of the terms should also apply to ion chromatographic techniques that employ other separation and detection mechanisms.

1.2 Because ion chromatography is a liquid chromatographic technique, this practice uses, whenever possible the terms and relationships identified in Practice E 682.

1.3 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 682 Practice for Liquid Chromatography Terms and Relationships[2]

3. Descriptions of Techniques

3.1 *Ion Chromatography, (IC)*—a general term for several liquid column chromatographic techniques for the analysis of ionic or ionizable compounds. Of the many useful separation and detection schemes, those most widely used have been the two techniques described in 3.2 and 3.3 in which ion exchange separation is combined with conductimetric detection. By describing only these two techniques, this practice does not mean to imply that IC is tied only to ion exchange chromatography or conductimetric detection.

3.2 *Chemically Suppressed Ion Chromatography, (Dual Column Ion Chromatography)*—In this technique, sample components are separated on a low capacity ion exchanger and detected conductimetrically. Detection of the analyte ions is enhanced by selectively suppressing the conductivity of the mobile phase through post separation ion exchange reactions.

3.3 *Single Column Ion Chromatography, (Electronically Suppressed Ion Chromatography)*—In this technique sample components are separated on a low capacity ion exchanger and detected conductimetrically. Generally, lower capacity ion exchangers are used with electronic suppression than with chemical suppression. Mobile phases with ionic equivalent conductance significantly different from that of the sample ions and a low electrolytic conductivity are used, permitting analyte ion detection with only electronic suppression of the baseline conductivity signal.

4. Apparatus

4.1 *Pumps*—Any of various machines that deliver the mobile phase at a controlled flow rate through the chromatographic system.

4.1.1 *Syringe Pumps*, having a piston that advances at a controlled rate within a cylinder to displace the mobile phase.

4.1.2 *Reciprocating Pumps*, having one or more chambers from which mobile phase is displaced by reciprocating piston(s) or diaphragm(s). The chamber volume is normally small compared to the volume of the column.

4.1.3 *Pneumatic Pumps*, employing a gas to displace the mobile phase either directly from a pressurized container or indirectly through a piston or collapsible container. The volume within these pumps is normally large as compared to the volume of the column.

4.2 *Sample Inlet Systems*, devices for introducing samples into the column.

4.2.1 *Septum Injectors*—The sample contained in a syringe is introduced directly into the pressurized flowing mobile phase by piercing an elastomeric barrier with a needle attached to a syringe. The syringe is exposed to pressure and defines the sample volume.

4.2.2 *Valve Injectors*—The sample contained in a syringe (or contained in a sample vial) is injected into (or drawn into) an ambient-pressure chamber through which the pressurized flowing mobile phase is subsequently diverted, after sealing against ambient pressure. The displacement is by means of rotary or sliding motion. The chamber is a section (loop) of tubing or an internal chamber. The chamber can be completely

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[1] This practice is under the jurisdiction of ASTM Committee E13 on Molecular Spectroscopy and is the direct responsibility of Subcommittee E13.19 on Chromatography.

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[2] *Annual Book of ASTM Standards,* Vol 14.02.

filled, in which case the chamber volume defines the sample volume, or it can be partially filled, in which case the syringe calibration marks define the sample volume.

4.3 *Columns*, tubes, containing a stationary phase and through which the mobile phase can flow.

4.3.1 *Precolumns*, positioned before the sample inlet system and used to condition the mobile phase.

4.3.2 *Concentrator Columns*, installed in place of the sample chamber of a valve injector and used to concentrate selected sample components.

4.3.3 *Guard Columns*, positioned between the sample inlet system and the separating columns and used to protect the separator column from harmful sample components.

4.3.4 *Separating Columns*, positioned after the sample inlet system and the guard column and used to separate the sample components.

4.3.5 *Suppressor Columns*, positioned after the separating column and a type of post column reactor where the conductivity of the mobile phase is selectively reduced to enhance sample detection.

4.4 *Postcolumn Reactors*, reaction systems in which the effluent from the separating columns is chemically or physically treated to enhance the detectability of the sample components.

4.4.1 *Conductivity Suppressors*, post column reactors in which the conductivity of the mobile phase is reduced through reactions with ion exchangers. Conductivity suppressors are differentiated by their type (cationic or anionic), by their form (H^+, Na^+, etc.), and by their method of regeneration (batch or continuous).

4.4.2 *Suppressor Columns*—Tubular reactors packed with ion exchangers. Suppressor columns require batch regeneration when the breakthrough capacity of the column is exceeded.

4.4.3 *Membrane Suppressors*—Reactors made from tubular shaped ion exchange membranes. On the inside of the tube flows the mobile phase; a regenerative solution surrounds the tube. These membrane suppressors can be in the form of an opened tube, hollow fiber suppressors, or a flattened tube for higher capacity. Tubular membranes can be packed with inert materials to reduce band broadening.

4.4.4 *Micromembrane Suppressor*—Reactors made from two sizes of ion-exchange screen. A fine screen is used for the mobile phase chamber and a coarse screen is used for the regenerant chambers. The mobile phase screen is sandwiched between ion-exchange membranes, and on either side of each membrane is a regenerant screen. The stack is laminated by pressure, causing intimate contact between screens and membranes. Mobile phase passes through a hole in the upper regenerant screen and membrane. It enters the screen-filled mobile phase chamber and passes through it. It then exits through a second set of holes in the upper membrane and regenerant screen. The regenerant flows countercurrent to the mobile phase through the screen-filled regenerant chamber.

4.5 *Detectors*—Devices that respond to the presence of eluted sample components. Detectors may be divided either according to the type of measurement or the principle of detection.

4.5.1 *Bulk Property Detectors*, measuring the change in a physical property of the liquid phase exiting the column. Thus a change in the refractive index, conductivity, or dielectric constant of a mobile phase can indicate the presence of eluting sample components. Conductimetric parameters, symbols, units and definitions are given in Appendix X1.

4.5.2 *Solute Property Detectors*, measuring the physical or chemical characteristics of eluting sample components. Thus, light absorption (ultraviolet, visible, infrared), fluorescence, and polarography are examples of detectors capable of responding in such a manner.

5. Reagents

5.1 *Mobile Phase*—Liquid used to sweep or elute the sample components through the chromatographic system. It may consist of a single component or a mixture of components.

5.2 *Stationary Phase*—Active immobile material within the column that delays the passage of sample components by one of a number of processes or their combination. Inert materials that merely provide physical support for the stationary phase are not part of the stationary phase. The following are three types of stationary phase:

5.2.1 *Liquid Phase*—A stationary phase that has been sorbed (but not covalently bonded) to a solid support. Differences in the solubilities of the sample components in the liquid and mobile phase constitute the basis for their separation.

5.2.2 *Interactive Solid*—A stationary phase that comprises a relatively homogeneous surface on which the sample components sorb and desorb effecting a separation. Examples are silica, alumina, graphite, and ion exchangers. In ion chromatography the interactive material is usually an ion exchanger that has ionic groups that are either ionized or capable of dissociation into fixed ions and mobile counter-ions. Mobile ionic species in an ion exchanger with a charge of the same sign as the fixed ions are termed "co-ions." An ion exchanger with cations as counter-ions is termed a "cation exchanger," and an ion exchanger with anions as counter-ions is termed an "anion exchanger." The ionic form of an ion exchanger is determined by the counter-ion, for example, if the counter-ions are hydrogen ions then the cation exchanger is in the acid form or hydrogen form, or if the counter-ions are hydroxide ions then the anion exchanger is in the base form or hydroxide form. Ionic groups can be covalently bonded to organic polymers (for example, styrene/divinylbenzene) or an inorganic material (for example, silica gel). Ion exchange parameters, symbols, units and definitions are given in Appendix X2. Separation mechanisms on ion exchangers are described in Appendix X3.

5.2.3 *Bonded Phase*—A stationary phase that comprises a chemical (or chemicals) that has been covalently attached to a solid support. The sample components sorb onto and off the bonded phase differentially to effect separation. Octadecylsilyl groups bonded to silica represent a typical example for a bonded phase.

5.3 *Solid Support*—Inert material to which the stationary phase is sorbed (liquid phases) or covalently attached (bonded phases). It holds the stationary phase in contact with the mobile phase.

5.4 *Column Packing*—The column packing consists of all the material used to fill packed columns. The two types are as follows:

5.4.1 *Totally Porous Packing*—One where the stationary phase is found throughout each porous particle.

5.4.2 *Pellicular Packing*—One where the stationary phase is found only on the porous outer shell of the otherwise impermeable particle. Surface agglomerated packings are considered to be a type of pellicular packing.

6. Readout

6.1 *Chromatogram*—Graphic representation of the detector response versus retention time or retention volume as the sample components elute from the column(s) and through the detector. An idealized chromatogram of an unretained and a retained component is shown in Fig. X1.1.

6.2 *Baseline*—Portion of a chromatogram recording the detector response when only the mobile phase emerges from the column.

6.3 *Peak*—Portion of a chromatogram recording detector response when a single component, or two or more unresolved components, elute from the column.

6.4 *Peak Base (CD in* Fig. X1.1*)*—Interpolation of the baseline between the extremities of a peak.

6.5 *Peak Area (CHFEGJD in* Fig. X1.1*)*—Area enclosed between the peak and the peak base.

6.6 *Peak Height (EB in* Fig. X1.1*)*—Distance measured in the direction of detector response, from the peak base to peak maximum.

6.7 *Peak Widths*—Represent retention dimensions parallel to the baseline. Peak width at base or base width, (KL in Fig. X1.1) is the retention dimension of the peak base intercepted by the tangents drawn to the inflection points on both sides of the peak. Peak width at half height, (HJ in Fig. X1.1) is the retention dimension drawn at 50 % of peak height parallel to the peak base. The peak width at inflection points, (FG in Fig. X1.1), is the retention dimension drawn at the inflection points (= 60.7 % of peak height) parallel to the peak base.

7. Retention Parameters, Symbols, and Units

7.1 Retention parameters, symbols, units, and their definitions or relationship to other parameters are listed in Table X3.1.

NOTE 1—The adjusted retention time, capacity ratio, number of theoretical plates, and relative retention times are exactly true only in an isocratic, constant-flow system yielding perfectly Gaussian peak shapes.

7.2 Fig. X1.1 can be used to illustrate some of the following most common parameters measured from chromatograms:

Retention time of unretained component, $t_M = OA$
Retention time, $t_R = OB$
Adjusted retention time, $t_R = AB$
Capacity factor, $k' = (OB - OA)/OA$
Peak width at base, $w_b = KL$
Peak width at half height, $w_h = HJ$
Peak width at inflection points, $= FG = 0.607(EB)$
Number of theoretical plates, $N = 16[(OB)/(KL)]^2 = 5.54[(OB)/(HJ)]^2$
Relative retention, r (Note 2) $= (AB)_i/(AB)_s$
Peak resolution, R_s(Note 2 and Note 3) $= 2[(OB)_j - (OB)_i]/(KL)_i + (KL)_j \simeq (OB)_j - (OB)_i/(KL)_j$

NOTE 2—Subscripts *i*, *j*, and *s* refer to some peak, a following peak, and a reference peak (standard), respectively.

NOTE 3—The second fraction may be used if peak resolution of two closely spaced peaks is expressed; in such as case $(KL)_i = (KL)_j$.

APPENDIXES

(Nonmandatory Information)

X1. Separation Mechanisms

X1.1 *Ion Exchange Chromatography*—Sample and mobile counter-ions compete to form neutral ion pairs with the fixed ions of an ion exchanger. When paired, the sample ions do not move through the ion exchange column. Separation is achieved because the fixed ions have different thermodynamic complexation constants resulting in chromatographic selectivity between ions.

TABLE X1.1 Conductometric Parameters

Parameter	Symbol	Unit[A]	Definition or Relation to Other Parameters
Conductance		S	The reciprocal of a measured resistance
Electrolytic conductivity	κ	$S\cdot cm^{-1}$	The reciprocal of the resistance of a 1-cm cube of liquid at a specified temperature.
Equivalent conductivity	Λ	$S\cdot cm^2\cdot equivalents^{-1}$	$\Lambda = \kappa/C$, where C is the total concentration (equivalents/cm^3) of positive or negative charge produced on dissociation of an electrolyte.
Ionic equivalent conductivity	λ	$S\cdot cm^2\cdot equivalents^{-1}$	The contribution of an individual ion to the equivalent conductivity of an electrolyte, for example, $\Lambda = \lambda_c + \lambda_a$, where λ_c is the ionic equivalent conductance of the cations and λ_a is the ionic equivalent conductance of the anions of an electrolyte.
Cell constant	θ	cm^{-1}	$\theta = \kappa R\ R_{solv}/R_{solv} - R$ R is the resistance measured when the cell is filled with a standard electrolyte solution and R_{solv} is the resistance when the cell is filled with solvent at the same temperature.

[A] The SI unit siemens (S) was formerly called mho (Ω^{-1}).