**ASTM** Designation: E 1511 – 93

# Standard Practice for
# Testing Conductivity Detectors Used in Liquid and Ion Chromatography[1]

This standard is issued under the fixed designation E 1511; 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 covers the testing of the performance of conductivity detectors used as the detection component of a liquid or ion chromatography system.

1.2 The values stated in SI units are to be regarded as the standard.

## 2. Referenced Documents

2.1 *ASTM Standards:*

E 1151 Practice for Ion Chromatography Terms and Relationships[2]

## 3. Terminology

3.1 See Practice E 1151.

3.2 *Definitions:*

3.2.1 *cell constant*—the cell constant ($K$) of a conductivity cell is equal to $1/A$, so $\kappa = G\,K$.

3.2.1.1 *Discussion*—If the cell constant of the flow-through cell used is equal to one, then the conductivity equals the conductance. Although the cell constant is often specified for conductivity detectors, there is little practical value in knowing the constant as long as the detector is properly calibrated for conductivity.

3.2.2 *conductance*—the conductance ($G$) of a solution is the inverse of the resistance measured between two electrodes in a cell, expressed in units of siemens (S), equal to inverse ohms.

3.2.2.1 *Discussion*—The term resistance refers specifically to the dc resistance to ionic current, independent of the capacitive reactance at the interfaces between the electrodes and the solution.

3.2.3 *conductivity*—since the conductance is dependent on both the conductive properties of the solution and on the dimensions of the electrodes and the cell, the conductivity ($\kappa$) of the solution is defined to be independent of electrode and cell dimensions. Specifically,

$$\kappa = G\,\frac{1}{A} \qquad (1)$$

where 1 is the distance between two planer disk electrodes and $A$ is the electrode's surface area.

3.2.3.1 *Discussion*—In liquid and ion chromatography, cell dimensions are commonly measured in centimetres, so the units of κ are S/cm. (Alternatively, the SI units of S/m may be used. S/m = 100 S/cm.)

3.2.4 *drift*—the average slope of the noise envelope expressed in nano siemens per centimetre per hour as measured over a period of 1 h.

3.2.5 *equivalent conductivity*— *of an ionic solute*, the contribution of the solute to the total conductivity of the solution, measured in microsiemens per centimetre, divided by its concentration in milliequivalents/litre.

3.2.6 *flow dependence rate*—the change in measured conductivity as a function of flow rate.

3.2.7 *limiting equivalent conductivity*—*of an ionic solute*, its equivalent conductivity extrapolated to infinite dilution.

3.2.8 *linear range*—*of a conductivity detector for a given solute in a specific solvent*, the concentration range of solute for which the detector response factor is within 5 % of the response factor in the middle of the range as determined from the linearity plot specified in Section 11.

3.2.8.1 *Discussion*—The lower limit may be limited by noise, and the upper limit by deviation from linearity. (The upper limit may instead be limited by the maximum full-scale deflection on the detector's least sensitive output range.)

3.2.9 *long-term noise*—the maximum amplitude in nano siemens per centimetre for all random variations of the detector output of frequencies between 2 and 60 cycles per hour.

3.2.9.1 *Discussion*—Long-term noise represents noise which can be mistaken for eluting peaks.

3.2.10 *minimum detectability*— *of a conductivity detector*, that concentration of solute in a specific solvent which corresponds to twice the short-term noise.

3.2.10.1 *Discussion*—Because of the difficulty of pumping solvents through the chromatographic system without any contamination of the solvents from the system, this quantity can only be measured with solutes retained by a column. Since minimum detectability is dependent on the chromatographic system used, it is not measured in this practice. However, if the minimum detectability of a solute is measured on one system with one detector, the minimum detectability can be predicted when other detectors are tested on the same system by comparing the measured values of short-term noise.

---

3.2.11 *response factor*—*of a conductivity detector*, the measured conductivity response of a solute divided by the solute concentration.

3.2.12 *response time of the detector*—the time required for the output of the detector to change from 10 % to 90 % of the new equilibrium value when the composition of the eluent is changed in a stepwise manner, within the linear range of the detector.

3.2.12.1 *Discussion*—A slow response time has the effect of limiting resolution for efficient peaks such as early eluting peaks and those from highly efficient columns or microbore columns. Response time is generally dependent on three factors: (*a*) cell volume, (*b*) volume of heat transfer tubing leading to the cell, and (*c*) electronic filtering of the output.

3.2.13 *sensitivity*—the detector response divided by concentration, which is also the response factor (11.1.1).

3.2.13.1 *Discussion*—Sensitivity is therefore by definition the same for all properly calibrated conductivity detectors. (Sensitivity is often confused with minimum detectability, which is dependent on both sensitivity and noise.) Therefore, the calibration of the detector should be measured, and if necessary, adjusted. Follow the manufacturer's procedure for calibrating the detector. The procedure in Section 9 is used by many manufacturers and is useful for the tests in this practice.

3.2.14 *short-term noise*—the maximum amplitude in nano siemens per centimetre for all random variations of the detector output of a frequency greater than one cycle per minute.

3.2.14.1 *Discussion*—Short-term noise determines the smallest signal detectable by a conductivity detector, limits the precision available for the determination of trace samples, and may set the lower limit of linearity.

## 4. Summary of Practice

4.1 Four different tests are performed to characterize a detector.

4.1.1 *Noise and drift* are measured while a solution is flowing through the detector cell. The test is performed using two different solutions: deionized water (DI) and 1 mM potassium chloride (KCl).

4.1.2 *Linear range* is determined by preparing a plot of response factor versus the log of solute concentration using standard solutions of KCl and hydrochloric acid (HCl) as solutes.

4.1.3 *Dependence of response on flow rate* is measured by pumping 1 mM KCl through the conductivity cell at several flow rates and measuring the detector output.

4.1.4 *Response time* is measured by measuring the time required for the detector output to change from that measured with DI water to that measured with 1 mM KCl.

## 5. Significance and Use

5.1 This practice is intended to describe the performance of a conductivity detector independent of the chromatographic system in terms that the analyst can use to predict overall system performance when the detector is coupled to the column and other chromatography system components.

5.2 Although it is possible to observe each of the several characteristics of a detector under different and unique conditions, it is the intent of this practice that a complete set of

detector specifications should be obtained at the same operating conditions, including the setup used for testing, flow rates, and temperatures. It should be noted that to specify a detector's capability completely, its performance should be measured at several sets of conditions within the useful range of the detector. The terms and tests described in this practice are sufficiently general so that they may be used at whatever conditions may be chosen for other reasons.

## 6. Reagents

6.1 Reagent chemicals are reagent grade or better.

6.1.1 *Deionized Water*, (DI water), 18 M-ohm.

6.1.2 *Potassium Chloride*, (KCl) dry powder.

6.1.3 *Hydrochloric Acid*, (HCl) standard 0.1000 *N* solution.

## 7. Preparation of Standards

7.1 *Potassium Chloride Standards*:

7.1.1 Prepare a 10-mM KCl standard stock solution. Weigh out 0.7455 g KCl (desiccated) and dissolve it in 18 M-ohm DI water in a 1-L plastic volumetric flask. Fill the flask to 1 L with DI water.

7.1.2 Prepare KCl standards from the 10-mM KCl standard stock solution. Using accurate Class A pipettes, pipette the volumes of the 10-mM standard stock solution listed below into 100-mL plastic volumetric flasks. For the 1-mM KCl standard, fill a 100-mL plastic volumetric flask with the 10-mM KCl solution and transfer to a 1-L plastic volumetric flask. Fill to the line with DI water.

| KCl Concentration, mm | Volume in 100 mL DI Water, mL |
|---|---|
| 0.05 | 0.5 |
| 0.1 | 1 |
| 0.2 | 2 |
| 0.5 | 5 |
| 1 | 100 mL in 1 L |
| 2 | 20 |
| 5 | 50 |
| 10   mM | No dilution |

7.2 *Hydrochloric Acid Standards*:

7.2.1 Prepare a 2.00-mM HCl standard stock solution by diluting 20.0 mL of standard 0.1000 *N* HCl into a 1-L plastic volumetric flask and filling to the line with DI water. If standard 0.1000 *N* HCl is not available, a 0.10-mM HCl solution can be prepared by diluting 8.3 mL of 12 *N* (37 %) concentration HCl into 1 L of DI water. (The concentration of this solution will be less accurate than that prepared from 0.1000 *N* HCl standard.)

7.2.2 Prepare the following HCl calibration standards from the 2.00-mM HCl standard stock solution. Use accurate Class A pipettes and 100- mL plastic volumetric flasks.

| HCl Concentration, mM | Volume in 100 mL DI Water, mL |
|---|---|
| 0.02 | 1 |
| 0.04 | 2 |
| 0.1 | 5 |
| 0.2 | 10 |
| 0.4 | 20 |
| 1 | 50 |
| 2 | No dilution |

## 8. Instrumentation Set-Up

8.1 Set up the chromatographic system according to the