



Standard Test Method for Operating Performance of Continuous Electrodeionization Systems on Feeds from 50–1000 $\mu\text{S}/\text{cm}^1$

This standard is issued under the fixed designation D 6529; 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 test method covers the determination of the operating characteristics of continuous electrodeionization (CEDI) devices using synthetic feed solutions and is not necessarily applicable to natural waters. This test method is a procedure applicable to solutions with a conductivity range from approximately 50 to 1000 $\mu\text{S}/\text{cm}$.

1.2 This test method covers the determination of operating characteristics under standard test conditions of CEDI devices where the electrically active transfer media therein is predominantly unregenerated. This results in more rapid achievement of steady state and shorter test time than when performing a test which requires the active media be predominantly regenerated.

1.3 This test method is not necessarily indicative of the following:

1.3.1 Long-term performance on feed waters containing foulants or sparingly soluble solutes, or both,

1.3.2 Performance on feeds of brackish water, sea water, or other high-salinity feeds,

1.3.3 Performance on synthetic industrial feed solutions, pharmaceuticals, or process solutions of foods and beverages, or,

1.3.4 Performance on feed waters less than 50 $\mu\text{S}/\text{cm}$, particularly performance relating to organic solutes, colloidal or particulate matter, or biological or microbial matter.

1.4 This test method, subject to the limitations previously described, can be applied as either an aid to predict expected deionization performance for a given feed water quality, or as a test method to determine whether performance of a given device has changed over some period of time. It is ultimately, however, the user's responsibility to ensure the validity of this test method for their specific applications.

1.5 *This standard does not purport to address all of the safety concerns, 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 or regulatory limitations prior to use.*

¹ This test method is under the jurisdiction of ASTM Committee D-19.08 on water and is the direct responsibilities of Subcommittee D19.08 on Membranes and Ion Exchange Materials.

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2. Referenced Documents

2.1 ASTM Standards:

D 1125 Test Methods for Electrical Conductivity and Resistivity of Water²

D 1129 Terminology Relating to Water²

D 1193 Specification for Reagent Water²

D 1293 Test Methods for pH of Water²

D 2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D-19 on Water²

D 4189 Test Method for Silt Density Index (SDI) of Water²

3. Terminology

3.1 *Definitions*—For definitions of general terms used in this test method, refer to Terminology D 1129.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *cell*—an independently fed ion-depleting chamber formed by two adjacent ion-exchange membranes, or by a membrane and an adjacent electrode.

3.2.2 *continuous electrodeionization (CEDI) device*—a device that removes ionized and ionizable species from liquids using electrically active media and using an electrical potential to influence ion transport, where the ionic transport properties of the active media are a primary sizing parameter. The CEDI devices typically comprise semipermeable ion-exchange membranes and permanently charged ion-exchange media. Examples include continuous deionization, electrodiagnosis, and packed-bed or filled-cell electro dialysis.

3.2.3 *current efficiency*—the ratio, expressed in percent, of the net transfer of ionized and ionizable solutes per unit cell within a CEDI device, expressed in chemical equivalents transferred per unit time, to the number of coulombs transferred from an external dc power source to each electrode pair, expressed in faradays per unit time. Calculation of current efficiency is described in 9.2.

4. Summary of Test Method

4.1 This test method is used to determine performance capabilities of CEDI devices with regard to extent of ion removal, pressure/flow relationships and electrical power consumption at standard or nominal operating conditions, electrical current characteristics, and the relative ability of the device

² Annual Book of ASTM Standards, Vol 11.01.

to remove ionized species when fed pretreated tap water. On this type of feed there is little water splitting or resin regeneration, thus only ionized species are removed. This test method is applicable to both new and used devices.

4.2 Pressure loss data is obtained. This information provides information relating to possible particulate plugging, fouling, or internal damage of the device. Deionization performance, concentrating stream pH, and electrical current transfer is monitored as a function of applied voltage. The initial ohmic (electrical) resistance, rate of change of ohmic resistance, and maximal ohmic resistance is determined. Also the electrical current efficiency as a function of deionization performance and voltage is determined. This information in combination with concentrating stream pH as a function of applied voltage provides basic design and performance information.

5. Significance and Use

5.1 The CEDI devices can be used to produce deionized water from feeds of pretreated water. This test method permits the relatively rapid measurement of key performance capabilities of CEDI devices using standard sets of conditions. The data obtained can be analyzed to provide information on whether changes may have occurred in operating characteristics of the device independently of any variability in feed water characteristics or operating conditions. Under specific circumstances, this test method may also provide sufficient information for plant design.

6. Apparatus

6.1 Description:

6.1.1 The test apparatus is schematically represented in Fig. 1. Feed water to the apparatus may be passed through a heat exchanger or other accessories, or both, to modify or control feed water temperature, or both, as desired. Alternately, data obtained from the operation of the apparatus may be normalized for temperature if normalization factors are known.

6.1.2 Feed water to the apparatus enters a holding tank (open or vented) of volume sufficient to maintain good control of water level and solute concentrations. The tank is unpressurized, ported to be capable of occasional cleanings or sanitizations, and incorporates needed safety features such as temperature and overflow protection. The tank also incorporates a drain valve. During operation of the apparatus, the drain valve may be used in combination with a valve controlling the rate of feed water to the apparatus to aid in control of solute concentrations, water level, and temperature within the tank. The tank supplies water to a recirculation pump designed to feed water to the CEDI device at a flow rate and pressure consistent with the ratings of the CEDI device. A recirculation line with a shut off valve from the pump discharge to the tank may be incorporated as required for proper pump operation.

6.1.3 The concentration and pH of the feed water to the CEDI device is controlled by metering pumps that meter a controlled flow of concentrated aqueous sodium chloride, and either sodium hydroxide or hydrochloric acid as needed into the feed water to maintain the desired feed water solute concentration and pH.

6.1.3.1 The concentrated reagents may be metered into the discharge line of the pump or alternately may be metered into

the tank. In either case, mixing means are required to ensure that the feed water concentrations do not fluctuate. Metering pump assemblies and controllers should be designed with proper safety interlocks and controls configured so as to be “fail safe.” Suitable instrumentation must be provided to monitor solute concentration (by means of electrical conductivity or resistivity, see Test Methods D 1125), pH (Test Methods D 1293), and temperature of the feed water to the CEDI device. Instrumentation may be in-line or alternately, appropriate sample taps may be incorporated to allow for determination off-line.

6.1.4 Feed water provided to the CEDI device should be plumbed as specified by the supplier, with appropriate flow and pressure controls, internal recirculations, drains, interlocks, safety controls, and other features as required. Pressure at the inlet and outlet and flow rates of each of the streams of interest must be monitored (for example, deionized water stream, concentrate stream, and electrode feed stream).

6.1.5 The CEDI device should be powered as specified by the supplier, with equipment and wiring to provide appropriate supply dc voltage and amperage, controls, interlocks, grounding, and safety features. Supply voltage and supply amperage to the CEDI device should be monitored at positions within the device or device assembly as specified by the supplier.

6.1.6 Streams leaving the CEDI device may be returned to the tank by means of return lines. Alternately, one or more of the streams may be sent either completely or partially to drain by means of appropriate valving if such operation provides easier control of desired feed water conditions. The outlet deionization stream is monitored for electrical conductivity or resistivity and optionally pH, and the outlet concentrating stream is monitored for pH and optionally electrical conductivity or resistivity. For CEDI devices with internal recirculation and “feed and bleed” features, solute concentrations must be measured at locations that are indicative of conditions within the CEDI module prior to mixing of recirculation flows.

6.2 Installation:

6.2.1 Materials of construction shall be as specified by the supplier of the CEDI device and in conformance to standard engineering practice.

6.2.2 Controls and monitors should be calibrated and maintained in accordance with the supplier’s requirements and standard engineering practice.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.³ Other grades may be used, provided it is first ascertained that the reagent is of

³ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

FIGURE 1
 PROCESS FLOW SCHEMATIC
 TEST METHOD FOR OPERATING PERFORMANCE OF
 CONTINUOUS ELECTRODEIONIZATION SYSTEMS ON FEEDS FROM 50-1000 $\mu\text{S}/\text{cm}$

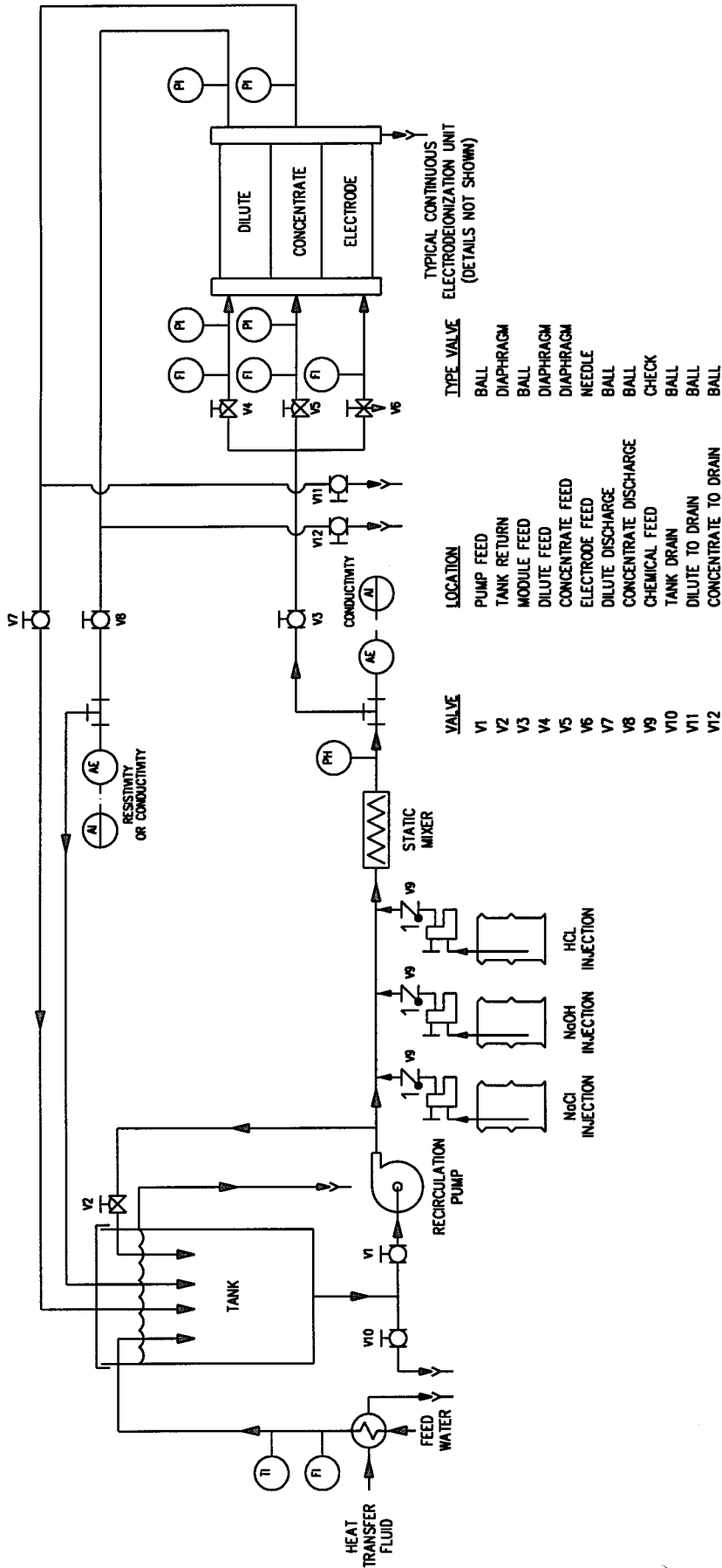


FIG. 1 Process Flow Schematic Test Method for Operating Performance of Continuous Electrodeionization Systems on Feeds from 50-1000 $\mu\text{S}/\text{cm}$

sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Purity of Water—Unless otherwise indicated, reference to water shall be understood to mean Type III reagent conforming to Specification D 1193 and meeting the following additional minimum quality specifications:

- $SDI_{15} < 4$ (Test Method D 4189)

7.3 Sodium Chloride Feed Solution—Dissolve enough sodium chloride to make a concentrated solution suitable for proper control of salinity fed to the device.

7.4 Sodium Hydroxide Solution—Dissolve or dilute enough concentrated sodium hydroxide to make a solution suitable for proper control of water pH fed to the device.

7.5 Hydrochloric Acid Solution—Dilute enough concentrated hydrochloric acid solution to make a solution suitable for proper control of water pH fed to the device.

8. Procedure

8.1 Startup:

8.1.1 Ensure that the tank and reagent feed reservoirs are sufficiently full, with adequate feed water rate to accommodate any losses of water caused by the positioning of the various drain valves. Control valves to the CEDI device should be closed and the device should be unpowered.

8.1.2 Turn on the recirculation pump and then slowly open the feed water throttling valves and various valves and recirculation devices on the CEDI device until the device is operating at nominal or supplier-recommended flow conditions. Modify throttling valves to adjust inlet and outlet pressures of the various device streams in accordance with supplier recommendations.

8.1.3 Operate the system with no dc power applied for a sufficient time to ensure adequate removal of any residual air from the piping and device. During this time, flows, pressures, feed solute concentrations, pH, and temperature should be adjusted until a desirable steady-state device feed water condition has been attained. Operation should be continued until feed, deionization stream and concentrating stream solute concentrations, pH values, and temperatures are all approximately the same. This ensures that the device is at equilibrium with the test solution, so that the results will not be affected by the feed stream on which the device was previously operated.

8.1.4 Feed pH should be maintained in a range from 6.0 to 8.0 and feed solute conductivity should be maintained at some specific value ($\pm 10\%$) in the 50 to 1000 $\mu\text{S}/\text{cm}$ range. The specific value depends on the device being tested, for instance to match the capacity of the dc power supply or to duplicate the conditions for which the CEDI device was designed.

8.2 Pressure Drop Measurements—Once steady state is achieved, as described in 8.1, measure and record pressures of the various inlet and outlet streams of the device. If necessary, normalize pressure differentials for temperature and compare to the supplier's specifications. If operating pressure specifications cannot be met, consult the supplier for the recommended course of action prior to proceeding with the collection of additional data.

8.3 Electrical Property and Deionization Performance Measurements:

8.3.1 Once steady state is achieved, as described in 8.1 and 8.2, turn on the dc power supply to the CEDI device, beginning at a low voltage. Initial applied dc voltage should be approximately 0.5 V per unit cell within the device (consult the supplier for the recommended values). A cell is the repeating unit of the device, and operating voltage expressed per cell therefore applies to devices with different numbers of cells. Continue to operate until steady state is achieved.

8.3.2 Measure and record dc voltage, dc amperage, device feed water temperature, deionizing stream inlet and outlet conductivity or resistivity, deionizing or concentrating stream outlet pH, or both, and deionizing stream flow rate. Fig. 2 is a sample test data sheet. After recording initial electrical and deionization properties, raise the applied dc voltage in increments of approximately 0.5 V per unit cell, each time recording the data previously described after a new steady state is achieved. Volts per cell may be normalized for feed water temperature if such a relationship has been developed. Continue this process until either: (1) the suppliers' recommended operating conditions for voltage, amperage, or outlet pH have been attained, or (2) the ohmic resistance of the device as determined by the quotient of voltage and amperage decreases from the previous recorded lower value of applied voltage.

8.4 Shut Down Procedure—The system should be shut down in accordance with the CEDI manufacturer's recommendations. If no specific recommendations are given, the following procedure should be suitable. Turn off applied dc voltage and continue to operate until feed, deionized, and concentrating stream outlet solute concentrations are approximately equal. Close valves for feed water to the CEDI device. Shut off the pH control metering equipment and sodium chloride feed equipment. Turn off the test stand recirculation pump.

9. Calculation

9.1 Pressure Drop—Calculate and record the pressure drop in the deionizing stream and concentrating stream respectively by subtracting deionizing stream outlet pressure from deionizing stream feed pressure and subtracting the concentrating stream outlet pressure from the concentrating stream feed pressure. Calculate and record the intermembrane pressure drop at the inlets and outlets respectively by subtracting the concentrating stream inlet pressure from the deionizing stream inlet pressure and by subtracting the concentrating stream outlet pressure from the deionizing stream outlet pressure. Pressure drop calculations can be normalized for temperature if such a correlation has been developed.

9.2 Electrical Properties—Calculate and record the electrical current efficiency (the ratio of chemical equivalents of deionization to the electrical equivalents of current passed) as follows:

$$\eta = \frac{Q_d \cdot (N_{d(in)} - N_{d(out)}) \cdot 160\,800}{n_{cp} \cdot I} \quad (1)$$

CEDI PERFORMANCE TEST								
TEST PARAMETER	UNITS	0 V/Cell	1 V/Cell	2 V/Cell	3 V/Cell	3.5 V/Cell	4 V/Cell	FINAL V/Cell
Elapsed Time	min	0.0						
Feed pH	pH							
Concentrate pH	pH							
Feed Conductivity	μS/cm							
Product Conductivity	μS/cm							
Salt Removal	%	--						
DC Voltage	volts	0.0						
Feed Temperature	°C							
DC Current	amps	0.0						
Current Efficiency	%	--						
Dilute Flow Rate	gpm							
Concentrate Flow Rate	gpm							
Electrode Flow Rate	gph							
Electrode Inlet Pressure	psig							
Dilute Inlet Pressure	psig							
Dilute Outlet Pressure	psig							
Dilute DP (Inlet - Outlet)	psid							
Concentrate Inlet Pressure	psig							
Concentrate Outlet Pressure	psig							
Concentrate DP (Inlet - Outlet)	psid							

FIG. 2 Sample Test Data Sheet

where:

- η = current efficiency, %,
- Q_d = deionizing stream flow rate between a given electrode pair, L/min,
- $N_{d(in)}$ = combined ionized and ionizable concentration of all species present in the deionizing stream inlet, chemical equivalents/L,
- $N_{d(out)}$ = combined ionized and ionizable concentration of all species present in the deionizing stream outlet, chemical equivalents/L,
- n_{cp} = number of independently fed diluting cells between electrodes within the device, and
- I = amperage passed between the electrodes (amperes).

$N_{d(in)}$ and $N_{d(out)}$ can be determined by means of direct analysis or titration, or by suitable correlation with electrical resistivity or conductivity. Calculate and record ohmic resistance in ohms by dividing voltage by the amperage between a given electrode pair. Voltage or ohmic resistance can be normalized for temperature if such a correlation has been developed.

9.3 *Deionization Performance*—For each ionized or ionizable species being monitored calculate the percent removal as follows:

$$R = \frac{C_{d(in)} - C_{d(out)}}{C_{d(in)}} \quad (2)$$

where:

- R = percent removal, %, and
 $C_{d(in)}$ = deionizing stream inlet concentration of a particular species (for example, sodium or chloride), and
 $C_{d(out)}$ = deionizing stream outlet concentration of a particular species.

10. Precision and Bias

10.1 Single-operator precision and bias was determined using a 4-cell continuous electrodeionization module, with a nominal flow rate capacity of 1.9 L/min (0.5 gpm). The module was tested in triplicate by six operators in one laboratory. The operators who participated represented a wide range of experience levels. Since other sources of variability should be relatively small (such as from the conductivity meter), the multiple-laboratory variability is expected to mimic the single-

operator precision and is not separately determined. The following results were obtained for the deionization performance:

$$x = 93.95 \text{ (\% removal)}$$

$$S_o = 0.72 \text{ (\% removal)}$$

where:

- x = arithmetic mean of the 18 determinations, and
 S_o = single-operator precision calculated in accordance with Practice D 2777.

10.2 Since known standards are not available, bias can not be determined.

11. Keywords

11.1 continuous electrodeionization; deionization; electrical current efficiency; electrical resistance; electrodeionization; electroregeneration; high-purity water; pressure differential

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