

**SEMATECH Provisional Test Method  
for Determining the Water Retention  
Capacity of Ion-Exchange Resins  
Used in UPW Distribution Systems**

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# SEMATECH Provisional Test Method for Determining the Water Retention Capacity of Ion-Exchange Resins Used in UPW Distribution

**Systems**

Technology Transfer # **92010940B-STD**

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**Abstract:** This test method determines the amount of water retained by hydrated ion-exchange resins. Procedures are provided for cation, anion, and mixed-bed resins. The method is applicable to both new and used resins. This document is in development as an industry standard by Semiconductor Equipment and Materials International (SEMI). When available, adherence to the SEMI standard is recommended.

**Keywords:** Ultrapure Water Distribution Systems, Testing, Water Retention, Ion-Exchange Resins

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## SEMASPEC #92010940B–STD

### SEMATECH Provisional Test Method for Determining the Water Retention Capacity of Ion-Exchange Resins Used in UPW Distribution Systems

#### 1. Introduction

- 1.1 *Purpose*—This test method covers the determination of water retained by ion-exchange resins when used for the treatment of ultrapure water (UPW) in the electronics industry.
- 1.2 *Scope*
- 1.2.1 This method is applicable to both new and used resins. The emphasis of this method is on new resins only.
- 1.2.2 This test method covers the determination of the amount of water retained by hydrated ion-exchange resins. The three procedures described herein apply to cation, anion, and mixed- bed ion-exchange resins.
- 1.3 *Limitations*—Comparisons between water retention values for different ion-exchange materials is meaningful only for new resins of the same type (e.g., cation) and with the same ionic form.

#### 2. Referenced Documents

- 2.1 *ASTM Standards*<sup>1</sup>
- |            |                                                                                               |
|------------|-----------------------------------------------------------------------------------------------|
| ASTM D1129 | Standard Definitions of Terms Relating to Water                                               |
| ASTM D1193 | Standard Specification for Reagent Water                                                      |
| ASTM D2187 | Standard Test Methods for Physical and Chemical Properties of Particulate Ion-Exchange Resins |
| ASTM D2687 | Sampling Particulate Ion-Exchange Materials, Practices for                                    |
- 2.2 *SEMATECH*<sup>2</sup>
- |                         |                                                                                                                    |
|-------------------------|--------------------------------------------------------------------------------------------------------------------|
| SEMASPEC #92010933B–STD | SEMATECH Guide to Test Methods for UPW Distribution System Components                                              |
| SEMASPEC #92010934B–STD | SEMATECH Provisional Test Method for Sample Preparation for Chemical Testing of UPW Distribution System Components |

#### 3. Terminology

- 3.1 *Acronyms and Abbreviations*
- 3.1.1 *PFA*—perfluoroalkoxy

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<sup>1</sup>American Society for Testing and Materials. 1916 Race St. Philadelphia, PA 19103.

<sup>2</sup>SEMATECH. 2706 Montopolis Dr. Austin, TX 78741.

3.1.2 *PTFE*—polytetrafluoroethylene

3.1.3 *UPW*—ultrapure water (see Section 7.1)

## 3.2 *Definitions*

3.2.1 *ion-exchange resin*—a synthetic organic, ion-exchange material.

3.2.2 *anion-exchange resin*—an ion-exchange resin capable of the reversible exchange of negatively charged ions.

3.2.3 *cation-exchange resin*—an ion-exchange resin capable of the reversible exchange of positively charged ions.

3.2.4 *mixed beds*—a physical mixture of anion-exchange material and cation-exchange material.

## 4. **Summary of Test Method**

This test method consists of the determination of loss of mass from the hydrated, dewatered ion-exchange resin upon drying at  $105 \pm 5^\circ\text{C}$ .

## 5. **Significance and Use**

5.1 The water retention capacity of an ion-exchange material is proportional to its pore volume. For new materials of the same functionality and polymer type, higher values indicate lower effective crosslinking.

5.2 Increases in water retention capacity of used materials as compared with the values for new material serve as an indicator of polymer crosslink breakage; decreases may indicate either loss of functionality or fouling of the ion-exchange material.

5.3 Since the numerical value is directly dependent on the ionic form of the material, careful preconditioning of new resin to known ionic forms, as described in ASTM D2187, is essential when such comparisons are made.

## 6. **Apparatus**

### 6.1 *Pretreatment Apparatus*

6.1.1 Column. The column should be transparent and vertically supported with an inside diameter of  $25 \pm 2.5$  mm ( $1.0 \pm 0.1$  in.) and an approximate length of 1500 mm (60 in.).

6.1.1.1 The bottom of the column should be closed with access to the column provided at the top and the bottom for admission and removal of solutions, as described in ASTM D2187.

6.1.1.2 Adequate means for measuring and regulating flow shall be provided.

6.1.2 Sample Support. The support should be designed so that the distance from the sample to the column outlet is at least 50 mm.

6.1.2.1 The recommended materials for fabricating the supports are polytetrafluoroethylene (PTFE) or perfluoroalkoxy (PFA) polymer screen, or porous plate.

- 6.2 *Dewatering Equipment*, including the following (also see Figure 1):
- 6.2.1 #2 Buchner Funnel.
  - 6.2.2 Suction Flask, 500 mL.
  - 6.2.3 Gas Wash Bottle, to act as a humidifier and a water trap.
  - 6.2.4 Open-Arm Mercury Manometer or Vacuum Gauge.
  - 6.2.5 Vacuum Source, with bleeder valve capable of creating a pressure differential of 40 mm Hg (mercury) below atmospheric pressure.
  - 6.2.6 #14 Rubber Stoppers.
  - 6.2.7 Rubber and Glass Tubing.
  - 6.2.8 Jar, 4-oz., with plastic-lined cap.
  - 6.2.9 Filter Paper, 7.5-cm, medium porosity.
  - 6.2.10 Wash Bottle.
  - 6.2.11 Spatula.
  - 6.2.12 Aluminum Weighing Pan.
  - 6.2.13 Analytical Balance, 0.001-g accuracy.
  - 6.2.14 Oven, mechanical convection.
  - 6.2.15 Desiccator.

## 7. **Materials**

- 7.1 *Test Fluid*. For purposes of this test, references to water shall be understood to mean ultrapure water as defined by maximum individual metal and anion impurity levels of 0.1 ppbw or less, total organic carbon (TOC) levels of 10 ppbw or less, nonvolatile residue levels of 0.1 ppmw or less, resistivity of 18 megohm-cm or greater, and reactive silica impurity of less than 1.0 ppb.

[Note: The term solids content is often used in reference to ion-exchange resins in lieu of water retention. Solids content is directly related to water retention, in that the sum of the two quantities expressed in percent is 100.]

### 7.2 *Reagents*.

- 7.2.1 *Reagent Grade Chemicals*, to be used in all tests. Unless otherwise indicated, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.<sup>3</sup> Other grades may be used only if it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.
- 7.2.2 *Sodium Chloride* (NaCl), reagent grade.

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<sup>3</sup> "Reagent Chemicals, American Chemical Society Specification," American Chemical Society (ACS), Washington, DC.

## 8. Precautions

8.1 *Safety Precautions*—This test method may involve hazardous materials, operations, and equipment. This test method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations before using this method.

### 8.2 *Technical Precautions*

8.2.1 Hydroxide form, strong base anion exchangers are unstable when heated above 40–50°C. Since oven temperatures for drying routinely exceed 100°C, the moisture retention of the anion exchange resin must be determined indirectly or in a different ionic form after conversion of the resin to a more stable ionic form.

8.2.2 When moisture retention values of different resins are compared, all must have the same ionic form or the comparisons will be meaningless.

## 9. Sampling, Test Specimens, and Test Units

9.1 *Sampling*—All sampling should be representative of the entire volume of ion-exchange resin under test. Guidance on methods to obtain representative samples of ion-exchange resin is provided in ASTM D2687.

## 10. Preparation of Apparatus

10.1 *Pretreatment*—(See Figure 2) Only the test method for hydrated anion-exchangers requires pretreatment to convert the resin from the less stable hydroxide ionic form to the more stable chloride ionic form. This pretreatment has been incorporated into the procedure in Sections 12.2.1 to 12.2.9.

## 11. Conditioning

11.1 The anion and cation exchangers require conditioning via hydration as outlined in Sections 12.1.1 to 12.1.2 and Sections 12.2.1 to 12.2.2. No similar hydration steps occur for mixed-bed, ion exchangers.

11.2 Make all measurements at  $25 \pm 5^\circ\text{C}$ .

## 12. Procedure

### 12.1 *Protocol for Hydrogen Form Hydrated Cation Exchangers*

12.1.1 Transfer approximately 100 mL of thoroughly mixed cation-exchange resin sample to a clean 200-mL container.

12.1.2 Hydrate the resin with an equal volume of reagent water for at least 30 minutes.

12.1.3 Transfer the hydrated resin to a Buchner funnel containing a medium porosity filter paper. Place the funnel on the dewatering apparatus (see Figure 1).

12.1.4 Adjust the vacuum with the bleeder control valve to maintain a pressure differential of  $40 \pm 5$  mm Hg below atmospheric pressure.

12.1.5 Drain the water in the funnel to the top of the resin sample. Seal the funnel with a #14 rubber stopper that is fitted with an inlet for air from a humidifier.

- 12.1.6 Maintain the vacuum at  $40 \pm 5$  mm Hg below atmospheric pressure for five minutes.
- 12.1.7 Remove the stopper and use a tissue to remove any moisture adhering to the inside of the funnel.
- 12.1.8 Use a spatula to transfer the dewatered resin to a dry 200-mL jar. Carefully mix the sample and immediately cap the jar.
- 12.1.9 Determine the weighing pan tare weight, (value *A* in equations in Section 13) to the nearest 0.001 g. Transfer 3–5 g of dewatered resin to the pan and reweigh (value *B* in equations in Section 13) to the nearest 0.001 g.
- 12.1.10 Place weighed sample in a  $105 \pm 5^\circ\text{C}$  convection oven to dry for at least 24 hours.
- 12.1.11 Remove the sample from the oven. Cool to ambient temperature in a desiccator for approximately 15 minutes. Reweigh (value *C* in equations in Section 13) to the nearest 0.001 g.

## 12.2 *Protocol for Hydrated Anion Exchangers*

[Note: The hydroxide ion forms strong base anion exchangers that are unstable when heated above 40 to  $50^\circ\text{C}$ . Since oven temperatures routinely exceed  $100^\circ\text{C}$ , the moisture retention of the anion-exchange resin must be determined indirectly or in a different ionic form (after conversion of the resin to a more stable ionic form). In this method, the anion-exchange resin is pretreated with NaCl to convert the resin from the hydroxide form to the chloride form. All subsequent analyses are performed using the chloride form. When moisture retention values of different resins are compared, all must have the same ionic form, or the comparisons will be meaningless.]

- 12.2.1 Dissolve 100.0 g of sodium chloride (NaCl) in 800 mL of water and dilute to 1.0 liter in a volumetric flask. This will form a 100-g/l sodium chloride solution.
- 12.2.2 Transfer approximately 100 mL of thoroughly mixed anion-exchange resin sample to a clean 200-mL jar.
- 12.2.3 Hydrate the resin with an equal volume of reagent water (or UPW) for at least 30 minutes.
- 12.2.4 Fill the pretreatment column one-half full with water. Transfer the entire contents of the 200-mL jar to the column using additional water, if necessary.
- 12.2.5 Backwash with water; use a flow rate that will maintain a 50% expansion of the bed. (For example, if the resin bed is 12 inches high, the water should be 6 inches above the height of the resin.) Adjust the backwash outlet tube to a height above the bed equal to 75% of the bed height. Continue backwashing for a minimum of 10 min. or until the effluent is clear.
- 12.2.6 Allow the resin to settle and then drain until the liquid level is 20 to 30 mm above the top of the bed. Pass NaCl solution (100 g/l) downflow through the bed at the approximate rate of 0.133 mL/min. per mL of sample for one hour. Discontinue the flow of NaCl solution.
- 12.2.7 Backwash with water for 10 min. at a flow rate sufficient to maintain a 50% expansion of the bed. Discontinue the flow of water.

- 12.2.8 Allow the bed to settle and then drain off the water at a rate of approximately 100 mL/min. until the water level is 20 to 30 mm above the top of the bed.
- 12.2.9 Rinse the bed with reagent water at the same flow rate as in Section 12.2.7 for 30 minutes.
- 12.2.10 Transfer the hydrated, pretreated resin to a Buchner funnel containing a medium porosity filter paper. Place the funnel on the dewatering apparatus (see Figure 1).
- 12.2.11 Adjust the vacuum with the bleeder control valve to maintain a pressure differential of  $40 \pm 5$  mm Hg below atmospheric pressure.
- 12.2.12 Drain the water in the funnel to the top of the resin sample. Seal the funnel with a #14 rubber stopper that is fitted with an inlet for air from a humidifier.
- 12.2.13 Maintain the vacuum at  $40 \pm 5$  mm Hg below atmospheric pressure for exactly five minutes.
- 12.2.14 Remove the stopper and use a tissue to remove any moisture adhering to the inside of the funnel.
- 12.2.15 Use a spatula to transfer the dewatered resin to a dry 200-mL jar. Carefully mix the sample and immediately cap the jar.
- 12.2.16 Determine the weighing pan tare weight (value *A* in equations in Section 13) to the nearest 0.001 g. Transfer 3–5 g of dewatered resin to the pan and reweigh (value *B* in equations in Section 13) to the nearest 0.001 g.
- 12.2.17 Place the weighed sample in a  $105 \pm 5^\circ\text{C}$  convection oven to dry for at least 24 hours.
- 12.2.18 Remove the sample from the oven. Cool to ambient temperature in a desiccator for approximately 15 minutes. Reweigh (value *C* in equations in Section 13) to the nearest 0.001 g.

### 12.3 *Protocol for Mixed Bed Resins*

[Note: Mixed bed resins are physical mixtures of two or more types of ion-exchange resins. The water retention of a mixed bed will represent a composite value of the individual water retention values of the components. This composite value has no physical significance. A more meaningful test involves separating the mixed bed into its components (as described in Section 10.5 of ASTM D2187) and then determining the water retention values of each component as described previously.]

This test is only performed on resins as received from the resin supplier. Adding water will cause resin separation. The resin must be obtained in hydrated form for this test to be valid.]

- 12.3.1 Determine the weighing pan tare weight (value *A* in Section 13) to the nearest 0.001 g.
- 12.3.2 Transfer 3–5 g of thoroughly mixed, as received resin to the pan and reweigh (value *B* in equations in Section 13) to the nearest 0.001 g.
- 12.3.3 Place the weighed sample in a  $105 \pm 5^\circ\text{C}$  convection oven to dry for at least 24 hours.
- 12.3.4 Remove the sample from the oven. Cool to ambient temperature in a desiccator for approximately 15 minutes.

12.3.5 Reweigh (value *C* in equations in Section 13) to the nearest 0.001 g.

### **13. Data Analysis**

#### *13.1 Calculation for Hydrated Cation Exchangers*

13.1.1 Calculate the water retention capacity, in percent, as follows:

$$\text{water retained in \%} = [(B - C)/(B - A)] \cdot 100$$

where:

A = weight of pan in grams

B = weight of pan and nondried cation resin in grams

C = weight of pan and dried cation resin in grams

#### *13.2 Calculation for Hydrated Anion Exchangers*

13.2.1 Calculate the water retention capacity, in percent, as follows:

$$\text{water retained in \%} = [(B - C)/(B - A)] \cdot 100$$

where:

A = weight of pan in grams

B = weight of pan and nondried anion resin in grams

C = weight of pan and dried anion resin in grams

#### *13.3 Calculation for Mixed Bed Resins*

13.3.1 Calculate the water retention capacity of the mixed bed, in percent, as follows:

$$\text{water retained in \%} = [(B - C)/(B - A)] \cdot 100$$

where:

A = weight of pan in grams

B = weight of pan and as-received mixed-bed resin in grams

C = weight of pan and dried mixed-bed resin in grams

### **14. Data Presentation**

#### *14.1 Report for Hydrated Cation Exchangers*

14.1.1 Report the percent water retained (by the hydrogen form cation-exchange resin) as the average of duplicate or triplicate values obtained.

#### *14.2 Report for Hydrated Anion Exchangers*

14.2.1 Report the percent water retained (by the chloride form anion-exchange resin) as the average of duplicate or triplicate values obtained.

#### *14.3 Report for Mixed Bed Resins*

14.3.1 Report the percent water retained (by the composite mixed bed of ion-exchange resins) as the average of duplicate or triplicate values obtained.

**15. Precision and Bias**15.1 *Method for Hydrated Cation Exchangers*

15.1.1 The precision of this test method may be expressed as follows:

$$S(T) = 0.017 x$$

$$S(O) = 0.004 x$$

where:

S(T) = overall precision

S(O) = single-operator precision

x = water retention capacity determined in percent

15.2 *Method for Hydrated Anion Exchangers*

15.2.1 The precision of this test method may be expressed as follows:

$$S(T) = 0.017 x$$

$$S(O) = 0.004 x$$

where:

S(T) = overall precision

S(O) = single-operator precision

x = water retention capacity determined in percent

15.3 *Method for Mixed Bed Resins*

15.3.1 The precision of this test method may be expressed as follows:

$$S(T) = 0.017 x$$

$$S(O) = 0.004 x$$

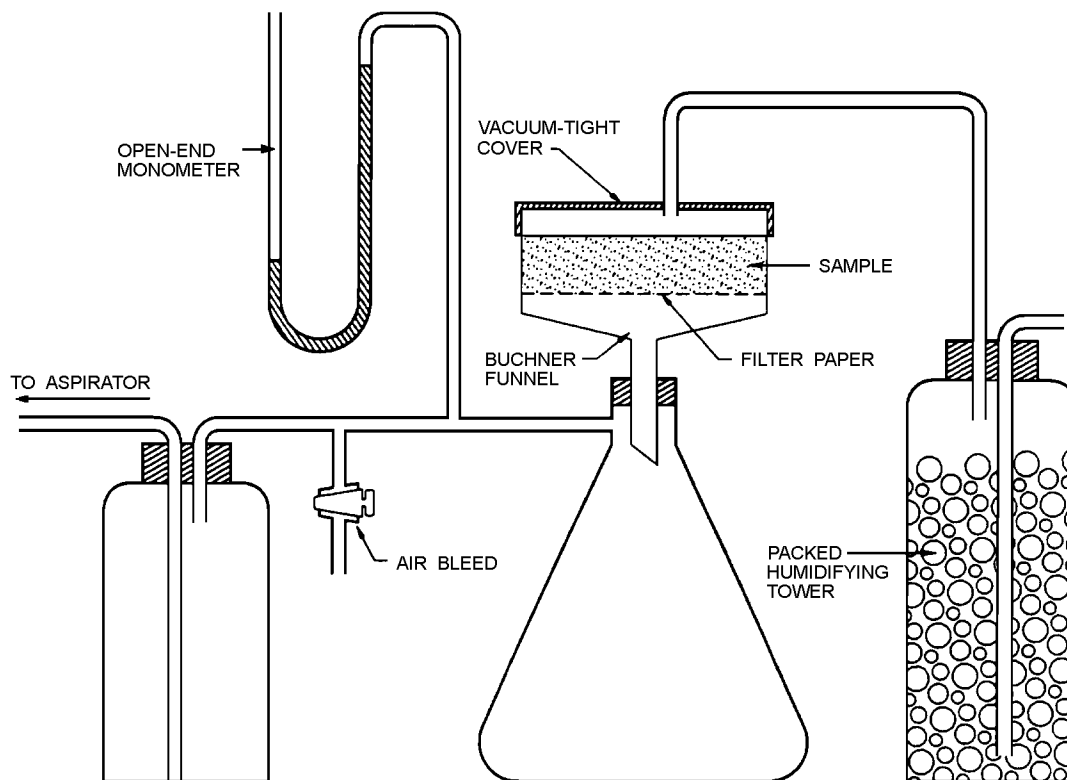
where:

S(T) = overall precision

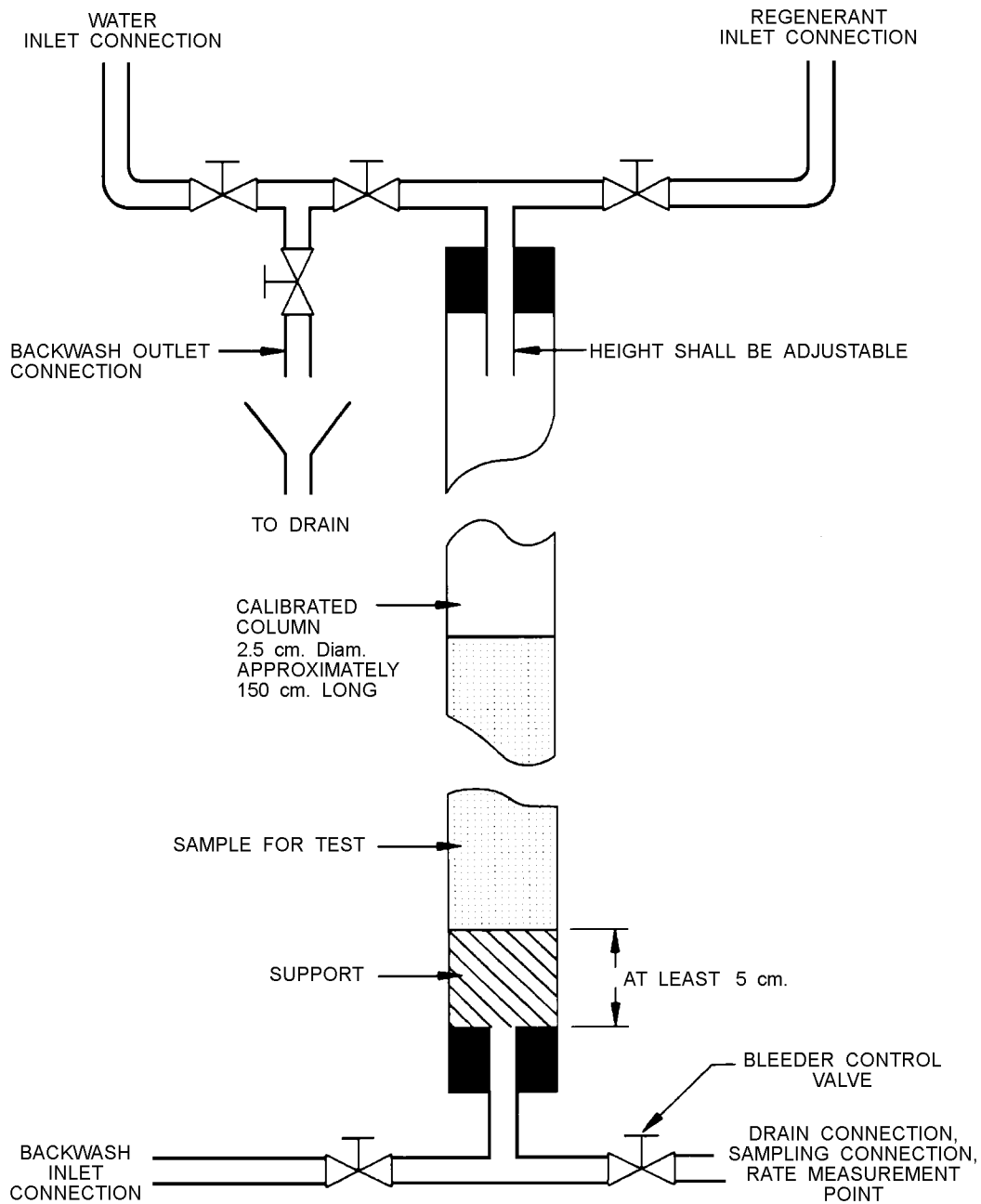
S(O) = single-operator precision

x = water retention capacity determined in percent

## 16. Illustrations



**Figure 1** Typical Arrangement of Water-Draining Apparatus



**Figure 2 Typical Arrangement of Apparatus for Performance Testing of Ion-Exchange Materials**

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