SEMATECH Provisional Test Method for Optical Analysis of Ion-Exchange Resin Beads Used in UPW Distribution Systems

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Abstract: This test method defines the procedure for optical testing of the physical integrity of ion-exchange resin materials (beads) used in the water purification process. The procedure uses an optical microscope at 20X magnification to determine the physical integrity of the beads. 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.

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1. Introduction

Ultrapure water (UPW) distribution systems contain ion exchange resin materials used in the water purification process. This document presents an optical test method for the evaluation of ion exchange resin beads.

1.1 *Purpose*—The purpose of this document is to define a method for testing the physical integrity of beads.

1.2 *Scope*—This document establishes an optical method for testing the physical integrity of ion exchange resin beads and provides a quantitative measurement technique. This procedure uses an optical microscope at 20× magnification to determine the physical integrity of beads.

1.3 *Limitations*

1.3.1 This methodology assumes that the operator is skilled enough to use an optical microscope.

1.3.2 The technique described here is limited to the assessment of beads to determine quantity of percent perfect beads.

2. Referenced Documents

2.1 ASTM D5127 Standard Guide for Electronic Grade Water[1]

2.2 Rohm and Hass Analytical Method No. RH-4. Perfect Bead Content. Issued 12/87[2]

3. Terminology

3.1 *Acronyms and Abbreviations*

3.1.1 *UPW*—ultrapure water

3.2 *Definitions*

3.2.1 *bead*—an individual spherical particle of an ion exchange resin material.

3.2.2 *cracked bead*—a bead that exhibits a visible crack when viewed at 20× magnification.

3.2.3 *fragmented bead*—a piece of resin material that, when viewed at 20× magnification, appears to indicate that it would form a whole bead if reassembled with other fragmented pieces.

3.2.4 *imperfect bead*—a cracked, pocked or fragmented bead.

3.2.5 *ion exchange resin*—a polymeric material used in ultrapure water systems to remove impurities from the liquid stream.

3.2.6 *percent perfect beads*—the number of perfect beads per 100 total beads. (See Section 11.)

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[1] American Society for Testing and Materials. 1916 Race St. Philadelphia, PA 19103.

[2] Rohm and Hass Company, Research Division. 727 Norristown Road. Spring House, PA 19477.

3.2.7 *perfect bead*—an uncracked, spherical bead that exhibits a whole appearance and that shows no signs of fragmentation when viewed at 20× magnification.

3.2.8 *pocked bead*—a bead that exhibits a surface depression when viewed at 20× magnification.

3.2.9 *total beads*—the summation of perfect beads plus cracked, pocked, and fragmented beads.

3.2.10 *ultrapure water*—type E-1 electronic grade water as defined in ASTM D5127.

4. Summary of Test Method

A sample of ion exchange resin beads is placed in a petri dish containing water and having a grid pattern. The beads are arranged into a closely packed monolayer. An optical examination of the monolayer at 20× magnification is done to determine the physical integrity of the beads. A calculation is then performed to quantify the findings.

5. Significance and Use

This method provides a means of distinguishing between various ion exchange resin bead products when physical integrity is significant in the selection of the product. Results may vary depending on the representativeness of the sample tested. The application of this test method is expected to yield comparable results among similar materials tested for purposes of qualification.

6. Apparatus

Optical Binocular Microscope, with 20× magnification capability, a direct white light brightfield, and an oblique light source.

7. Materials

7.1 *Glass Petri Dish*, 100 mm, with a grid of approximately 6.4-mm (1/4-in.) squares.

7.2 *Spatula*, or equivalent means to dispense beads.

8. Safety Precautions

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

9. Sampling, Test Specimens, and Test Units

9.1 *Sample Preparation*

9.1.1 Add sufficient water to cover the bottom of the petri dish.

9.1.2 Spread a representative sample of beads on the wetted surface of the petri dish to form a closely packed, uniform monolayer. For a bead with an average diameter of 600 µm, each square of the grid will contain approximately 100 beads.

9.1.3 Avoid unnecessary contact with the beads that may damage the sample.

10. Test Procedures

10.1 Illuminate the petri dish with light sources and focus the microscope at 20× magnification. Under this magnification, approximately 100–150 beads are visible in the field of view.

10.2 Select a square at random and estimate the total number of beads contained in the square by multiplying the number across by the number down.

10.3 Estimate the number of whole beads that any fragments would have yielded if reconstructed, and use this number in determining both the total number of beads and the number of imperfect beads. For example, if a square contained 99 whole beads and 3 fragments that would be equal to 1 whole bead if combined, this would be equivalent to 100 total beads.

10.4 Within the same square, count the number of other beads falling into the imperfect bead category and add that number to the value obtained in step 10.3 for fragmented beads.

10.5 Repeat steps 10.2 to 10.4 for a total of five samplings.

11. Data Analysis

11.1 *Calculations*

11.1.1 Subtract the number of imperfect beads from the total number of beads and divide the difference by the total number of beads.

11.1.2 Multiply the dividend obtained in Section 11.1.1 by 100. Record the resulting product as percent perfect beads. (See Figure 1.)

11.1.3 Record results for all 5 determinations, as well as the average obtained from the 5 determinations.

11.1.4 Calculate % perfect beads as follows:

$$\%\ Perfect\ Beads\ =\ \frac{average\ number\ of\ perfect\ beads}{total\ number\ of\ beads}x100\ =\ \frac{A-B}{A}x100$$

where: A = total # of beads
B = # of imperfect beads

12. Precision and Bias

Since physical integrity varies from one bead to another, precision and bias cannot be determined for this procedure.

13. Illustrations

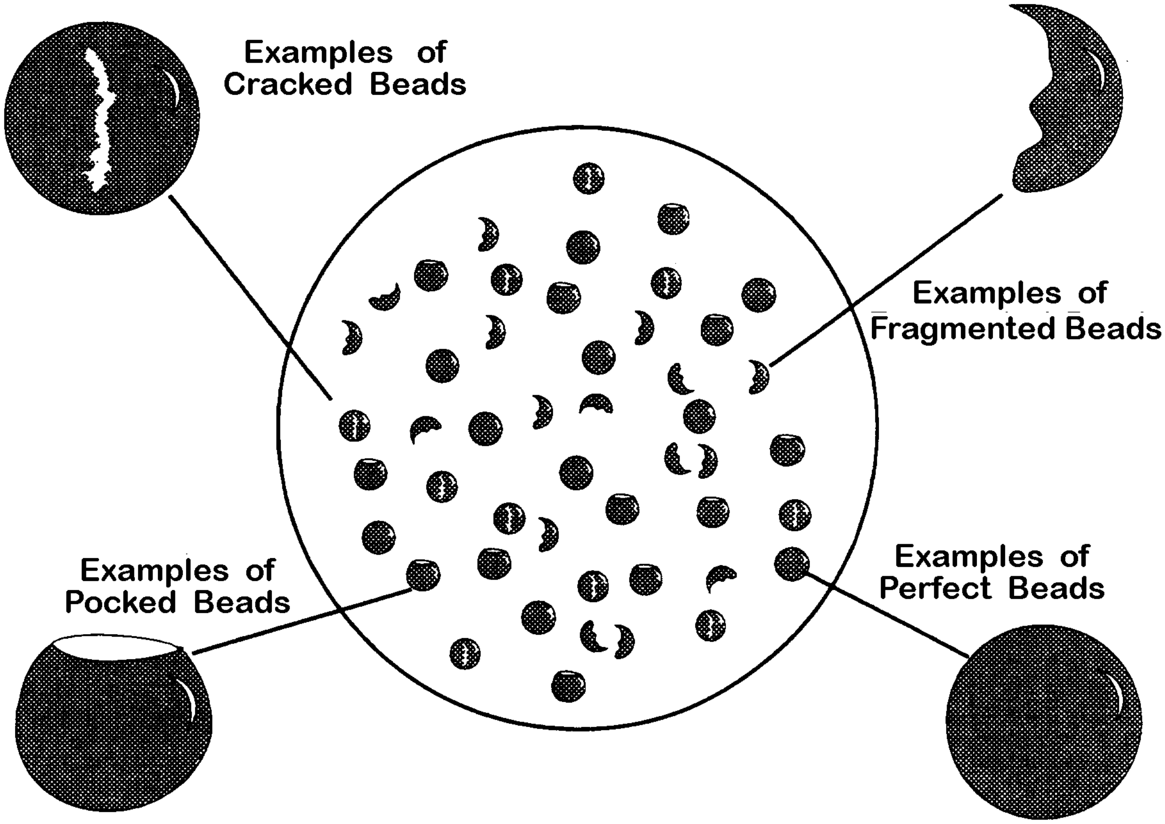

Figure 1 Example of a Microscopic Field Containing Perfect and Imperfect Beads

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