Designation: D 3923 – 94 (Reapproved 1998)

Standard Practices for Detecting Leaks in Reverse Osmosis Devices [1]

This standard is issued under the fixed designation D 3923; 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 These practices cover detecting leaks in which there is a direct communication between the feed or concentrate, or both, and the permeate. Several types of leaks are possible with the various configurations of reverse-osmosis devices.

1.2 *Types of Leaks*:

1.2.1 With hollow-fiber devices, feed or concentrate leakage, or both, into the permeate stream by leaks through the tube sheet and past the tube sheet O-ring are possible. "Leaks" caused by broken fibers are not covered by these practices.

1.2.2 With spiral-wound devices, leaks may occur through damage of the membrane surface itself by punctures or scratches, by glue-line failure, and by O-ring leaks on product tube interconnectors.

1.2.3 With tubular devices, leaks due to membrane damage, tube end seal leaks, and leaks from broken tubes or product headers are possible.

1.3 Three leak test practices are given as follows:

	Sections
Practice A—Tube Sheet and O-Ring Leak Test for Hollow Fiber Devices	6 to 9
Practice B—Vacuum Test for Spiral Wound Devices	10 to 12
Practice C—Dye Test for Spiral Wound and Tubular Devices	13 to 18

1.4 *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 of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1129 Terminology Relating to Water [2]

D 1193 Specification for Reagent Water [2]

D 4194 Test Methods for Operating Characteristics of Reverse Osmosis Devices [3]

E 60 Practice for Photometric and Spectrophotometric Methods for Chemical Analysis of Metals [4]

E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near Infrared Spectrophotometers [5]

3. Terminology

3.1 For definitions of terms used in these practices, refer to Terminology D 1129.

3.2 For descriptions of terms relating to reverse osmosis, refer to Test Methods D 4194.

3.3 *Definitions of Terms Specific to This Standard:*

3.3.1 *leak*—bypassing of the intact membrane from the feed side to the permeate side.

4. Summary of Practice

4.1 The hollow-fiber device being tested is operated at low pressure with the permeate tube sheet exposed (the fiber bundle is held in place by a "spider" device designed for the specific model under test). Any significant leak in the tube sheet or O-ring seal is detected visually by inspection.

5. Significance and Use

5.1 These practices may be used to determine whether a reverse-osmosis device is free of leaks if the mechanical integrity of the device is to be confirmed. They may also be used to detect leaks in reverse-osmosis devices whose operating performance indicates a possible leak. These practices may be used for either new or used devices.

6. Apparatus

6.1 *"Spider" Device*, designed for the specific model of hollow-fiber device being tested, is available from the supplier. The "spider" is designed to take the place of the permeate end plate and permeate collection grid/block while securing the fiber bundle from movement. This allows visual observation during low-pressure operation with the fiber bundle retained in its original position.

7. Purity of Reagents

7.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall

[1] These practices are under the jurisdiction of ASTM Committee D-19 on Water and are the direct responsibility of Subcommittee D19.08 on Membranes and Ion Exchange Materials.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 11.02.

[4] *Annual Book of ASTM Standards*, Vol 03.05.

[5] *Annual Book of ASTM Standards*, Vol 14.01.

conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[6] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determinations.

7.2 Unless otherwise indicated, references to water shall be understood to mean Type III reagent water conforming to Specification D 1193.

PRACTICE A—TUBE SHEET AND O-RING LEAK TEST FOR HOLLOW FIBER DEVICES

8. Scope

8.1 This practice is applicable to detecting feed or concentrate leakage, or both, through the tube sheet and past the tube sheet O-ring into the permeate in hollow-fiber devices.

9. Procedure

9.1 Connect a centrifugal pump with 1.4 MPa (200 psig) capability and a throttling valve to the feed port of the hollow-fiber device. Install a pressure gage and valve on the concentrate port of the reverse-osmosis device.

9.2 Remove the permeate end plate and auxiliary equipment in accordance with the supplier's instructions to expose the face of the permeate tube sheet.

9.3 Install a "spider" device (available from supplier) designed for the specific model under test and secure the "spider" with the snap or segmented ring that held the permeate end plate in place.

9.4 Place the hollow-fiber device in the horizontal position and orient the open concentrate line to the highest point (12 o'clock). Allow water to flow through the device at line pressure (approximately 350 kPa; 50 psig) to remove any trapped air in the device. Slowly close concentrate line valve to pressurize the unit to 350 kPa (50 psig). While *standing clear of the tube sheet*, start the pump and increase pressure slowly until a maximum pressure of 1.05 MPa (150 psig) is obtained. Proceed with the tube sheet inspection.

NOTE 1—The leak test should take approximately 15 min to determine the integrity of the tube sheet and tube sheet O-ring.

9.5 Examine for leaks by observing the product water as it exits the tube sheet face. If leaks do not exist, the product water appears to ooze out from the tube sheet face. However, if a significant leak is present in the tube sheet or O-ring seal, a forceful spray or stream will be observed. During inspection, rotate the device 180° to examine the lower portion of the tube sheet for leaks. This is necessary since leaks in the lower portion of the tube sheet are not easily discernible because of the accumulation of product water.

9.6 *Shutdown Procedure*:

9.6.1 Shut off the centrifugal pump and allow the pressure to reach zero before disconnecting the reverse-osmosis device.

9.6.2 Replace permeate end plate and all auxiliary equipment in accordance with the supplier's instructions.

9.6.3 Take care to ensure that the membranes are kept wet at all times and are properly sanitized or winterized, or both, (based on supplier's recommendations) for long-term storage (more than 5 days).

PRACTICE B—VACUUM TEST FOR SPIRAL WOUND DEVICES

10. Scope

10.1 This practice is applicable to detecting leaks in water-rust spiral-wound reverse-osmosis devices, new or used, when such leaks are significant enough to prevent the device from holding a vacuum. These leaks may be due to a damaged membrane, glue-line failure, or leaks in O-ring seals. This test is useful as a screening procedure and is not intended as a means of absolute verification of such leaks.

11. Summary of Practice

11.1 The device is evaluated with one end of the permeate collection tube sealed. A vacuum gage on the other end of the tube is observed. A rapid decay in vacuum indicates a leak.

12. Procedure

12.1 Seal one end of the permeate collection tube with a suitable leak-tight cap. Connect the other end of the permeate tube to a vacuum gage and a valved vacuum source.

12.2 Evacuate the element to 84 to 101 kPa vacuum (25 to 30 in. Hg vacuum). Close the isolation valve and observe the reading on the vacuum gage. Note the rate at which the vacuum decays. A rapid decay (greater than 20 kPa/ min (6 in.Hg/ min)) will indicate the presence of a leak.

12.3 *Shutdown Procedure*:

12.3.1 Slowly release the vacuum on the reverse-osmosis device and allow the device to reach atmospheric pressure before disconnecting.

12.3.2 Take care to ensure that the membranes are kept wet at all times and are properly sanitized or winterized, or both, for long-term storage (based on supplier's recommendations).

PRACTICE C—DYE TEST FOR SPIRAL WOUND AND TUBULAR DEVICES

13. Scope

13.1 This practice is applicable to detecting leaks in spiral wound or tubular reverse-osmosis devices, new or used, which are due to lack of or loss of mechanical integrity.

14. Summary of Practice

14.1 The practice consists of passing a solution of a dye, known to be rejected by the membrane, through the device under standard conditions as specified in Test Methods D 4194. The concentration of the dye in the permeate relative to that in the feed is measured either spectrophotometrically or by visual comparison of the color intensity. A dye passage of greater than 0.5 % indicates a leak.

[6] *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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

15. Apparatus

15.1 The test apparatus required is schematically described in Test Methods D 4194.

15.2 *Nessler Tubes or Photometer*—A set of 50-mL matched Nessler tubes or a photometer suitable for measurements at a wavelength of 590 nm is required.

NOTE 2—Filter photometers and photometric practices used in this practice shall conform to Practice E 60. Spectrophotometers shall conform to Practice E 275.

16. Reagents

16.1 *Dye Feed Solution (Methyl Violet 2B)*—Prepare a 100-mg/L dye feed solution by adding 0.1 g of methyl violet 2B/L of solution to water containing 1.5 g of NaCl/L.

NOTE 3—Other dyes may be used for this test if they have been shown to give equivalent results, for example, form stable solutions, are relatively insensitive to pH changes in the pH 4 to 8 range; give measurable absorbance values in a similar concentration range, etc. If another dye is used, a suitable wavelength must be determined for measurement.

17. Procedure

17.1 Install the test equipment in accordance with 8.1 through 6.3 of Test Methods D 4194.

17.2 Start up and operate the test system in accordance with Section 8 of Test Methods D 4194.

17.3 *Data Acquisition*:

17.3.1 Allow the system to equilibrate for 30 min while maintaining constant flow, pressure, and temperature conditions. At the end of this period, take a 100-mL sample of the feed and permeate. Record the flows, pressures, and conductivities of the feed, concentrate, and permeate streams as well as the permeate temperature.

17.3.2 Using dilutions where necessary, measure the absorbance of the feed and permeate samples against a water blank with a filter photometer or spectrophotometer at 590 nm. Alternatively, compare the permeate color intensity with suitable dilutions of the feed using Nessler tubes.

17.4 *Shutdown Procedure*—Thoroughly flush the device with water to remove all traces of the dye. Use the highest recommended concentrate flow rate at 350 kPa (50 psig) feed pressure. After flushing, allow the pressure to reach zero before disconnecting the reverse-osmosis device or carrying out maintenance on the piping system. Take care to ensure that the membranes are kept wet at all times and are properly sanitized or winterized, or both, (based on supplier's recommendations) for long-term storage (more than 5 days).

18. Calculation

18.1 Compute flows, conversions, salt passage, and rejection in accordance with Section 18 of Test Methods D 4194.

$$\text{Dye passage, \% (determined photometrically)} = A_p / A_F \times D_F \times 100$$

where:

A_p = absorbance of permeate of 590 nm,
A_F = absorbance of diluted feed at 590 nm, and
D_F = dilution factor for feed.

18.2 The dye passage can be determined directly by visual comparison of the permeate with appropriate dilutions of the feed in Nessler tubes and expressed as a percent of the feed.

18.3 A leak is diagnosed if the percent dye passage is greater than 0.5 %.

19. Keywords

19.1 dye test; leaks; O-ring test; reverse osmosis; tubesheet leak test; vacuum test