Designation: D3923 – 23

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

This standard is issued under the fixed designation D3923; 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 (RO) and nanofiltration (NF) 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:

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1129 Terminology Relating to Water

D1193 Specification for Reagent Water

D4194 Test Methods for Operating Characteristics of Reverse Osmosis and Nanofiltration Devices

D6161 Terminology Used for Microfiltration, Ultrafiltration, Nanofiltration, and Reverse Osmosis Membrane Processes

D6908 Practice for Integrity Testing of Water Filtration Membrane Systems

E60 Practice for Analysis of Metals, Ores, and Related Materials by Spectrophotometry

E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in these practices, refer to Terminology D1129 and D6161.

3.1.2 *concentrate, n*—stream exiting a crossflow membrane device that has increased concentration of solutes and particles over the feed stream; portion of the feed steram that does not pass through the membrane; the stream in which dissolved solids or particulates, or both, are concentrated in a membrane separation process.

3.1.3 *hollow-fiber (HF) membrane, n*—self-supporting membrane fibers that have a hollow bore like a cylinder; in reverse osmosis, the membrane is usually on the outside with the bore conveying the permeate; in ultra and micro filtration, the membrane might be on the inside or outside of the fiber.

3.1.4 *leak, n*—bypassing of the intact membrane from the feed side to the permeate side.

3.1.5 *nanofiltration (NF), n*—crossflow process with pore sizes designed to remove selected salts and most organics

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

Current edition approved Jan. 1, 2023. Published February 2023. Originally approved in 1980. Last previous edition approved in 2014 as D3923 – 08 (2014). DOI: 10.1520/D3923-23.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

above about 300 molecular weight range, sometimes referred to as loose RO; a pressure-driven membrane separation process in which particles and dissolved molecules larger than about 2 nm are rejected.

3.1.6 *permeate, n*—that portion of the feed which passes through the membrane.

3.1.7 *reverse osmosis (RO), n*—separation process in which one component of a solution is removed from another component by flowing the feed stream under pressure across a semipermeable membrane that causes selective movement of solvent against its osmonic pressure difference; RO removes ions based on electrochemical forces, colloids, and organics down to 150 molecular weight; may also be called hyperfiltration.

3.1.8 *spiral wound membrane, n*—a flat sheet membrane with one or more feed channel spacers, and barrier layers, all of which are rolled into a spiral configuration.

3.1.9 *tubular membrane, n*—the element, similar to hollow-fiber but with a bore diameter >5 mm (see *hollow-fiber*); used mostly in MF and UF and sometimes wit RO and NF when particulate loading is high.

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 RO or NF 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 RO or NF 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 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 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 D1193.

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 Drain the liquid from the feed side of the membrane.

9.2 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 RO or NF device.

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

9.4 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.

9.5 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.

9.6 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

[3] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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.

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.

9.7 *Shutdown Procedure:*

9.7.1 Shut off the centrifugal pump and allow the pressure to reach zero before disconnecting the RO or NF device.

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

9.7.3 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 spiral-wound RO or NF 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 (see Practice D6908).

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 Drain the liquid from the feed side of the membrane and let it remain open to the atmosphere. For membrane devices placed horizontally, the feed and exit ports must be located on the bottom of the device housings in order for this test to work.

12.2 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.

12.3 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.

12.4 *Shutdown Procedure:*

12.4.1 Slowly release the vacuum on the RO or NF device and allow the device to reach atmospheric pressure before disconnecting.

12.4.2 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 RO or NF devices, new or used, which are due to lack of or loss of mechanical integrity (see Practice D6908).

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 D4194. 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. The dye chosen should be known to not absorb to the membrane under study.

15. Apparatus

15.1 The test apparatus required is schematically described in Test Methods D4194.

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 E60. Spectrophotometers shall conform to Practice E275.

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 D4194.

17.2 Start up and operate the test system in accordance with Section 8 of Test Methods D4194.

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.