



## Standard Practice for Integrity Testing of Water Filtration Membrane Systems<sup>1</sup>

This standard is issued under the fixed designation D 6908; 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 approval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This standard covers the determination of the integrity of water filtration membrane elements and systems using air based tests (pressure decay and vacuum hold), soluble dye, continuous monitoring particulate light scatter techniques, and TOC monitoring tests for the purpose of rejecting particles and microbes. The tests are applicable to systems with membranes that have a nominal pore size less than about 1  $\mu\text{m}$ . The TOC, and Dye, tests are generally applicable to NF and RO class membranes only.

1.2 This standard does not purport to cover all available methods of integrity testing.

1.3 *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:<sup>2</sup>

- D 1129 Terminology Relating to Water
- D 2777 Determination of Precision and Bias of Applicable Tests Methods of Committee D19 on Water
- D 3370 Practices for Sampling Water from Closed Conduits
- D 3864 Guide for Continual On-Line Monitoring Systems for Water Analysis
- D 3923 Practice for the Determination of Leaks Within a Reverse Osmosis Device
- D 4839 Total Carbon and Organic Carbon in Water by Ultraviolet, or Persulfate Oxidation, or Both, and Infrared Detection
- D 5173 On-Line Monitoring of Carbon Compounds in Water by Chemical Oxidation, by UV Light Oxidation, by Both, or by High Temperature Combustion Followed by Gas Phase NDIR or by Electrolytic Conductivity

- D 5904 Total Carbon, Inorganic Carbon and Organic Carbon in Water by Ultraviolet, Persulfate Oxidation and Membrane Conductivity Detection
- D 5997 On-Line Monitoring of Total Carbon, Inorganic Carbon in Water by Ultraviolet, Persulfate Oxidation, and Membrane Conductivity Detection
- D 6161 Terminology Used for Crossflow Microfiltration, Ultrafiltration, Nanofiltration and Reverse Osmosis Membrane Processes
- D 6698 Test Method for the On-Line Measurement of Turbidity Below 5 NTU in Water
- E 20 Practice for Particle Size Analysis of Particulate Substances in the Range of 0.2 to 75 Micrometers by Optical Microscopy
- E 128 Maximum Pore Diameter and Permeability of Rigid Porous Filters for Laboratory Use
- F 658 Practice for Defining Size Calibration, Resolution, and Counting Accuracy of a Liquid-borne Particle Counter Using Nearmonodisperse Spherical Particulate Material

### 3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, refer to Terminologies D 6161 and D 1129.

3.1.1 For description of terms relating to cross flow membrane systems, refer to Terminology D 6161.

3.1.2 For definition of terms relating to dissolved carbon and carbon analyzers, refer to D 5173, D 5904 and D 5997.

3.1.3 *bubble point*—when the pores of a membrane are filled with liquid and air pressure is applied to one side of the membrane, surface tension prevents the liquid in the pores from being blown out by air pressure below a minimum pressure known as the bubble point.

3.1.4 *equivalent diameter*—the diameter of a pore or defect calculated from its bubble point using Eq 1 (see 9.3). This is not necessarily the same as the physical dimensions of the defect(s).

3.1.5 *integrity*—measure of the degree to which a membrane system rejects particles of interest. Usually expressed as a log reduction value (LRV).

3.1.6 *log reduction value (LRV)*—a measure of the particle removal efficiency of the membrane system expressed as the log of the ratio of the particle concentration in the untreated and treated fluid. For example, a 10-fold reduction in particle

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.08 on Membranes and Ion Exchange Materials.

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<sup>2</sup> 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.

concentration is an LRV of 1. The definition of LRV within this Standard is one of many definitions that are used within the industry. The user of this standard should use care as not to interchange this definition with other definitions that potentially exist. The USEPA applies the LRV definition to pathogens only.

3.1.7 *membrane system*—refers to the membrane hardware installation including the membrane, membrane housings, interconnecting plumbing, seals and valves. The membrane can be any membrane with a pore size less than about 1  $\mu\text{m}$ .

3.1.8 *multiplexing*—the sharing of a common set of physical, optical, and/or electrical components across multiple numbers of sensors. Each sensor is capable of monitoring a unique sample. Each sample is monitored separately and referenced against its unique baseline characteristics. Individual sensors can be used for each sample or samples can be serially routed to a common sensor for individual analysis.

3.1.9 *UCL*—a generic term to represent the aggregate quantity of material that causes an incident light beam to be scattered. The value can be correlated to either turbidity or to specific particle count levels of a defined size.

3.1.10 *relative standard deviation (RSD)*—a generic continuous monitoring parameter used to quantify the fluctuation of the particulate light scatter baseline from a laser-based incident light source. As an example, the RSD may be calculated as the standard deviation divided by the average for a defined set of measurements that are acquired over a short period of time. The result is multiplied by 100 to express the value as a percentage and is then reported as % RSD. The sample monitoring frequency is typically in the range of 0.1 to 60 seconds. The RSD parameter is specific for laser-based particulate light-scatter techniques which includes particle counters and laser turbidimeters. The RSD is can be treated as an independent monitoring parameter. Other methods for RSD calculations may also be used.

#### 4. Significance and Use

4.1 The integrity test methods described are used to determine the integrity of membrane systems, and are applicable to systems containing membrane module configurations of both hollow fiber and flat sheet; such as, spiral-wound configuration. In all cases the practices apply to membranes in the RO, NF, and UF membrane classes. However, the TOC and Dye Test practices do not apply to membranes in the MF range or the upper end of the UF pore size range (0.01  $\mu\text{m}$  and larger pore sizes) due to insignificant or inconsistent removal of TOC material by these membranes.

4.2 These methods may be used to identify relative changes in the integrity of a system, or used in conjunction with the equations described in 9.4, to provide a means of estimating the integrity in terms of log reduction value. For critical applications, estimated log reductions using these equations should be confirmed by experiment for the particular membrane and system configuration used.

4.3 The ability of the methods to detect any given defect is affected by the size of the system or portion of the system tested. Selecting smaller portions of the system to test will increase the sensitivity of the test to defects. When determining the size that can be tested as a discrete unit, use the guidelines supplied by the system manufacturer or the general guidelines provided in this standard.

4.4 The applicability of the tests is largely independent of system size when measured in terms of the impact of defects on the treated water quality (that is, the system LRV). This is because the bypass flow from any given defect is diluted in proportion to the systems total flowrate. For example, a 10-module system with a single defect will produce the same water quality as a 100-module system with ten of the same size defects.

#### 5. Reagents and Materials

5.1 *Reagents*—As specified for the TOC analyzer in question. D 5173 lists requirements for a variety of instruments.

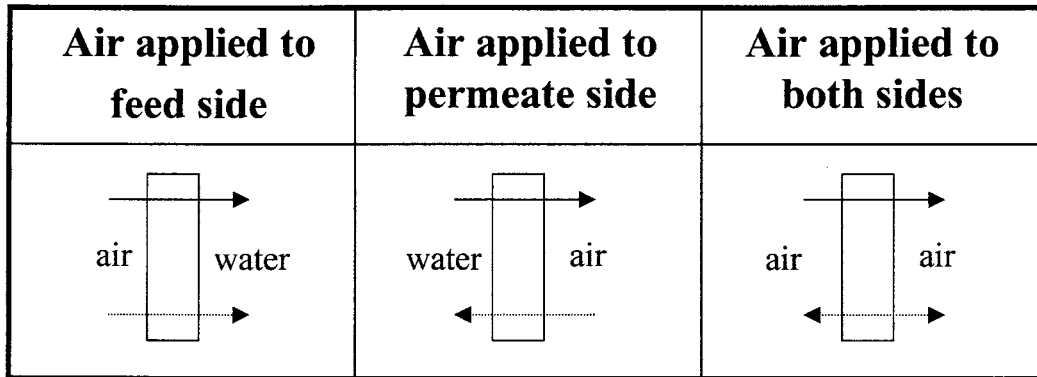
5.2 *Soluble Dye Solution*—Use FD&C or reagent grade dyes such as FD&C Red #40, dissolved in RO permeate, or in ASTM Reagent Grade Type IV water.

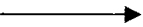
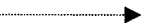
5.3 *Light Scatter Standards*—See Test Method D 6698 for the selection of appropriate turbidity standards. In addition, polystyrene latex standards of a defined size and concentration may be used in place of a turbidity standard as long as count concentration is correlated to instrument response.

5.4 *Light Obscuration Standards*—Standards that are used for the calibration of particle counters, namely polystyrene latex spheres should be used. Consult the instrument manufacturer for the appropriate type and size diameter of standards to be used.

#### 6. Precision and Bias

6.1 Neither precision nor bias data can be obtained for these test methods because they are composed of continuous determinations specific to the equipment being tested. No suitable means has been found of performing a collaborative study to meet the requirements of Practice D 2777. The inability to obtain precision and bias data for methods involving continuous sampling or measurement of specific properties is recognized and stated in the scope of Practice D 2777.



 Normal water permeation direction  
 Air leaking direction

NOTE—The last example also represents the vacuum decay test when a partial vacuum is applied to one side of the membrane.  
**FIG. 1 Various Configurations for the Pressure Decay Test**

**PRACTICE A—PRESSURE DECAY AND VACUUM DECAY TESTS**

**7. Scope**

7.1 This practice covers the determination of integrity for membrane systems using the pressure decay test (PDT) and vacuum decay test (VDT).

7.2 The tests may be used on membranes in all classes, RO through MF, and are suitable for hollow fibers, tubular and flat sheet (such as spiral wound) configurations. However, the PDT is most commonly employed for in-situ testing of UF and MF systems and the VDT for testing NF and RO elements and systems. See Practice D 3923.

**8. Summary of Practice**

8.1 *Principles*—The tests work on the principle that if air pressure is applied to one side of an integral, fully wet membrane at a pressure below the membrane bubble point, there will be no airflow through the membrane other than by diffusion through liquid in the membrane wall. If a defect or leak is present then air will flow freely at this point, providing that the size of the defect is such that it has a bubble point pressure below the applied test pressure. The configurations for applying air and water are shown in Fig. 1.

8.1.1 Air based tests are means of applying air, at a pressure below the membrane bubble point, to one side of a wet membrane and measuring the air flow from one side to the other. Air flow can be measured directly, but more commonly, it is derived from pressure or vacuum decay. In the PDT air flow is measured as the rate of pressure decay when one side of a membrane system (either the feed or filtrate side) is isolated and pressurized with air. In the VDT an air pressure differential is generated by isolating one side of a wet membrane and applying a partial vacuum with atmospheric pressure on the other side. Air flow is measured as the rate of vacuum

decay on the isolated side of the membrane. The results of both the PDT and VDT are a direct measure of the membrane system integrity.

8.2 *Limitations and Applications*—The tests are limited to monitoring and control of defects greater than about 1 to 2 μm (see 9.3, Selection of Test Pressure).

8.2.1 The tests can be applied in various forms provided a differential pressure below the bubble point is established across a wet membrane with air on the relative high pressure side of the membrane. Some examples are included in Fig. 1.

8.2.2 Both the PDT and VDT are described here in their most common forms. In the case of the PDT this is with one side of the membrane pressurized with air and the other filled with liquid vented to atmosphere. In the case of the VDT, air is typically present on both sides and vacuum is applied to the permeate side.

**9. Procedure**

9.1 *Pressure Decay Test (PDT)*—The pressure decay test can be carried out by pressurizing either side of the membrane (see Fig. 1). For complete wet-out of all the membrane in the system, the system should be operated at its normal pressure before the test is performed. The steps involved in the PDT are:

9.1.1 Drain the liquid from the side of the membrane to be pressurized (referred to here as the upstream side).

9.1.2 Open the downstream side of the membrane system to atmosphere. This ensures air that leaks or diffuses is free to escape without creating backpressure, and establishes the downstream pressure as atmospheric pressure.

9.1.3 Isolate and pressurize the upstream side with air to the test pressure. Then isolate the air supply. Do not exceed the test pressure as this could lead to blowing out smaller pores than

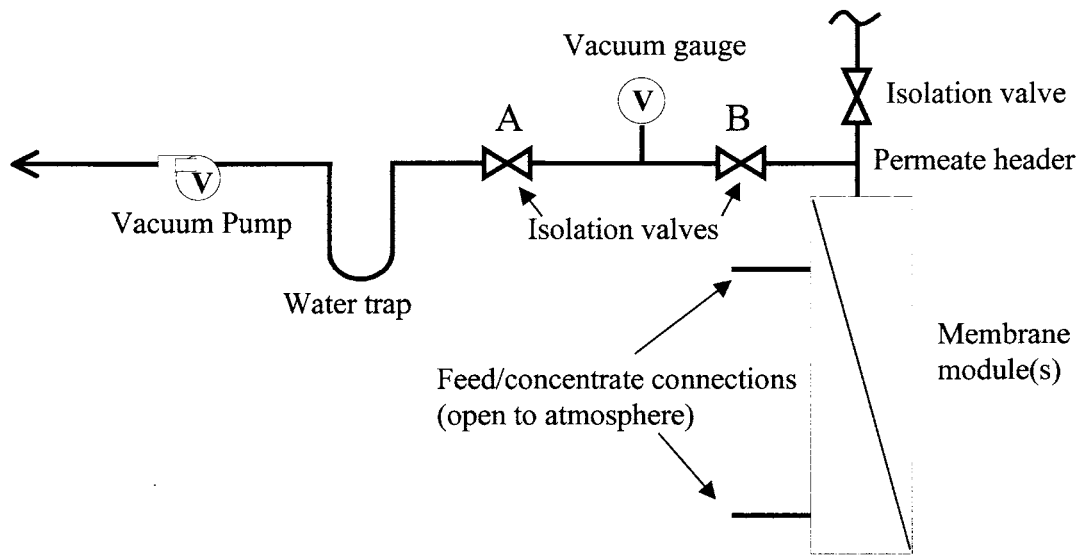


FIG. 2 Connection Arrangement for the VDT

intended resulting in a higher PDT. Record this pressure as  $P_{test,max}$  the maximum test pressure.

9.1.4 After allowing time for the decay rate to stabilize record the initial pressure,  $P_i$ , and commence timer.<sup>3</sup>

9.1.5 After at least 2 min, record the final pressure,  $P_f$ , and the time taken for the pressure to decay from  $P_i$  to  $P_f$  ( $t$ ). The time period can be extended in order obtain a more accurate result if the pressure decay rate is slow.<sup>4</sup>

9.1.6 Calculate the Pressure Decay Rate (PDR) as follows and record the result along with the test conditions (temperature, average test pressure  $P_{test,avg}$  and maximum pressure  $P_{test,max}$ ):

$$PDR_{measured} = \frac{P_i - P_f}{t}$$

where:

$PDR_{measured}$  = measured pressure decay rate, kPa/min at the average test pressure,  $P_{test,ave} = P_i + P_f / 2$ ,

$P_i$  = initial pressure, kPa gauge,

$P_f$  = final pressure, kPa gauge,

$t$  = time taken for pressure to decay from  $P_i$  to  $P_f$  mins, and

$P_{test,max}$  = maximum test pressure given as the pressure at the start of the test, kPa.

9.1.7 The PDR will result from diffusion through the membrane wall, as well as leaks through defects, damaged membranes, or seals. The diffusive component of the airflow is not related to the integrity, so a more accurate estimate of the nondiffusive pressure decay can be obtained by subtracting the diffusive flow from the measured flow. The diffusive compo-

nent can be estimated either by calculation or experimental determination of the diffusive flow, such as laboratory measurements or by measuring the PDR on a system confirmed suitably integral by other means. In such cases, the measured PDR result is corrected as follows:

$$PDR_{corrected} = PDR_{measured} - PDR_{diffusion}$$

where:

$PDR_{diffusion} = PDR_{measured}$  for the integral system, at the same  $P_{Test}$  and temperature.

9.1.8 For most practical applications of the test sufficient accuracy can be obtained by taking the conservative approach and assuming that all the pressure decay is related entirely to leaks ( $PDR_{diffusion} = 0$ ).

9.2 Vacuum Decay Test—The VDT is conducted with air on both sides of the membrane. For complete wet-out of all the membrane in the system, the system should be operated at its normal pressure before the test is performed. The steps involved in the VDT are:

9.2.1 Drain the liquid from the feed side of the membrane (referred to here as the upstream side), 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 to work.

9.2.2 Use the equipment connected in this order (see Fig. 2): a vacuum pressure gauge, an isolation valve, a water trap that will not buckle at vacuum, and a vacuum pump, to the permeate manifold that serves one or more membrane devices. Addition of another isolation valve (B) at the permeate header allows easy connection of the equipment without disrupting operation of the membrane system.

9.2.3 Open isolation valves A and B and run the vacuum pump to evacuate the permeate side until the pressure gauge shows a stable vacuum. The water removed during this operation is collected in the water trap. Close isolation valve A. Start the stopwatch and record the initial vacuum ( $P_i$ ). The test vacuum can be selected using the guidelines in 9.3.

<sup>3</sup> The pressure decay rate at the start of the test is usually quite high due to displacement of some of the liquid in the membrane wall. The time taken for the decay rate to stabilize will be different for different systems, but may take up to 3 min.

<sup>4</sup> Due to the nonlinear decay in pressure with time and the desire to simplify the equations by using the first order approximation for decay rate, the maximum time should be such that  $P_f$  is no more than 10 % lower than  $P_i$ .

9.2.4 After the determined time (60 s is a typical time, 120, 180 or 300 s will yield a more sensitive test) record the final pressure ( $P_f$ ) and the time ( $t$ ) for reaching this value.<sup>4</sup>

9.2.5 Calculate the Vacuum Decay Rate (VDR) as follows:

$$VDR_{measured} = \frac{P_f - P_i}{t}$$

where:

- $VDR_{measured}$  = measured vacuum decay rate, kPa/min at the average test pressure,  $P_{test,ave} = P_i + P_f / 2$ ,  
 $P_i$  = initial vacuum, kPa gauge,  
 $P_f$  = final vacuum, kPa gauge,  
 $t$  = time taken for vacuum to decay from  $P_i$  to  $P_f$ , mins, and  
 $P_{test,max}$  = maximum test vacuum given as the pressure at the start of the test, kPa.

9.2.6 The VDR will result from diffusion through the membrane wall, as well as leaks through defects, damaged membranes, or seals. The diffusive component of the airflow is not related to the integrity, so a more accurate estimate of the nondiffusive vacuum decay can be obtained by subtracting the diffusive flow from the measured flow. The diffusive component can be estimated either by calculation or experimental determination of the diffusive flow, such as laboratory measurements or by measuring the VDR on a system confirmed suitably integral by other means. In such cases, the measured VDR result is corrected as follows:

$$VDR_{corrected} = VDR_{measured} - VDR_{diffusion}$$

where:

- $VDR_{diffusion}$  =  $VDR_{measured}$  for the integral system, at the same  $P_{test}$  and temperature.

If  $VDR_{diffusion}$  is unknown, the conservative approach is to set  $VDR_{diffusion} = 0$ .

9.3 Selection of Test Pressure—The test pressure selected determines the minimum equivalent diameter of a defect that can contribute to the pressure or vacuum decay rate. The relationship between the test pressure and the equivalent defect diameter is given by Eq 1. Defects smaller than this will be too small for the bubble point to be overcome and thus will not contribute to airflow. Larger defects will allow airflow as the bubble point will be exceeded by the applied test pressure. Details on the derivation of this equation and its use in determining maximum pore size for membranes can be found in Method E 128.<sup>5</sup>

$$d = \frac{4\gamma \cos\theta}{\Delta P_{test,max}} \quad (1)$$

where:

- $\Delta P_{test,max}$  = the maximum differential test pressure applied across the membrane. This is the  $P_{test,max}$  recorded during the test corrected for any static head contribution,  
 $\gamma$  = surface tension at the air-liquid interface,  
 $\theta$  = liquid-membrane contact angle, and  
 $d$  = equivalent diameter of the smallest defect included in the test.

9.3.1 For the theoretical case of a perfectly hydrophilic membrane, the contact angle is zero, and assuming water at 25°C (surface tension 72 dynes/cm), Eq 1 simplifies to Eq 2, with  $d$  in micrometres and  $P_{test,max}$  in kilopascal:

$$d = \frac{288}{\Delta P_{test,max}} \quad (2)$$

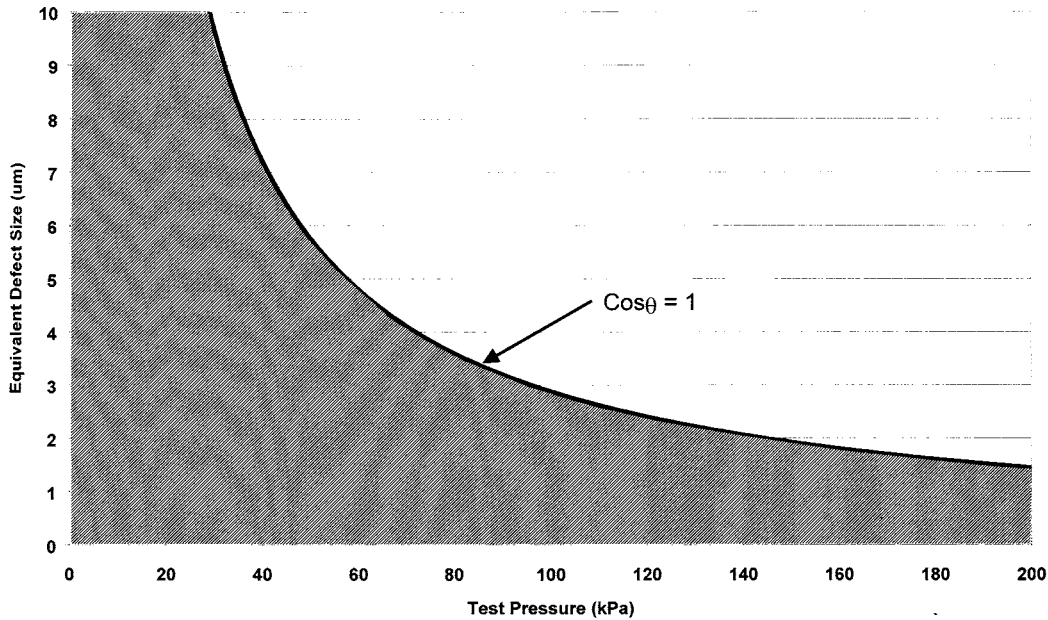
9.3.2 Fig. 3 shows the relationship between test pressure and equivalent defect diameter expressed by Eq 1 and assuming a surface tension of 72 dynes/cm. The solid line represents Eq 2; that is, the conservative situation of  $\cos\theta = 1$ . In practice most membranes used in water treatment have a contact angle greater than zero, which is represented by the shaded region under the solid line in Fig. 3. If the contact angle is known or can be determined, Eq 1 may be used. However, if the contact angle is not known, a conservative estimate of the test pressure required can be made by applying Eq 2.

9.3.3 The test pressure is usually selected to ensure that the minimum defect diameter picked up by the test is smaller than contaminates or particles of interest. For example, Eq 2 indicates that a test pressure of 100 kPa would include all defects larger than or equal to 3  $\mu\text{m}$ . A lower pressure could be used for less hydrophilic membranes. For example, if the contact angle is 60 degrees (typical for polypropylene, polysulfone, or PVdF) Eq 1 indicates that defects of 3  $\mu\text{m}$  would be included at a test pressure of 50 kPa. An even lower test pressure may be used for larger defects, such as for example detection of broken fibers in a hollow fiber system.

9.3.4 In practice the applied test pressure is rarely more than 300 kPa, which is usually sufficient to include defects smaller than most pathogens of interest. At this pressure limit the test is not suitable for direct validation of virus rejection as these particles are very small (typically less than 0.01  $\mu\text{m}$ ) with a corresponding test pressure of several thousand kilopascals.

9.4 Interpreting PDR and VDR Results as Log Reduction Values—Both the PDR and the VDR are measurements of the airflow from one side of the membrane to the other under a known set of test conditions (temperature and pressure). This information can be used to estimate the flow of liquid through the same defects during filtration conditions. This provides an estimate of the membrane bypass flow and thereby an estimate of the log removal of particles for the system. One approach is based on the Hagen-Poiseuille law, which assumes laminar flow through cylindrical defects. Whilst this method provides a useful estimate, its applicability is limited to small fibers (< 400  $\mu\text{m}$  ID) where the criteria for laminar flow are more closely approximated. The method is described in 9.4.1 and a detailed derivation, along with the assumptions required, is contained in Appendix X1. An alternative method is to experimentally

<sup>5</sup> Eq 1 is often modified to include a correction factor referred to as the pore shape factor or the Bechold Constant. This is a value < 1 and takes into account the irregular shape of membrane pores. For the purpose of this practice the shape factor is assumed to be 1 as this is the most conservative position, and the shape of any particular defect detected by these tests is not known.



NOTE—The solid line represents Eq 2.

FIG. 3 The Relationship Between Test Pressure and Equivalent Defect Diameter (Eq 1, Water at 25°C)

measure the relationship between liquid and air flows for the worst case failure mode. This is typically a broken fiber at the pot for most hollow fiber MF or UF systems. This approach, described in 9.4.3, assumes that all the measured gas flow is due to “worst case” failures and so provides a conservative estimate of bypass flow and LRV for the system. While these approaches have been applied in practice, data covering a range of different membrane configurations, test conditions, and fiber diameters are not yet available. Regardless of the chosen method the relationship between integrity test results and LRV should be verified by experiment in the field on the particular membrane and configuration used.

9.4.1 *The Laminar Flow Approach Using the Hagen-Poiseuille (H-P) Law*—This approach assumes laminar flow through cylindrical defects and is most suitable for small diameter fibers (200 to 400 µm lumen diameter). A detailed derivation along with key assumptions is contained in Appendix X1. The equations required to convert the PDR and VDR results obtained using the method described here to a log reduction value, are given below as Eq 3 and 4 respectively:

For PDR:

$$LRV_e = \log_{10} \left( \frac{Q_{filt} P_{atm}}{CF \cdot PDT \cdot V_{system}} f_1 f_2 \right) \quad (3)$$

and for VDR:

$$LRV_e = \log_{10} \left( \frac{Q_{filt} P_{atm}}{CF \cdot VDT \cdot V_{system}} f_1 f_2 \right) \quad (4)$$

where:

- $f_1$  = viscosity correction factor =  $\mu_{water} / \mu_{air}$
- $f_2$  = pressure correction factor =  $\frac{P_{u,test}^2 - P_{d,test}^2}{2P_{atm} TMP}$
- $Q_{filt}$  = filtrate flowrate (m<sup>3</sup>/s),

- $P_{u,test}$  = upstream pressure during the PDT or VDT =  $P_{test,avg}$  for PDT and  $P_{atm}$  for VDT, (kPa absolute),
- $P_{d,test}$  = downstream pressure during the PDT or VDT =  $P_{atm}$  for PDT and  $P_{test,avg}$  for VDT, (kPa absolute),
- $P_{atm}$  = atmospheric pressure (kPa absolute),
- $CF$  = concentration factor. This represents the increase in the contaminant concentration that could occur on the upstream side of the membrane relative to the feed water concentration due to the operating mode. This would typically be equal to 1 for dead-end systems, but could be higher for cross flow or feed and bleed modes,
- $PDR$  = pressure decay rate (kPa/s),
- $VDR$  = vacuum decay rate (kPa/s),
- $TMP$  = transmembrane pressure during filtration (kPa),
- $V_{system}$  = volume pressurised (or under vacuum) during test (m<sup>3</sup>),
- $\mu_{water}$  = the viscosity of the liquid during filtration (Pa·s),
- $\mu_{air}$  = the viscosity of the air during the test (Pa·s), and
- $LRV_e$  = estimated log reduction value.

9.4.2 *Example Calculation of the Log Reduction of Particles from the PDT Using the H-P Approach*—Estimate the LRV for a membrane system operating at a filtrate flowrate of 50 L/s and a transmembrane pressure of 70 kPa. The water temperature is 20°C, and the PDR for the system is 2.5 kPa/min at 100 kPa test pressure and 27°C. The system is operating in dead-end mode so  $CF = 1$ . The viscosity of water at 20°C is  $1.00 \times 10^{-3}$  Pa·s and air at 27°C is  $1.84 \times 10^{-5}$  Pa·s. The pressurized system volume during the PDT is 400 L.

First calculate  $f_1$  and  $f_2$ :

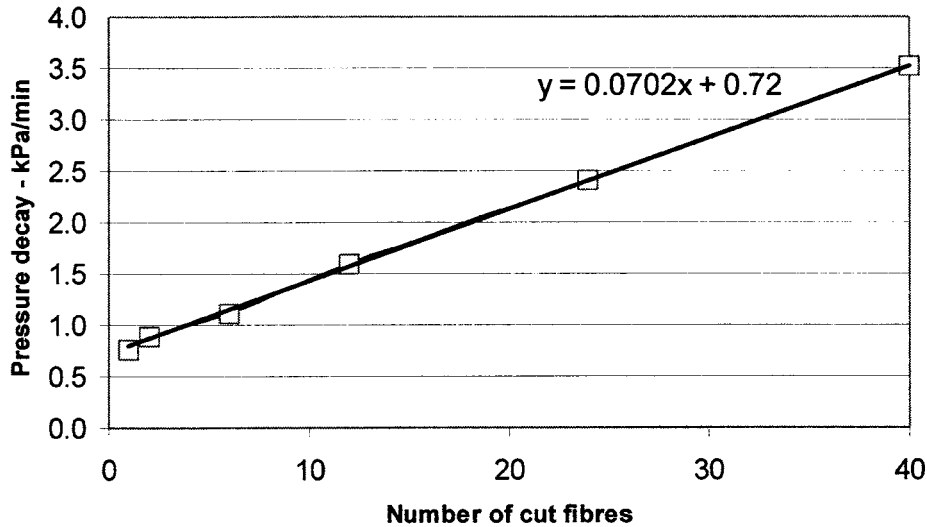


FIG. 4 PDR Values

$$f_1 = \frac{\mu_{water}}{\mu_{air}} = \frac{1.00 \times 10^{-3}}{1.84 \times 10^{-5}} = 54.35$$

$$f_2 = \frac{P_{u,test}^2 - P_{d,test}^2}{2P_{atm} TMP} = \frac{(201.3 \text{ kPa})^2 - (101.3 \text{ kPa})^2}{2 \cdot 101.3 \text{ kPa} \cdot 70 \text{ kPa}} = 2.13$$

Estimate the LRV from Eq 3 as follows:

$$LRV_e = \log_{10} \left( \frac{Q_{filt} P_{atm}}{CF \cdot PDT \cdot V_{system}} f_1 f_2 \right)$$

$$= \log_{10} \left( \frac{50 \times 10^{-3} \text{ m}^3/\text{s} \cdot 101.3 \text{ kPa}}{1 \cdot 2.5/60 \text{ kPa/s} \cdot 400 \times 10^{-3} \text{ m}^3 \cdot 54.35 \cdot 2.13} \right)$$

$$= 4.5$$

Note that from Eq 2 the test pressure of 100 kPa equates to a minimum defect size of 2.9  $\mu\text{m}$  (conservatively). So the LRV of 4.5 calculated above is the minimum LRV for particles greater than 2.9  $\mu\text{m}$  diameter.

9.4.3 *Experimental Approach to Correlating Test Results and System LRV Using Equivalent Number of Broken Fibers*— This approach relies on measuring the relationship between gas flow and bypass flow for “worst case” defects for hollow fiber systems, and assuming that all bypass will be through such defects. This approach provides a conservative estimate of LRV that can be applied to most membrane diameters and configurations. For hollow fiber membrane systems the worst case failure will usually be a fiber that is cut cleanly at the fiber-pot interface. This provides the shortest bypass path and the largest possible diameter. The steps involved are:

(1) Experimentally determine the gas flow through a single fiber, cut at the pot, at the selected test pressure (call this  $Q_{G,atm,fiber}$ ). Preferably this is carried out in field tests using one or more modules of the full-scale design, or alternatively in a laboratory using the same membrane fiber and potting materials.

(2) For the same configuration determine the water flow through the lumen ( $Q_{L,fiber}$ ) at a range of pressures to establish the bypass flow vs TMP curve for a single fiber. This can be done experimentally using short fiber lengths in the laboratory, or by theoretical calculation combined with experimental determination of friction factor (for turbulent flow).

(3) Evaluate the system LRV using the following:

(a) Measure the PDR (or VDR) for the system. Calculate the gas flow using Eq 5 (for PDT) or Eq 6 (for VDT). Note that these are the equations derived as Eq X1.4 and X1.5 in Appendix X1.

$$Q_{G,atm} = PDR \frac{V_{system}}{P_{atm}} \quad (5)$$

$$Q_{G,atm} = VDR \frac{V_{system}}{P_{atm}} \quad (6)$$

(b) Calculate the equivalent number of broken fibers for the system (see Fig. 4) as:

$$N_{equivalent} = \frac{Q_{G,atm}}{Q_{G,atm,fiber}} \quad (7)$$

(c) Calculate the liquid bypass flow,  $Q_{bypass}$  by multiplying the equivalent number of broken fibers by the flow per fiber at the operating TMP (from the data generated in step 2):

$$Q_{bypass} = N_{equivalent} \times Q_{L,fiber} \quad (8)$$

Eq 5 can be written for an individual fiber as  $Q_{G,atm,fiber} = PDR_{fiber} V_{system} / P_{atm}$  where  $PDR_{fiber}$  is the pressure decay rate corresponding to  $Q_{G,atm,fiber}$ . Combining with Eq 7 and 8 gives:

$$Q_{bypass} = \frac{PDR_{corrected}}{PDR_{fiber}} \cdot Q_{L,fiber} \quad (9)$$

(d) Calculate the estimated LRV using Eq 10 (also Eq X1.2):

$$LRV_e = \log_{10} \left( \frac{Q_{filt}}{Q_{bypass}} \right) \quad (10)$$

Substituting Eq 9 into Eq 10:

$$LRV_e = \log_{10} \left( \frac{PDR_{fiber} \cdot Q_{filt}}{PDR_{corrected} \cdot Q_{L,fiber}} \right) \quad (11)$$

A similar derivation for VDT gives:

$$LRV_e = \log_{10} \left( \frac{VDR_{fiber} \cdot Q_{filt}}{VDR_{corrected} \cdot Q_{L,fiber}} \right) \quad (12)$$

The values for  $Q_{G,atm,fiber}$  and  $Q_{L,fiber}$  can be calculated using known hydraulic formulae (such the Darcy-Weisbach equations) including consideration of entrance and exit losses,

however for nonlaminar flow situations solving these requires an iterative approach as well as establishing values for surface roughness which must be experimentally determined. When using theoretical calculation of  $Q_{L,fiber}$ , consideration should also be given to flow through the free end of the cut fiber as well as the pot, although in most cases this will be small compared to the flow through the pot.

**9.4.4 Example of the Experimental Method Using the Equivalent Number of Broken Fibers**—The following example is taken from data presented in Kothari and St. Peter.<sup>6</sup> The filtration unit is a hollow fiber microfilter using membranes with an internal diameter of 250  $\mu\text{m}$ . Results from a study looking at the impact on PDR of cutting fibers are presented. Fibers were cut near the pot, giving a cut fiber length of approximately 125 mm, with the long end of the fiber approximately 1035 mm. Temperature is assumed to be 5°C (viscosity  $1.62 \times 10^{-3}$  Pa·s), with a filtrate flow of 120 000 L/h. Data up to 400 cut fibers is presented, although only the data up to 40 cut fibers is used here as the test pressure was reasonably constant between tests at an average of 100 kPa.

Number of Cut Fibers	PDR (kPa/min)	PDR Starting Pressure (kPa)
0	0.69	101.8
1	0.76	101.7
2	0.90	101.6
6	1.10	100.9
12	1.58	100.3
24	2.41	98.4
40	3.51	95.8

**Step 1. Determine the Relationship Between Gas Flow and Fibers Cut at the Pot**—In order to do this the above PDR values are plotted producing the graph shown in Fig. 4. The slope of the line of best fit represents the change in pressure decay for each cut fiber, and the intercept represents the gas flow due to diffusion only (at 100 kPa test pressure). This could be converted to a gas flow using Eq 5, however for this example it is more useful to leave it as a PDR per cut fiber.

**Step 2. Determine the Liquid Flowrate Through a Single Broken Fiber at the Pot**—In this case we will calculate the flowrate from theory, although it could also be determined by laboratory measurement. Using Eq X1.7 for laminar flow in hollow cylinders at a filtration TMP of 50 kPa, including allowance for both ends of the cut fiber, gives:

$$Q_{L,fiber} = \frac{\pi d^4 TMP}{128 L \mu}$$

$$= \frac{\pi(250 \times 10^{-6} \text{ m})^4 \cdot 50 \times 10^3 \text{ Pa}}{128 \cdot 1.62 \times 10^{-3} \text{ Pa}\cdot\text{s}} \cdot \frac{1000 \text{ L}}{\text{m}^3} \cdot \frac{3600 \text{ s}}{\text{h}}$$

$$\cdot \left( \frac{1}{0.125 \text{ m}} + \frac{1}{1.035 \text{ m}} \right) = 0.095 \text{ L/h}$$

Checking Reynolds number confirms this is laminar flow and hence the equation is valid. An allowance for entrance and exit losses could be made, however, given the low Reynolds number this correction will be minor and the value as calculated above is conservative.

**Step 3. Calculate the Relationship Between PDR and Bypass Flowrate**—Using Eq 11 gives:

$$LRV_e = \log_{10} \left( \frac{PDR_{fiber} \cdot Q_{filt}}{PDR_{corrected} \cdot Q_{L,fiber}} \right)$$

$$= \log_{10} \left( \frac{0.0702 \times 120\,000 \text{ L/h}}{PDR_{corrected} \times 0.095 \text{ L/h}} \right)$$

$$= \log_{10} \left( \frac{88\,674}{PDR_{corrected}} \right)$$

$$= 4.95 - \log_{10} (PDR_{corrected})$$

$$= 4.95 - \log_{10} (PDR_{measured} - 0.72)$$

The estimated LRV's using the above equation are tabulated below for varying numbers of cut fibers. The LRV's calculated according to the H-P method (as described in 9.4.1) are also included for comparison. The difference between the two methods of estimating the LRV is small in this case (0.05 to 0.1 log). As the fiber diameter increases the limitations of the assumptions involved in the H-P method will become greater, and the experimental approach might be more suitable. Particle count data are also included to indicate the difficulty of using conventional water quality methods to verify integrity at these levels.

No. of Cut Fibers	PDT (kPa/min)	LRV <sub>e</sub> Equivalent Broken Fibers Method (see 9.4.3)	LRV <sub>e</sub> H-P Method (see 9.4.1)	Total Particle Count (counts/mL)
0	0.69			1.40
1	0.76	6.37	6.47	1.07
2	0.90	5.70	5.80	7.50
6	1.10	5.37	5.46	2.60
12	1.58	5.01	5.10	3.00
24	2.41	4.72	4.79	1.30
40	3.51	4.50	4.55	2.30

<sup>6</sup> Kothari, H., St. Peter, E., "Utility Perspective on Regulatory Approval for Microfiltration Treatment Facilities in Wisconsin," Proceedings of AWWA Annual Conference, June 11-15 2000, Denver, CO.



## PRACTICE B—USE OF TOTAL ORGANIC CARBON ANALYZERS FOR MONITORING INTEGRITY OF REVERSE OSMOSIS OR NANOFILTRATION MEMBRANE SYSTEMS

### 10. Scope

10.1 This practice is applicable where the membrane system and water source will allow the monitoring of TOC both upstream and downstream of the system, and at least order of magnitude difference from the feed can be measured in the permeate (product) water. See [D 4839](#).

### 11. Summary of Practice

11.1 *Carbon Analysis Summary*—There are two processes involved in TOC analysis—first dissolved carbon is oxidized to CO<sub>2</sub> and then the concentration of CO<sub>2</sub> is detected and the result is interpreted using a customized calibration curve. To eliminate interference from inorganic carbon (carbonate, bicarbonate, and dissolved CO<sub>2</sub>) the sample is split into two streams. Both streams are acidified to convert inorganic carbon (IC) to CO<sub>2</sub>, and one stream is treated further to oxidize the organic carbon to CO<sub>2</sub>. The samples are sent to separate CO<sub>2</sub> detectors—one for IC and one for Total Carbon (TC). TOC is the difference between the TC and IC results. [D 5173](#) and [D 5997](#) give detailed descriptions of the various techniques used to perform on-line monitoring of carbon compounds in water. Instruments using these methods require approximately six minutes to analyze one sample.

11.2 *Sampling from the Permeate Stream*—Practices [D 3370](#) describes standard practices for sampling water from closed conduits. A side stream from the permeate line is diverted to the TOC analyzer. The length of this line should be as short as possible. Most analyzers have a flushing cycle between samples and by-pass during analysis, which is diverted to drain. The volume of sample is very small compared to the by-pass flow (as little as 0.35 mL/min versus 30 to 220 mL/min for flush).

11.3 *Establishing Baseline Data*—When the system has stabilized after start-up, the feed, permeate and concentrate streams are analyzed for TOC concentration. If the instrument used can handle the range in concentrations, with different calibration curves, then it is best to use the same instrument as will be used for integrity monitoring. The instrument can be used off line in grab sample mode for these tests. It is important to perform enough repeat sample analyses to ensure the sample lines are completely filled with the test solution. Testing the permeate sample first will make this task easier. Sample size should be large enough to reflect normal variations due to temperature and time of day.

11.4 *Concentrate Sampling*—The concentrate stream is tested to determine the system's mass-balance. It may be that organic carbon is adsorbing to the membrane. If so, there may be break-through later on when all adsorption sites are taken up and a new permeate baseline will be necessary.

11.5 *TOC Monitoring*—Follow instructions for the particular TOC analyzer in service. Be sure to keep the power on, chemicals fresh, pre-filters clean and UV or IR sources in good working order. Become familiar with the data output for your analyzer. It should provide the time, alarms, cause of the alarm, alerts when analysis conditions have been changed and a

description of the new conditions. View permeate TOC concentration on a graph with the feed and permeate baseline concentrations marked.

11.5.1 *Decision Point*—A decision point must be established for your particular process depending on the degree of risk associated with a breach of integrity.

11.5.2 *Variability*—Process fluctuations, temperature, changes in chemical cartridges, fouling of the TOC analyzer inlet pre-filter, changes in flow to the analyzer can all affect the TOC analysis. The degree of variability depends on the process and operation of the analyzer. The decision point should not be reached due to normal process variability.

### 12. Significance and Use

12.1 TOC Monitoring can be used effectively when the difference between average feed and product TOC concentration is at least one order of magnitude. TOC monitoring, as a tool for monitoring integrity, is used to identify relative changes in the integrity of a system. The sensitivity of the method is dependent on:

12.1.1 The capabilities of TOC instrument,

12.1.2 The size of the system as measured by permeate flow, and

12.1.3 The change in permeate TOC concentration that corresponds to a significant leak.

12.2 TOC analyzers are affected by conditions outlined below. For interference specific to a particular analyzer, contact the manufacturer. A baseline permeate TOC level must be established within the limits of the instrument that is still significantly different from the challenge or average feed concentration by one order of magnitude.

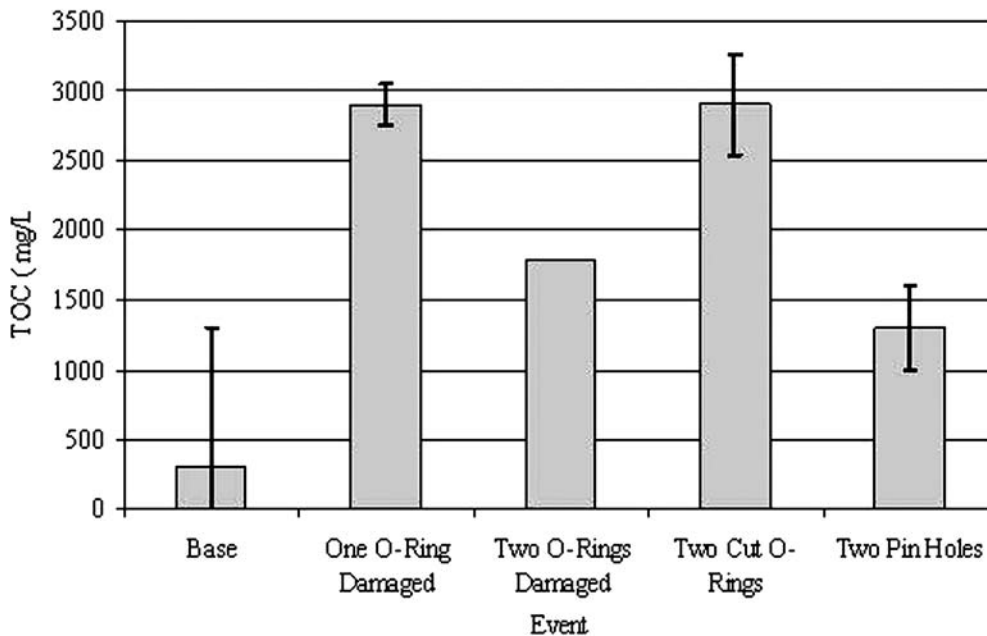
12.3 The size of the system monitored by one sample point should be determined using a risk/cost analysis. The risk is the potential for harm or legal action if there is a leak in the system. The cost is the price of additional sample points or additional analyzers.

12.4 The change in permeate TOC concentration corresponding to a significant leak (as defined by the risk/cost analysis) will depend on the volume of permeate produced by intact membrane in the monitored unit.

12.5 When determining the size that can be tested as a discrete unit, consider the change in TOC concentration expected from a leak that should initiate action. The change should be greater than 3 standard deviations of the average product concentration measured for that system. [Fig. 5](#) shows change in permeate TOC concentration in an RO system with different types of damage. The feed and concentrate concentrations were approximately 5 and 10 mg/L, respectively.

### 13. Interferences

13.1 *Changes in Inorganic Carbon Concentration*—Instability in the pretreatment acidification process can cause fluctuations in the inorganic carbon concentration of the permeate stream. If adjustment is not made in the acidification process to drive off excess IC, then the TOC results will be high.



TOC concentration during damage events. TOC does detect damage reliably. Value for damage event B is from one sample.

NOTE—Error bars indicate 3 standard deviations from the average (Chapman and Linton).<sup>7</sup>

**FIG. 5 Change in TOC Concentration with Different Types of Damage**

13.2 *Changes in Background Conductivity*—Changes in sample background conductivity will corrupt the comparison of CO<sub>2</sub> conductivity with the calibration curve. Since TOC analyzers can be much more sensitive than conductivity sensors, breaches in integrity should be detected due to increase in TOC concentration before there is a significant change in permeate conductivity.<sup>7</sup>

13.3 *Particulates*—Particles suspended in the water stream may cause blockage in the monitor over time.

<sup>7</sup> Chapman, M. W., Linton, K., “Evaluation of Integrity Monitoring Methods for Reverse Osmosis Membrane Systems,” 4/2000, USBR-DWPR Report #55. Available from S. Martella, USBR M.S. D-8230, P.O. Box 25007, Denver, CO 80225-0007.

**14. Apparatus**

14.1 D 5173 shows block diagrams of several designs of on-line TOC analyzers that have been introduced successfully.

**15. Interpretation of Results**

15.1 Permeate and feed (or average of feed and concentrate) TOC concentrations should be plotted over time. Using the feed concentration will provide the more conservative benchmark and simplify the procedure.

15.2 When the system has stabilized after start-up, calculate the standard deviation of the permeate and feed TOC concentrations. If permeate concentration exceeds three standard deviations from the average, check the system to determine the cause (see Fig. 6).

**PRACTICE C—SOLUBLE DYE TEST**

**16. Scope**

16.1 This guide is applicable to RO and NF membrane systems, including those with spiral, tubular or flat sheet configuration elements. The guide describes the application of two soluble dyes, Red Dye # 40 and Rhodamine WT. Both dyes have a molecular weight of approximately 500. See Practice D 3923.

**17. Summary of Practice**

17.1 This test works on the principle that a dissolved dye that is nearly completely rejected by an intact membrane element will pass through a membrane or seal defect into the

permeate at an increased rate that indicates a leak that is capable of passing significant amounts of microbial material.

17.2 A solution of controlled concentration of a dye, known to be rejected at a rate of 99.0 % or greater ( $\geq 2$  log) by the membrane, is circulated through the system under standard operating conditions as recommended by the manufacturer. The concentration of the dye in the permeate and in the feed is measured with a spectrophotometer for dyes that adsorb light maximally at a specific wavelength or with a fluorometer for fluorescing dyes that adsorb at one wavelength and emit at a second wavelength. A leak, or loss of integrity, will be indicated by increased dye passage, as measured by a critical percent increase in the permeate concentration. The membrane