



Designation: D6908 – 06

Standard Practice for Integrity Testing of Water Filtration Membrane Systems¹

This standard is issued under the fixed designation D6908; 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 This practice 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 μm . The TOC, and Dye, tests are generally applicable to NF and RO class membranes only.

1.2 This practice 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:*²

D1129 Terminology Relating to Water

D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water

D3370 Practices for Sampling Water from Closed Conduits

D3864 Guide for Continual On-Line Monitoring Systems for Water Analysis

D3923 Practices for Detecting Leaks in Reverse Osmosis and Nanofiltration Devices

D4839 Test Method for Total Carbon and Organic Carbon in Water by Ultraviolet, or Persulfate Oxidation, or Both, and Infrared Detection

D5173 Test Method for 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

D5904 Test Method for Total Carbon, Inorganic Carbon, and Organic Carbon in Water by Ultraviolet, Persulfate Oxidation, and Membrane Conductivity Detection

D5997 Test Method for On-Line Monitoring of Total Carbon, Inorganic Carbon in Water by Ultraviolet, Persulfate Oxidation, and Membrane Conductivity Detection

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

D6698 Test Method for On-Line Measurement of Turbidity Below 5 NTU in Water

E20 Practice for Particle Size Analysis of Particulate Substances in the Range of 0.2 to 75 μm by Optical Microscopy³

E128 Test Method for Maximum Pore Diameter and Permeability of Rigid Porous Filters for Laboratory Use

F658 Practice for Calibration of a Liquid-Borne Particle Counter Using an Optical System Based Upon Light Extinction³

3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, refer to Terminologies D6161 and D1129.

3.1.1 For description of terms relating to cross flow membrane systems, refer to Terminology D6161.

3.1.2 For definition of terms relating to dissolved carbon and carbon analyzers, refer to D5173, D5904 and D5997.

¹ 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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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.

³ Withdrawn.

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 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 μm .

3.1.8 *multiplexing*—the sharing of a common set of physical, optical, and/or electrical components across multiple system sample points. Two approaches of multiplexing are considered in this practice: sensor multiplexing and liquid multiplexing. Sensor multiplexing monitors a unique sample with a dedicated sensor. Sensors are linked to a centralized location, where data processing and the determinative measurement is performed. Liquid multiplexing uses a common instrument to measure multiple process sample streams in a sequential manor. Samples are fed to the common analyzer via a system of a manifold, valves and tubing.

3.1.9 *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.

3.1.10 *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.

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 μm 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. **D5173** lists requirements for a variety of instruments.

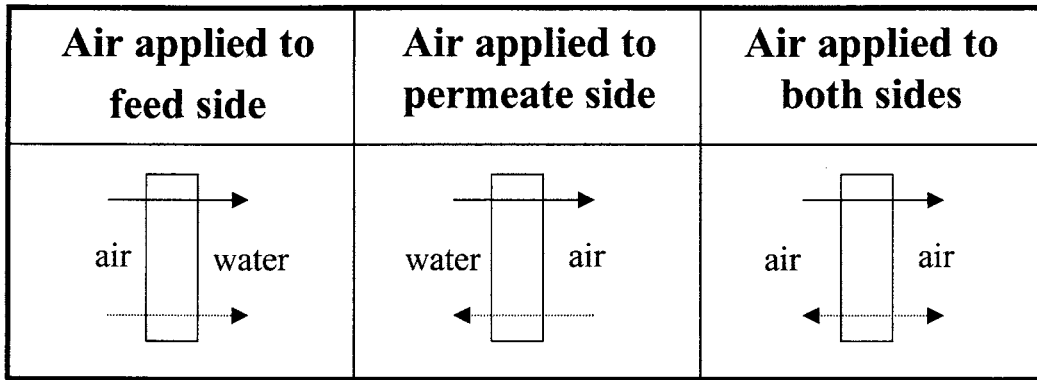
5.2 *Soluble Dye Solution*—Use FD&C or reagent grade dyes such as FD&C Red #40, dissolved in RO permeate, or in ASTM Reagent Grade Type IV water.

5.3 *Light Scatter Standards*—See Test Method **D6698** 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 **D2777**. 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 **D2777**.



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 D3923.

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

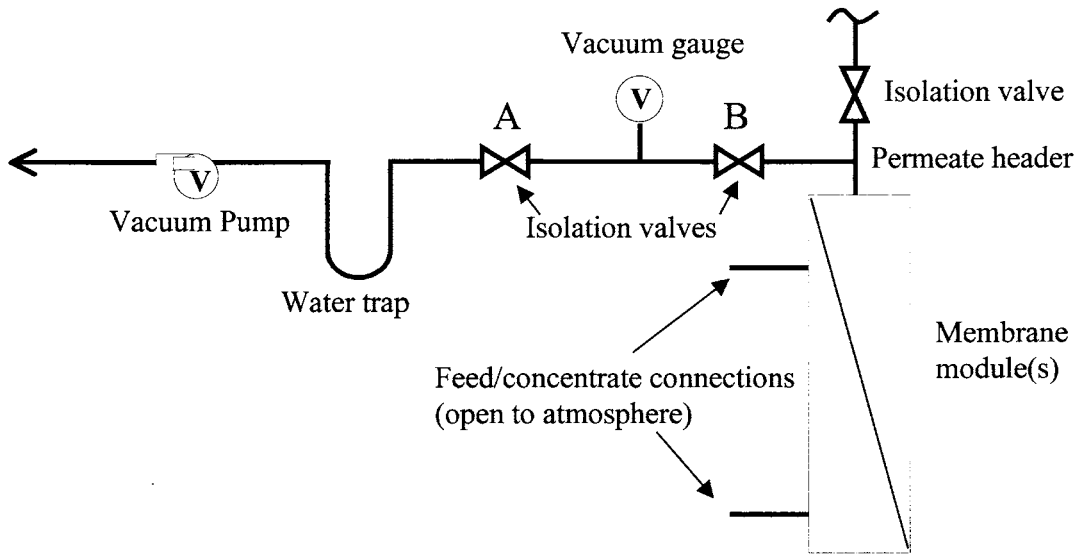


FIG. 2 Connection Arrangement for the VDT

9.1.4 After allowing time for the decay rate to stabilize record the initial pressure, P_i , and commence timer.⁴

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.⁵

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 component can be estimated either by calculation or experimental determination of the diffusive flow, such as laboratory mea-

surements 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.

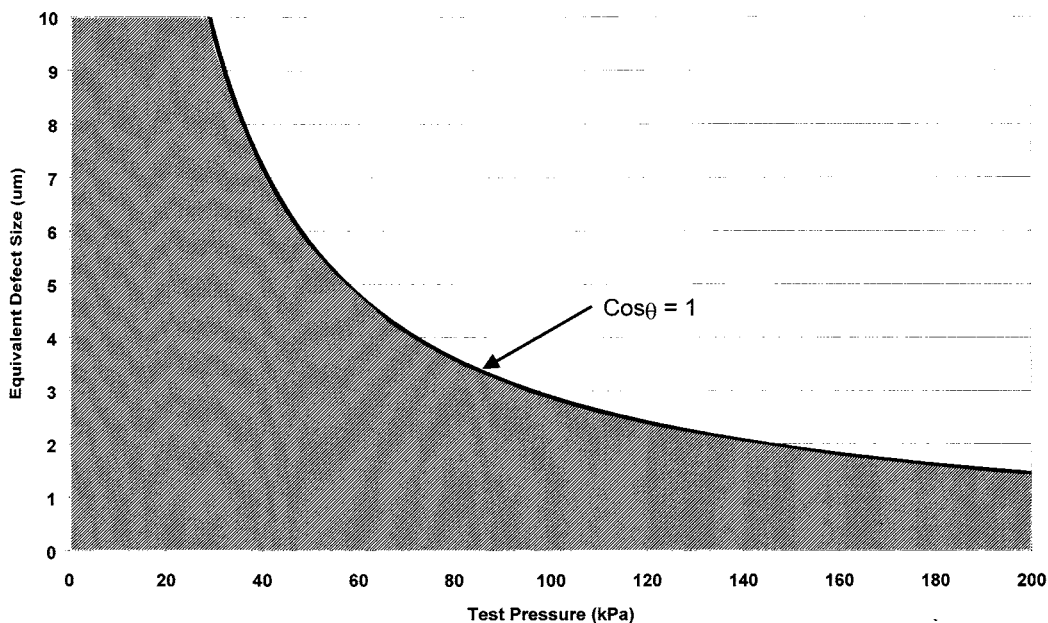
9.2.2 Use the equipment connected in this order (see Fig. 2): a vacuum pressure gauge, an isolation valve, a water trap that will not buckle at vacuum, and a vacuum pump, to the permeate manifold that serves one or more membrane devices. Addition of another isolation valve (B) at the permeate header 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.

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.⁵

⁴ 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.

⁵ 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 .



NOTE—The solid line represents Eq 2.

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

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 E128.⁶

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

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,
- γ = surface tension at the air-liquid interface,
- θ = 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:

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

⁶ 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.

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 contaminants 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 μm . 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 μm 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 μm) 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 μm 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 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 = μ_{water} / μ_{air} ,
- f_2 = pressure correction factor = $P_{u,test}^2 - P_{d,test}^2 / 2P_{atm} TMP$,
- $\frac{Q_{filt}}{P_{u,test}}$ = filtrate flowrate (m^3/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),
- $\frac{CF}{CF}$ = 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,
- $\frac{PDR}{PDR}$ = pressure decay rate (kPa/s),
- $\frac{VDR}{VDR}$ = vacuum decay rate (kPa/s),
- $\frac{TMP}{TMP}$ = transmembrane pressure during filtration (kPa),
- $\frac{V_{system}}{V_{system}}$ = volume pressurised (or under vacuum) during test (m^3),
- μ_{water} = the viscosity of the liquid during filtration (Pa·s),
- μ_{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 1.00×10^{-3} Pa·s and air at 27°C is 1.84×10^{-5} Pa·s. The pressurized system volume during the PDT is 400 L.

First calculate f_1 and f_2 :

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

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

Estimate the LRV from Eq 3 as follows:

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