CHAPTER 11

Testing Filter Media

The bulk of this Handbook has been concerned with the various types of filter media, and has described their properties, as concern filtration. The final two chapters show how these properties are measured, and describe the standards that govern the various detailed characteristics.

11.1 Introduction

A large and ever increasing number of standard tests are available for characterizing either filters or their associated media. These standards are established either by national authorities, such as BSI (the British Standards Institution) and ASTM (the American Society for Testing Materials), by specific industry organizations, such as TAPPI (the American Pulp and Paper Industry) or by regional or international organizations, such as CEN (Comité Européen de Normalisation) and ISO (the International Standards Organization). These standards typically define in detail the recommended testing equipment, its method of operation and the associated procedures for processing and interpreting data. Some of them are of broad relevance, but many are focused on specific types of media, for example sintered metals, or applications, for example lubricating oil for internal combustion engines.

The objective of this chapter is to present an overview of the relatively few principles that underlie the resultant multiplicity of standard test procedures, so that the reader will be better able to assess and interpret much of the data provided in earlier chapters. There is no attempt here to provide detailed guidance on the execution of any of the tests; readers requiring this information are recommended to refer to the appropriate published standards relevant to their geographical location and industrial context.

Apart from mechanical strength, the properties of filter media of particular interest are five of the six 'filtration-specific properties' identified in Table 1.6 of Chapter 1, namely:
1. the smallest particle that the medium is able to retain;
2. the efficiency with which particles of a defined size are retained;
3. the resistance of the medium to the flow of clean fluid through it;

4.   the dirt-holding capacity of the medium; and
5.   the tendency of the medium to blind, especially when used repeatedly in an operating cycle that includes cleaning, especially where particles adhere tenaciously to the medium.

   However, a lot can be learned about a filter medium by examination of its structure. Any newly developed material will normally have undergone microscopic examination to enable its relation to other media to be seen, and to permit some initial estimates as to its likely performance. A novel technique is now available[1] that permits the production of three-dimensional images of materials that are soft enough to slice (i.e. mainly natural fibres and polymers). The technique involves the slicing of a block of material very thinly (to an accuracy of 0.1 μm), the illumination of the new surface with ultraviolet light in a special microscope, and the capturing of the image on a large format digital camera. A thousand images may be taken, and these are then assembled to give a 3-D picture of the material.

11.2 Testing Filtration Characteristics

Most of the test procedures designed to characterize a medium in respect of the filtration-specific properties involve 'challenging' the medium, either with a suitable clean fluid, or with a fluid containing dispersed particles of selected and controlled characteristics.
   Challenging with a clean fluid permits evaluation of:

- the permeability or resistance to flow per unit area of medium, such as the flow rate of air or water under a defined pressure; and
- the size of the pores of the medium, in terms of the ideal cylindrical pores assumed in the bubble point test, and hence an approximation of the size of particle that the medium can retain by straining mechanisms.

Challenging with a fluid containing dispersed particles permits the determination of:

- the smallest particle that can be retained with 100%, which is the 'absolute rating' of a medium;
- the relationship between particle size and retention efficiency, typically expressed as a grade efficiency curve;
- the relationship between the quantity of material filtered and the increasing resistance to flow, and hence the dirt-holding capacity, of the medium under the specific operating conditions; and
- a first indication if the medium tends to blind rapidly when used repeatedly with an operating cycle that includes cleaning.

   The notes that follow describe the main testing procedures applied to continuous media (i.e. those formed originally in sheets or rolls, or as cartridge constructions).

It should be noted, however, that these tests of filtration performance may not be the only ones of interest for a filter medium – it may be necessary to follow the performance of the same piece of material over a long lifetime, for which simple weighing after successive cleaning cycles may be sufficient[2].

11.2.1 Permeability

The immense variety of expressions formerly used for the permeability of filter media is illustrated by Table 1.10, in Chapter 1. This table was originally assembled in 1966[3], since when there has fortunately been considerable progress in standardization, so that now permeabilities are generally expressed in two main forms, even if in a considerable variety of units. The more common form, appropriate for sheets of media but effectively treating thickness as a constant, characterizes them in terms of the rate of flow of a specified fluid, usually air, per unit area. A far less widely used form, which is more rigorous fundamentally and takes cognisance of the thickness, characterizes a medium by its permeability coefficient.

11.2.1.1 Permeability coefficient

The permeability coefficient of a medium, K_p, is defined by the Darcy equation describing flow through a porous layer:

$$P/L = Q\mu/AK_p \tag{11.1}$$

where P = the differential pressure across the medium (Pa); L = the depth or thickness of the bed or medium (m); Q = the volumetric flow rate of fluid (m^3/s); μ = the kinematic viscosity of the fluid (Ns/m^2); A = area occupied by flow (m^2). When all of these parameters are expressed in SI units, as indicated, then K_p has the units of m^2.

However, K_p is frequently reported in inconsistent units, notably darcies, where the viscosity is defined in centipoise, the differential pressure in atmospheres and the other parameters in centimetres and seconds so that:

1 darcy = (1 cm^3/cm^2/s).(1 centipoise)/(1 atmosphere/cm)

Hydrologists and soil scientists prefer the term hydraulic conductivity, expressed as the velocity of water percolating per unit hydraulic gradient. Factors for conversion of K_p from m^2 to some other units are given in Table 11.1.

Table 11.1   Permeability coefficient units

1 darcy	≡	0.99×10^{-12} m^2
1 m^2	≡	1.013×10^{12} darcy
	≡	9.8×10^8 cm s^{-1} (for water at 20°C)
	≡	2.78×10^{12} ft day^{-1}
	≡	2.08×10^{13} US gallon day^{-1} ft^{-2}

Equation (11.1) assumes that the flow regime within the porous layer is laminar, which appears to be correct for the greater majority of filtration applications, where the flow rate per unit area corresponds to a maximum superficial velocity of 0.4 cm/s. However, situations can occur where other flow conditions exist, as demonstrated by Heertjes in respect of woven fabrics and by Morgan for sintered metals. Both made use of the accepted criterion of flow regime, which is the Reynolds number, adapting this to the structural forms of their own studies.

Heertjes[4] used a definition of Reynolds number, Re, based on the pore diameter:

$$Re = \rho u_p d_p / \mu \qquad (11.2)$$

where ρ = density of the fluid; u_p = fluid velocity through the pore; d_p = pore diameter; μ = fluid viscosity. He reported a transition zone in the range 3<Re<7, separating the laminar and turbulent regions. Once the flow is fully turbulent, the proportionality between flow rate and pressure is replaced by $P^{0.55}$.

Instead of the pore diameter, Morgan[5] utilized a dimensional factor M for packed beds of spherical particles, based on the porosity, e, and the specific surface area, S_v, such that:

$$M = (\text{pore volume})/(\text{surface area}) = e/S_v(1-e) \qquad (11.3)$$

His data indicate a fairly sharp transition from laminar to turbulent flow, as shown for five different grades of sintered metals in Figure 11.1. Morgan reports that practical Re values occasionally range as high as 70, when a 50% error can occur if the differential pressure is calculated assuming laminar flow. To avoid this error, he proposed a modified form of equation (11.1):

$$P/L = Q\mu/AK_p + Q^3\rho/A^2K_2 \qquad (11.4)$$

where K_2 = an inertia permeability, with units of length. In many instances, the second term is so small that it may be ignored; this applies especially to liquids where the viscosity is high.

11.2.1.2 Air permeability

The most common form for expressing permeability disregards the thickness of the medium, so that the permeability is empirically quantified by the flow rate of air per unit area, under a defined differential pressure.

An appropriate example is the Frazier scale widely used internationally in the paper and textile industries; this is based on the flow of air and was formerly specified as cfm/sq. ft at 0.5 inch WG. Metric versions require care since they may use various combinations of definitions of air volume (litres or cubic metres), time (minutes or seconds), area (square centimetres, square decimetres or square metres), and differential pressure (mm WG or Pa). Conversion factors for the units used in expressing air permeabilities are shown in Table 11.2, while associated pressure unit conversions are given in Table 11.3.

Considerably higher pressures (than the 12.5, 20 or 25 mm WG used in air tests), ranging up to 1 bar, may be used where flow rates are relatively low, due either to testing with water instead of air or to the fineness of pores in media such as membranes.

11.2.1.3 Measuring permeability

Suitable measurements can be made with apparatus of varying degrees of sophistication, as illustrated by the following examples.

A very simple measuring device is the Gurley Densometer or Air Resistance Tester, shown in Figure 11.2 and used in the paper industry. With this instrument, pressure is provided by a vertical piston that slides down under its own weight, thus forcing a known volume of air through a standard orifice holding the sample being tested. The number of seconds taken for the predetermined volume of air to flow through the sample provides an empirical definition of its permeability.
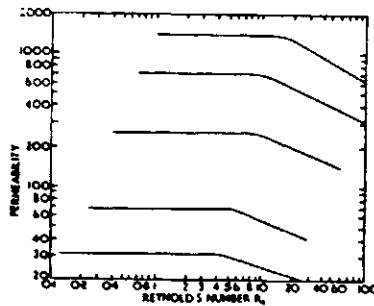

Figure 11.1. Change in flow regime at higher velocities through sintered metals, demonstrated by plot of Reynolds number versus permeability coefficient K_p.

Table 11.2   Conversion factors for various air permeability units

		l/m²/s	l/dm²/min	cm³/cm²/s	cfm	m³/m²/min	m³/m²/h
1 l/m²/s	=	1.00	0.600	0.100	0.197	0.0600	3.60
1 l/dm²/min	=	1.67	1.00	0.167	0.328	0.100	6.00
1 cm³ cm²/s	=	10.0	6.00	1.00	1.97	0.600	36.0
1 cfm/ft²	=	5.08	3.05	0.508	1.00	0.305	18.3
1 m³/m²/min	=	16.7	10.0	1.67	3.28	1.00	60.00
1 m³/m²/h	=	0.278	0.167	0.0278	0.0547	0.0167	1.00

Table 11.3   Conversion factors for air permeability

		Pa	mbar	cm WG	in WG
1 Pa	=	1.00	0.0100	0.0102	0.00402
1 mbar	=	100	1.00	1.02	0.402
1 cm WG.	=	98.1	0.981	1.00	0.394
1 in WG	=	249	2.49	2.54	1.00

The Frazier Precision Instrument Company manufactures a Differential Pressure Air Permeability Machine in two models. The first model, the Low Pressure Machine, with air flow generated by suction up to pressure differentials of 5 kPa (0.05 bar), was developed by the US National Institute of Standards and Technology, for measuring the air permeabilities of textile-type materials; it is the acknowledged standard of the US Government and the US textile industry and, in practice, is widely used for any materials that can be fitted to the physical dimensions of the equipment. In conjunction with the US Air Force, Frazier developed a second model, the High Pressure Machine; this utilizes the same principle but with pressurized air flow, providing much greater versatility of use due to its higher attainable air flow with differential pressures up to 0.7 bar.

An example of a Low Pressure Machine is shown in Figure 11.3, its operating principles being illustrated schematically in Figure 11.4 as a version that incorporates the 'guarded cylinder principle'. This latter isolates the test area of a sample from any leakage that may occur around the peripheral clamp seal with locally rigid media, such as woven wire; for flexible media, such as paper and textiles, simple clamps are adequate, while special forms of clamp are used under other circumstances as indicated in Figure 11.5.

The same principles are also the basis of various other devices, such as the SDL Electronic Air Permeability Tester in Figure 11.6, which was developed by the Shirley Institute for all kinds of flat materials. It uses a suction pump to draw air through a circular hole in an interchangeable test head. The test head used is selected to suit the desired standard test procedure, in accordance with options such as those listed in Table 11.4. The specimen, which may be a sheet up to 120 cm × 60 cm, is clamped with a piece of it over the opening in the test head, using a quick release handle. This automatically starts the suction pump to establish and maintain airflow at the pre-selected test pressure between 98 and 2500 Pa

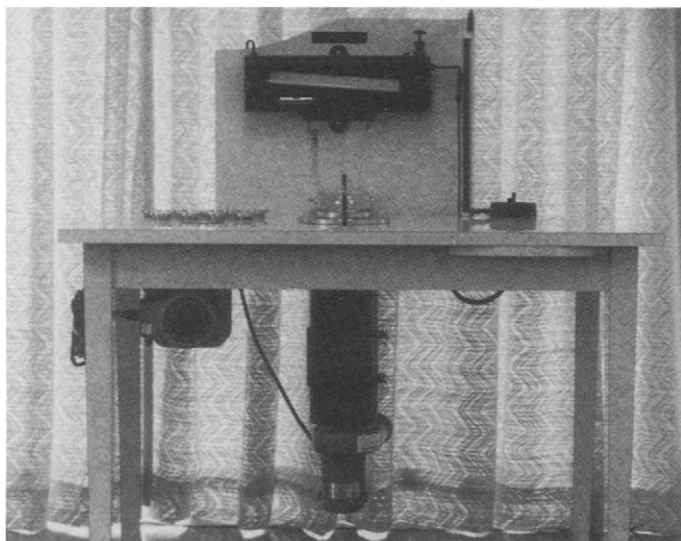*Figure 11.2. Gurley Densometer for measuring air permeability.*

Figure 11.3. Frazier Low Pressure Machine *for measuring air permeability.*
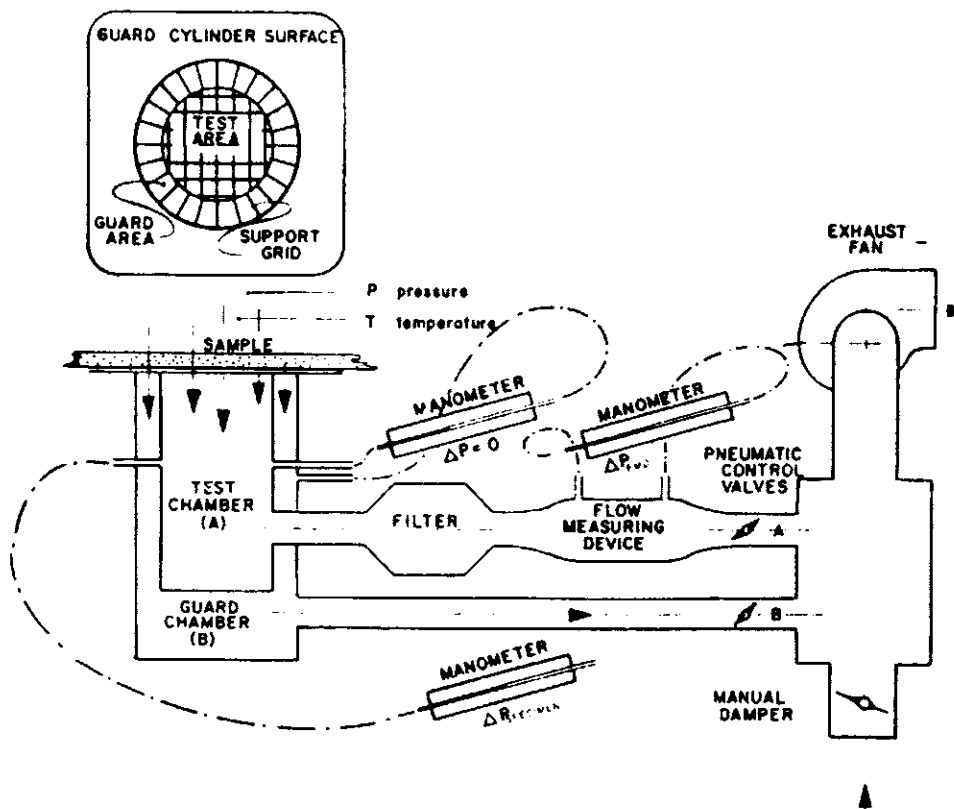

Figure 11.4. Schematic of the Frazier Differential Pressure Air Permeability Measuring Machine.

(0.001–0.025 bar). After a few seconds, the air permeability is digitally displayed in the pre-selected units of measurement, based on measurement of the airflow with a variable orifice.

Permeability measurements can be made automatically by pore analysers such as the Coulter Porometer shown in Figure 11.14.
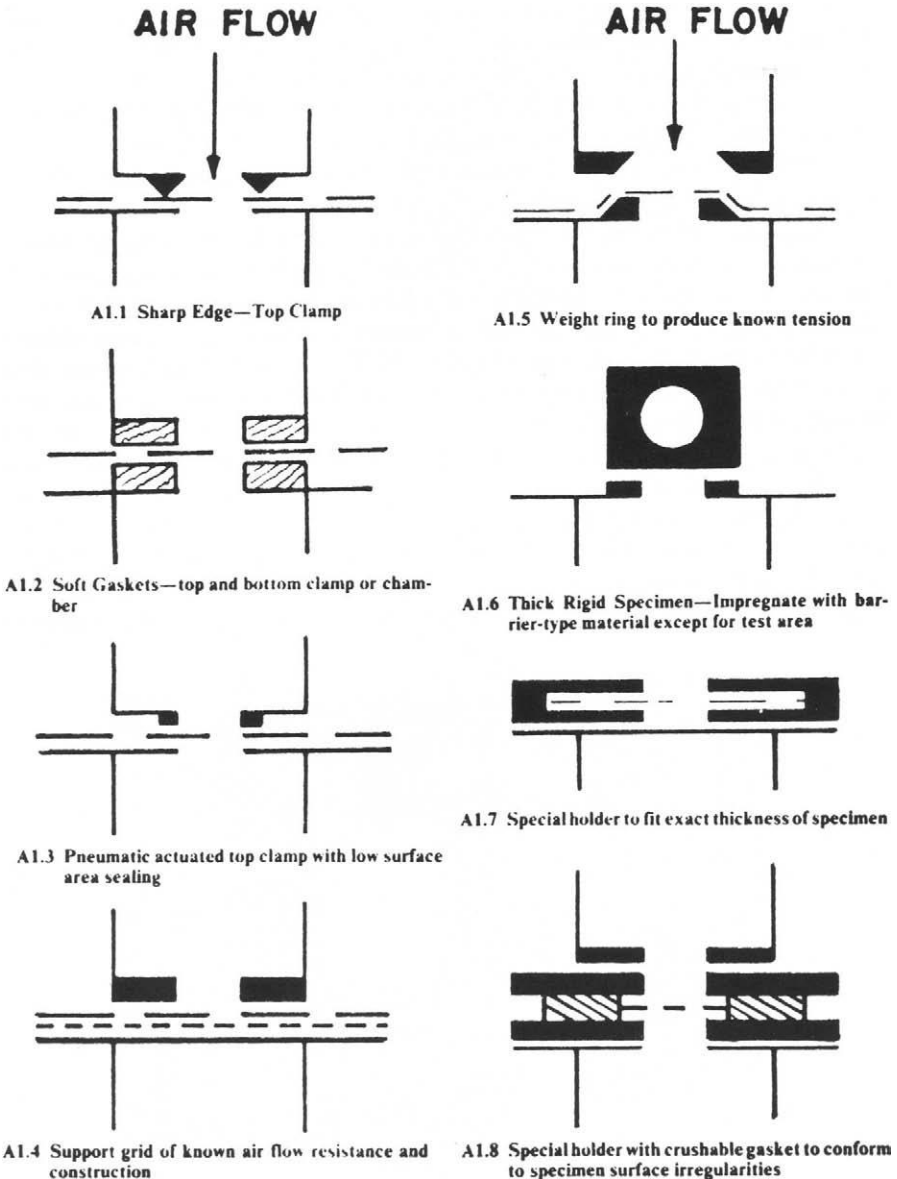
AIR FLOW

AIR FLOW

A1.1 Sharp Edge—Top Clamp

A1.5 Weight ring to produce known tension

A1.2 Soft Gaskets—top and bottom clamp or chamber

A1.6 Thick Rigid Specimen—Impregnate with barrier-type material except for test area

A1.3 Pneumatic actuated top clamp with low surface area sealing

A1.7 Special holder to fit exact thickness of specimen

A1.4 Support grid of known air flow resistance and construction

A1.8 Special holder with crushable gasket to conform to specimen surface irregularities

Figure 11.5. Frazier's suggested clamping arrangements for various types of flat media.

11.2.2 Pore size

Figure 11.7 shows schematically[6] the various types of pore that may occur in a porous material; those that are relevant to filtration are identified as 'through pores'. The four most commonly used methods for measuring the pore size of porous materials are briefly summarized below; the first two are of particular relevance to filter media and are therefore discussed at greater length in this section.

Bubble point testing, also known as *liquid expulsion testing*, utilizes a controlled air pressure to empty through pores that had previously been filled with a wetting liquid. A simple relationship between the pressure, the properties of the liquid and the diameter of an ideal circular pore permits calculation of the equivalent pore diameter. This method is normally used for pores in the size range 0.05–50 µm, but is, of course, only a secondary test, since it does not actually measure a pore dimension.

Challenge tests determine the effective size of open pores by challenging them with suspensions of particles of known sizes. This method is typically used for pores in the size range 0.005–100 µm, and this is now a direct measure of through pore size.

Mercury porosimetry, known also as mercury intrusion, involves filling the pores with mercury under pressures up to 400 MPa. The volume of mercury forced in, which can be measured very accurately, is related to pore size and pressure by the same relationship used in the bubble point test. This method, which is the subject of BS 7591:Part 1:1992, is reported to be suitable for many materials with pores in the size range 0.003–400 µm, and especially in the range 0.1–100 µm.

Gas adsorption, as described in BS 7591:Part 2:1992, typically involves measuring the quantity of nitrogen adsorbed as its relative pressure is progressively increased at a constant cryogenic temperature. The minimum size of pore that can be studied is restricted by the 0.4 nm size of the nitrogen molecule;
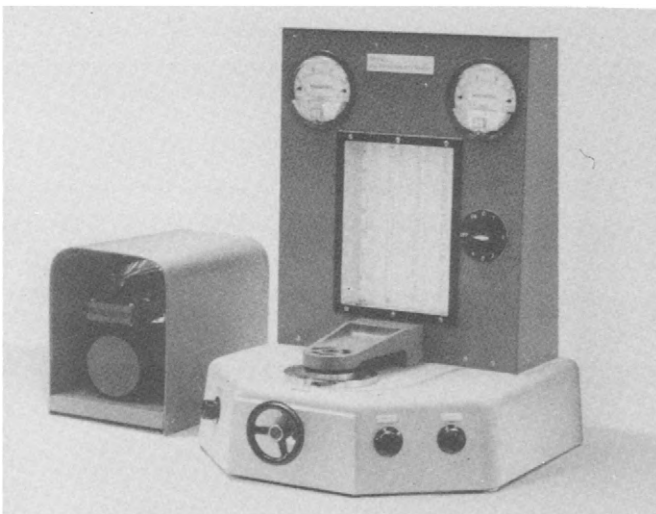

Figure 11.6. Shirley air permeability tester.

the maximum is limited to about 50 nm by the practical difficulty of measuring the amount of nitrogen adsorbed at high relative pressure. The method is therefore most appropriate for pores in the size range 0.0004–0.04 μm.

11.2.2.1 Equivalent pore size by a bubble point test

A very simple form of bubble point test apparatus is shown in Figure 11.8. This includes an enlargement of the holder in which a disc sample of the medium under test is inserted, so that it is submerged under a layer of liquid. Care must be taken to ensure that the sample is thoroughly wetted, with all of its pores filled with liquid; some test procedures require this to be done by vacuum impregnation, so as to eliminate any occluded pockets of air.

The test liquid needs to be chosen so that it will efficiently wet the material of the medium. Recommended liquids include white spirit for fabrics (BS 3321:1986), fully chlorinated hydrocarbon for paper, polymer membranes and cloth (BS 7591:Part 4:1993), and the liquids listed in Table 11.5 for metals.

In essence, the test procedure comprises increasing the air pressure slowly whilst observing the surface of the liquid in the reservoir. Typically, two different pressure values are noted, the first corresponding to the largest pore, which is detected by the appearance of the first stream of bubbles; as the pressure is increased, general bubbling develops, providing an indication of the average pore size.

Table 11.4   International test standards for air permeability

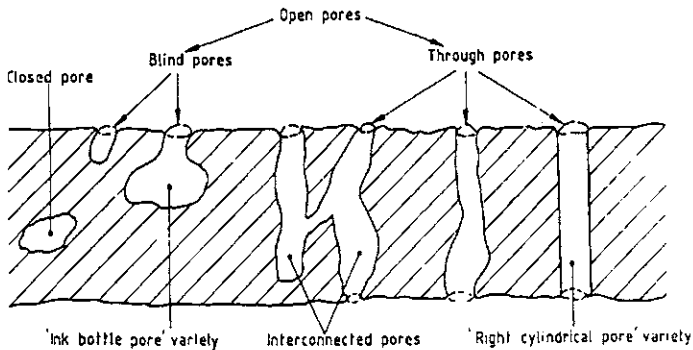| Test standard	Country	Test area (cm^2)	Test pressure (Pa)	Unit of measure
DIN 53, 887	Germany	20	200	$l/m^2/s$ or $l/dm^2/min$
AFNOR G 07-11	France	20 or 50	196	$l/m^2/s$
BS 5,636	England	5	98	$cm^3/cm^2/s$
ASTM D 737	U.S.A.	38	125	cfm
JIS L 1096-A	Japan	38	125	$cm^3/cm^2/s$
EDANA 140.1	Nonwovens	20 or 50	196	$l/m^2/s$
TAPPI T 251	Paper	20 or 38	125	cfm or $cm^3/cm^2/s$

Figure 11.7. Schematic representation of types of pores.

The equivalent pore size corresponding to each pressure reading can be calculated using the following equation:

$$d = [4\sigma(\cos\theta)/P] \times 10^6 \qquad (11.5)$$

where d = the equivalent pore diameter (mm), σ = the surface tension of the liquid (N/m), θ = the contact angle between the liquid and the pore wall (degrees), P = the pressure (Pa). For the preferred test liquids, which are fully wetting, the contact angle is zero, so that the above equation simplifies to:

$$d = (4\sigma/P) \times 10^6 \qquad (11.6)$$

A more elaborate, manually operated apparatus is shown schematically in Figure 11.9. This, together with several extracts, are reproduced with permission from British Standard 7591[6], to provide a summary of a systematic procedure for determining the following parameters:

- the maximum pore diameter;
- the minimum pore diameter;
- the mean flow pore diameter; and
- the pore size distribution.

The procedure involves conducting two test runs on a sample during each of which flow rate versus pressure readings are logged at frequent intervals. First is a wet run on the wetted sample, with a continuing increase of pressure until all the pores are empty of liquid, as shown when the flow rate/pressure plot becomes
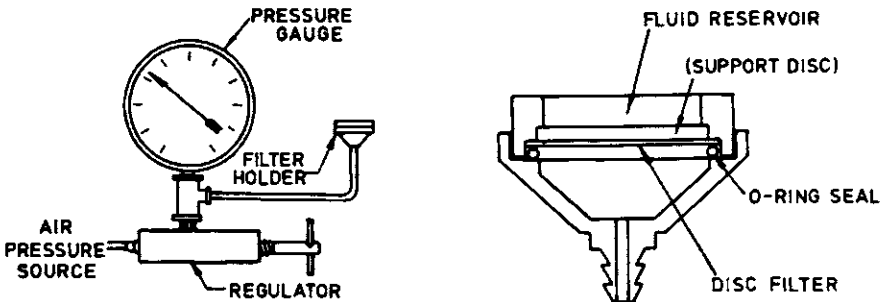

Figure 11.8. A simple bubble point test apparatus.

Table 11.5 Liquids for bubble point testing of porous metals

Test liquid	Density (g/cm³)	Surface tension @ 20°C (N/m)
Methanol	0.79	0.0225
Ethanol (95%)	0.805	0.023
Isopropanol	0.79	0.0215
Carbon tetrachloride	1.59	0.027

linear, as shown in Figure 11.10. The air pressure is then reduced to zero and the dry run commenced immediately through the now dried sample still in the holder; readings are recorded until the dry run plot overlays the wet run plot, and are then continued until the maximum permissible pressure is reached.

The point at which the wet run curve leaves the baseline approximates to the bubble point pressure, from which the maximum pore diameter may be calculated. Similarly, the point where the wet run and dry run points converge corresponds to the minimum pore diameter. Superimposing an extra 'half of dry run' plot on the data plot of Figure 11.10, by simply halving each dry flow value, identifies the mean flow pore diameter at the intersection of this extra plot and the wet run curve.

The pore size distribution is calculated from the wet and dry run plots in Figure 11.10, but for clarity the relevant part is shown separately in Figure 11.11. Repetitive calculations are made for a sequence of small pressure intervals, as indicated by the example in Figure 11.11, between a low pressure, l, and a higher pressure, h.

Assuming that in this example $l = 0.06$ MPa and $h = 0.065$ MPa, and that the surface tension of the test liquid = 0.016 N/m, then the corresponding pore sizes are 1.07 and 0.98 mm. Hence, the percentage, B, of pores between these diameters is given by:

$$B = [(\text{wet flow } h/\text{dry flow } h) - (\text{wet flow } l/\text{dry flow } l)] \times 100 \quad (11.7)$$
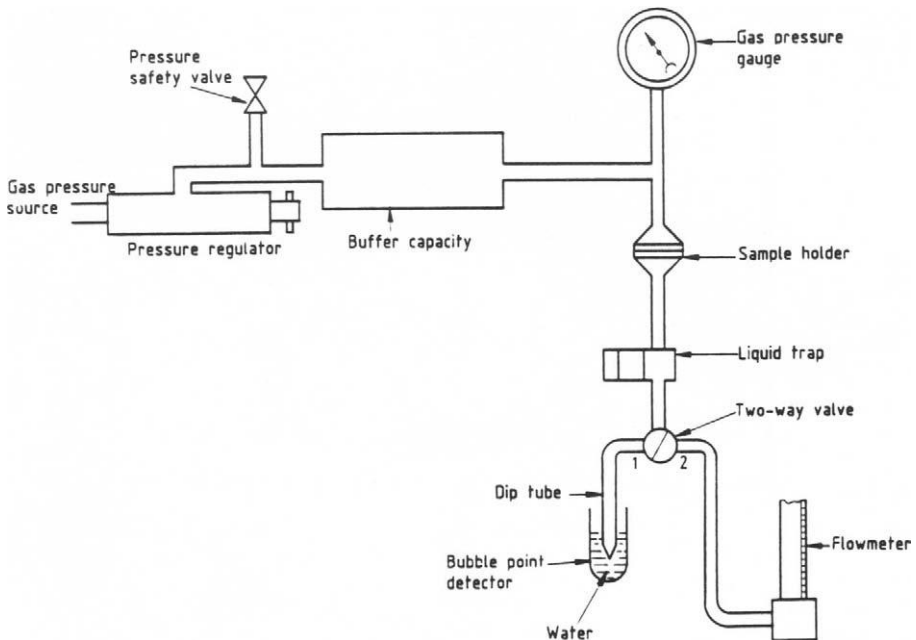$$= (0.4/9 - 0.2/8) \times 100 = 2\%$$

Figure 11.9. The basic form of a typical manually controlled bubble point apparatus.

The pore size distribution by flow is computed by accumulating values of B from the maximum to the minimum pore size. The resultant data may be presented in the form of either Figure 11.12 or Figure 11.13.

The test procedures outlined above, and the computational analysis of the measured data, can be conducted automatically by equipment such as the Coulter Porometer illustrated in Figure 11.14(a) and (b). This is a microprocessor-controlled, menu-driven instrument operating at pressures up to 13 bar, suited to pores from macro-size down to 0.05 mm; the analysis time is typically under 10 min. The medium under test is in the form of a disc that is
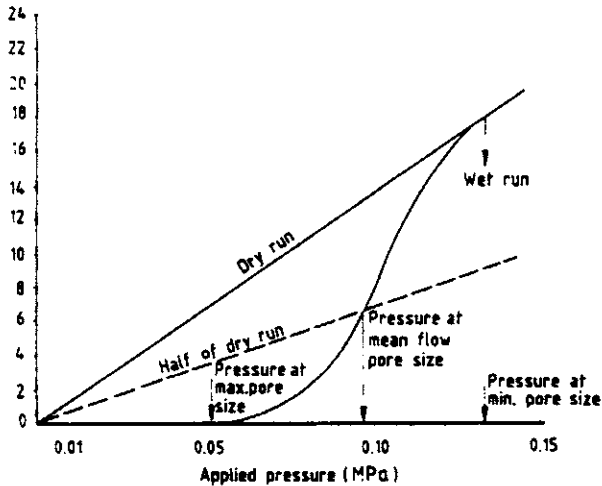

Figure 11.10. Typical plot of flow rate versus applied pressure for wet and dry runs, performed on a single test sample.
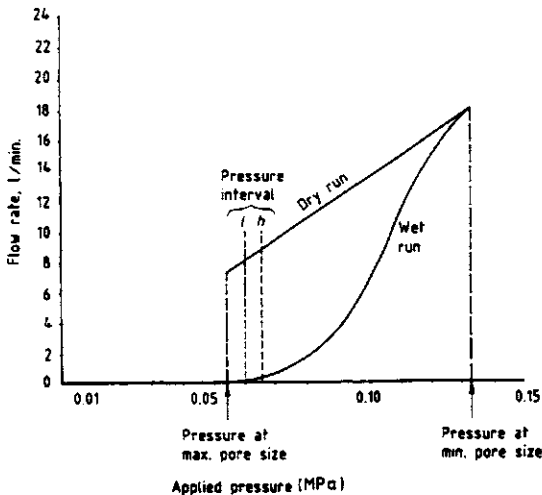

Figure 11.11. Adapting Figure 11.10 for repetitive calculations of small pressure intervals.

mounted in a suitable sample holder after being thoroughly wetted. The resultant data may either be displayed or printed out.

The instrument can also be used to measure permeability of a sample of filter medium. A further use is to determine the integrity of a filter cartridge (i.e. the absence of any significant leaks in it), by the Pressure Hold analysis option. This involves isolating a pressurized filter and monitoring the pressure over a timed period.

11.2.2.2 Effective pore size by challenge tests

The process of a challenge test involves presenting a filter with a fluid containing a known concentration of a defined particulate, and then analysing the filtrate downstream of the filter to determine how much of the particulate material has passed through, and of what sizes. The particulate in the presented fluid may be monosized, or it may comprise either particle sizes over a specified range, or a distribution of sizes over given limits of size.

With test materials of mixed particle size, the largest particle passing is an indication of the size of the largest pore in the filter medium. The medium may be described as 'absolute' to all particles greater than some slightly larger size; with monosized particles, the percentage of particles passing characterizes the filtration efficiency of the medium.

It is clear from this brief introduction that challenge tests rely heavily upon the ability of the tester to measure the particles, for both number (or quantity) and
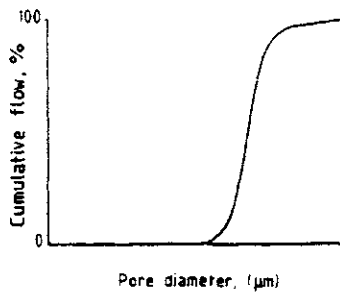

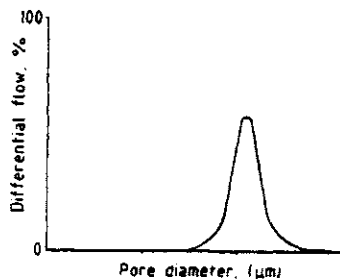*Figure 11.12. Cumulative flow pore size distribution.*

Figure 11.13. Differential flow pore size distribution.

size (or size distribution) both ahead and after the medium being tested. This size and quantity determination accounts for most of the variation among the techniques employing this method.

A well-known example of the challenge process is the glass bead test. In this, a sheet or disc of filter medium is fitted within a filter holder and a suspension of glass beads is sucked through the filter. The suspension comprises beads of specific diameters over a range covering the expected pore size of the filter. Beads that have passed through the filter are trapped on an analysis membrane and examined by a microscope to determined the largest size; in doing this, great care must be taken to ensure that no stray beads inadvertently appear on the membrane, because even just one large bead can totally alter the assessment of
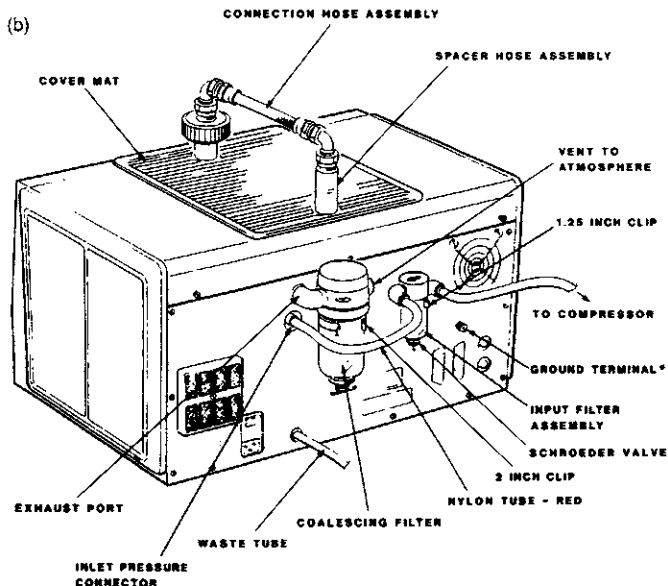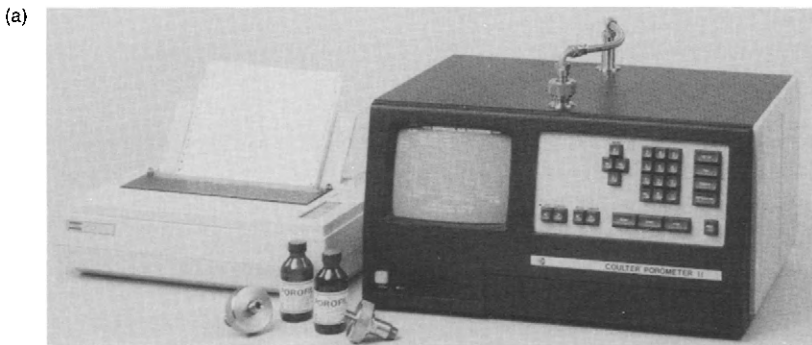

Figure 11.14. (a) A Porometer II *automated pore analyzer – front view with sample holder on top;* (b) a Porometer II *automated pore analyzer – schematic of rear view.*

the filter pore size. A glass bead test is included in the US standard for testing filters for hydraulic power systems in military vehicles; examples of the grades of glass beads and other special spherical particles available commercially are given in Table 11.6.

One of the problems with the glass bead test, or with any synthetic dust-like material, is the creation of a size distribution to satisfy the needs of the test. A range of glass microspheres is now available[7], with a narrow size distribution. The various size fractions are produced by means of sonic energy sieving, which enables the solid to be processed down to 15 μm in the dry state (below 15 μm such separations have to be done in liquid suspension, to overcome the electrostatic properties of the particles). One of the particular applications of these closely sized microspheres is in the sizing of screens for use in keeping sand out of undersea oil (which otherwise would cause costly abrasion).

The pharmaceutical industry utilizes a critically important (as regards health) challenge test based on a suspension of the bacterium *Pseudomonas diminuta ATCC* 19146, in which each square centimetre of filter medium surface is challenged with as many as 10 million bacteria. Provided no bacterium passes through, the medium is classed as 0.2 μm absolute rating, despite the fact that these bacteria are slightly larger than 0.2 μm. The detailed sterilization testing procedure, using the system shown schematically in Figure 11.15 is described by Howard and Nicholaus[8].

In reality, numerous factors combine to make questionable the principle of characterizing media by this form of challenge test, which is strictly only suitable where the pores are clearly formed, as in meshes and screens, where the challenge particles are spherical and where the filtration mechanism is simple straining. Under these circumstances it can be very accurate. It is more meaningful generally to express the performance of a medium in terms of its efficiencies against particles of a range of sizes.

A particularly severe challenge test is reported by Endo *et al.*[9] in the testing of a sintered ceramic filter medium made from spherical particles of alumina.
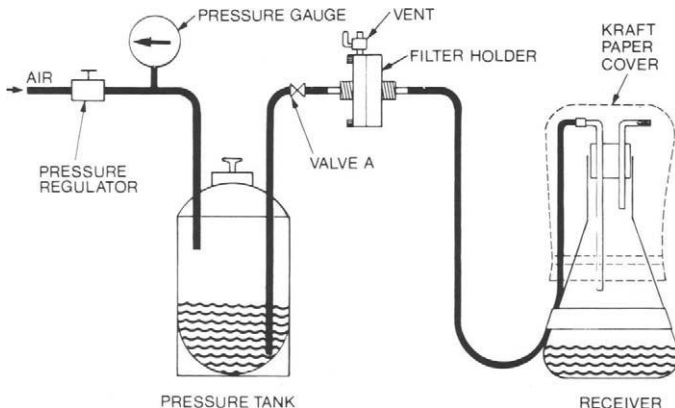

Figure 11.15. System for sterile filtration test.

Table 11.6 Examples of highly graded spherical particles[a]

Description	Size range	Grades	State	Examples Nominal size (µm)	Certified mean diameter (µm)	Size uniformity SD[b]	CV[c]
Certified standards							
Polystyrene nanospheres	20–900 nm	26	1% in water	20	19±1.5	–	–
				100	102±3	5.3	7.5%
Polymer microspheres	1.0–160 µm	26	0.2–5% in water	1.0	0.99±0.02	0.010	1.0%
	200–1000 µm	10	Dry spheres	200	202±4.0	7.5	3.7
Silica microspheres	0.5–1.6 µm	4	2.0% in water	0.5	0.46±0.03	0.02 µm	4.3%
				1.6	1.57±0.06	0.04 µm	2.5%
Borosilicate glass microspheres	2–20 µm	6	Dry spheres	2	2.5±0.5	1.0 µm	40%
				20	20.2±1.4	1.7 µm	8.4%
Soda-lime glass microspheres	1.5–2000 µm	30	Dry spheres	1.5	2.1±0.5	0.9 µm	43%
				750	756±23	22.7 µm	3.0
Research microspheres				*Mean diameter (µm)*			
Polystyrene latex	0.028–3.7 µm	42	Dry spheres	0.028	–	0.0031	11%
				0.652	–	0.0048 µm	0.7%
Polystyrene DVB[d]	3.2–220 µm	15	Dry spheres	3.2	–	1.4 µm	43%
				220	–	16.5 µm	7.5%

[a] Duke Scientific Corporation.
[b] SD=standard deviation.
[c] CV=coefficient of variance.
[d] DVB=cross-linked with 4–8% of divinylbenzene.

The membrane was formed of a 20 μm layer of 0.6 or 0.84 μm spheres supported on a 2 mm layer of 15 μm spheres, and has a high collection efficiency, but with a high differential pressure. The challenge was a polydisperse sodium chloride aerosol, with penetrations as low as 10^{-9} in the size range 0.02–0.14 μm. The particle concentration was determined with a condensation nucleus counter.

11.2.3 Filtration efficiency

The basic principles of challenge testing, utilizing either mixed size or monosized particles, are adapted according to the nature of the fluid (liquid or gas), and the relevant filtration mechanisms (surface straining or depth), associated with the structure of the medium. An overview of the relationship between these variables, the main categories of practical application and the several forms for expressing filtration efficiency, is provided by Table 11.7.

11.2.3.1 Test dusts, aerosols and filtration efficiency

Filtration efficiency is usually stated in terms of the percentage of particles of a certain size that would be stopped and retained by a filter medium. This raises two quite difficult problems:

- where a test dust comprises particles of a range of sizes, what is the actual size to which the percentage efficiency relates?
- the numerical differences in percentage efficiency of a wide variety of media are often relatively small, many media being over 95% efficient – therefore, is percentage efficiency a meaningful basis for comparison of different media?

These two dilemmas are resolved by the use of test particulates of known distribution and by a more sensitive expression for filtration efficiency. As Table 11.8 indicates, a considerable variety of standard test dusts has evolved, many tailored to specific areas of application; one dust widely used for both liquid and gas phase applications is AC Fine Test Dust (equivalent to SAE J 726 Fine), which is described in more detail in Table 5.3 of Chapter 5.

There are two alternative expressions for percentage efficiency; one is percentage penetration, the other is the Beta ratio (β ratio). Very high efficiency air filters, for which efficiencies range upwards from 99.99%, are sometimes characterized in terms of percentage penetration, and are in fact classified as ULPA (Ultra Low Penetration Air) filters; thus, Eurovent class EU 15 can be described as having an efficiency of 99.9995% or a penetration of 0.0005%.

The β ratio is based on counts of particles of specific sizes and is defined as:

$$\beta_n = N_u/N_d$$

where N_u = number of particles $> n$ μm per unit volume of liquid upstream; N_d = number of particles $< n$ μm per unit volume of liquid downstream. The

Table 11.7 Summary of challenge test and filtration efficiency categories

Fluid	Particle size	Comments	Test variables	Efficiency expression	Application examples
Liquid	1. Mixed	Most common	Single pass or multi-pass	1. Grade efficiency curves	General use
				2. Particle size for 98% efficiency	General use
				3. β factor	Hydraulic power systems; critical pharmaceuticals
	2. Monosized	Special cases	Single pass	% efficiency for one size only	Microorganism removal
Gas/air	1. Mixed	Air filter standard	Single pass	% weight retention	Air filter for coarse and fine dusts
	2. Monosized	Air filter standard	Single pass	% efficiency or % penetration for one size only	HEPA and ULPA air filters

percentage filtration efficiency, E, is related to β by the expression $E = 1 - 1/\beta$. Corresponding values for E and β are given in Table 11.9, together with examples of particle number counts. Figure 11.16 shows a typical plot of the β ratios versus particle size for a filter medium challenge test, and demonstrates the usage of this mode for characterizing a medium; thus $\beta_{17} = 200$ indicates an upstream/downstream ratio of 200 for 17 µm particles.

11.2.3.2 Filtration of liquids

Two different techniques are used for determining the efficiency when filtering liquids, respectively identified as the single-pass test and the multipass test. Although these tests have much in common, there is a significant difference in

Table 11.8   Examples of test dusts[a]

Designation	Material		Size range (µ)
BS 1701 Coarse	Quartz		0–150
BS 1701 Fine	Quartz		0–75
BS 2831 No. 2	Fused alumina		0–10
BS 2831 No. 3	Fused alumina		8–32
DEFSTAN[b] 0755	Quartz sand		100–1000
SAE[c] J 726 Fine/AC Fine	Mineral sand		0–125
SAE[c] J 726 Coarse/AC Coarse	Mineral sand		0–200
MIRA[d] Grade 1/BS4552	Fused alumina		2.5–9
MIRA[d] Grade 2/BS4552	Fused alumina		3–11
MIRA[d] Grade 3/BS4552	Fused alumina		6.0–21
MIRA[d] Grade 4/BS4552	Fused alumina		15.0–53
MIRA[d] Grade 5/BS4552	Fused alumina		27.0–90
ASHRAE[e] 52/76	Molacco black	23% (wt.)	
	SAE J 726 fine	72%	0–80
	Cotton linters	5%	
BG[f] Test dust	Black iron oxide	79%	
	Red iron oxide	12%	
	Silica flour	8%	0–400
	Paint residue	1%	
	Cotton linters	0.05%	
RR[g] lubricant contaminant	Lamp black	60%	
	Bearing steel	10%	
	Red iron oxide	5%	0–420
	AC Fine	15%	
	Metco 31 seal material	10%	

[a]   Dusts to these and other specifications are manufactured by ParticleTechnology Ltd.
[b]   DEFSTAN, Defence Standard (Ministry of Defence. UK).
[c]   SAE, Society of Automotive Engineers. Grades equivalent to Arizona Road Dust.
[d]   MIRA, Motor Industry Research Association.
[e]   ASHRAE, American Society of Heating, Refrigerating and Airconditioning Engineers.
[f]   BB, British Gas.
[g]   RR, Rolls Royce.

the particle size distribution presented to the filter, which may significantly affect the stated efficiency of the medium.

The single-pass test, as its name implies, passes a consistent, unchanging distribution of particles through the test circuit just once. A typical circuit for a single-pass test rig for pressure filtration is shown schematically in Figure 11.17. The filter medium, ranging from say a 47 mm diameter disc to a 300 × 300 mm sheet, is held in a leak-free support, with sampling points sited as close to the filter as possible. Bottle samples of fluid are sometimes taken for later examination and analysis; however, this has the disadvantages of risking contamination and of the two samples not being precisely in step, both of which can be avoided by appropriate on-line analysis.

A different version of a single-pass test rig is illustrated in Figure 11.18. This shows the standard assembly used to conduct laboratory bench tests on slurries for making preliminary assessments of the suitability of process filters such as rotary drum, disc and belt filters. The circular cloth-covered filter disc, connected

Table 11.9 Comparison of β-ratios and % efficiencies

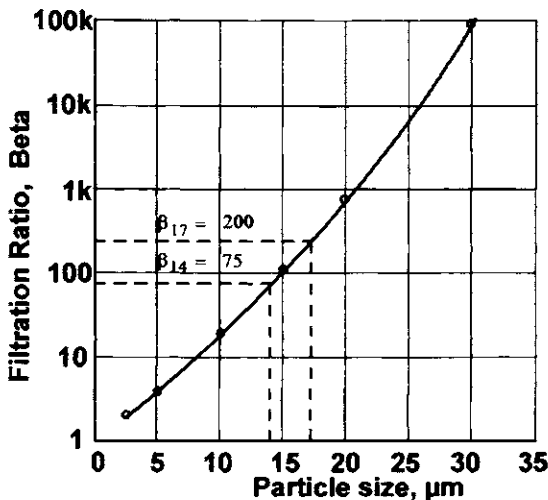| Examples of numbers of particles		% efficiency	β ratio
Upstream	Downstream		
10 000	5000	50	2
10 000	1000	90	10
10 000	500	95	20
10 000	100	99	100
10 000	10	99.9	1000
10 000	1	99.99	10 000

Figure 11.16. Plot of β ratio versus particle size.

to a flexible rubber hose, can be inverted into a container of slurry for a timed period, such as 1 minute, so as to mimic the stage-wise cycle of these continuous filters. Preliminary tests can be conducted with various types and grades of filter cloth to permit initial selection in terms of criteria such as clarity of filtrate, rate of filtration, and ease and completeness of discharge of the filter cake. Full details of the experimental procedures are provided by Dahlstrom and Silverblatt[10].

By contrast, the objective of the multipass test with the pressure filtration circuit shown in Figures 11.19 and 11.20 is to challenge the filter with a gradually increasing percentage of smaller particles: this is felt to be more representative of real systems in which a fluid is recirculated repeatedly and where larger particles are not only removed by filtration but are also being ground down to smaller dimensions. This test was originally developed for hydraulic oils but has become the basis of standards relating to other fluids such as lubricating fluids and water: for example, the internal combustion engine lube oil filter standard is ISO 4585. Therefore the multipass test, as defined by ISO
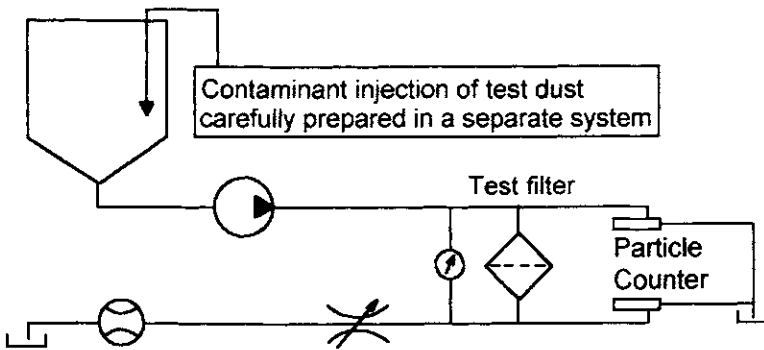

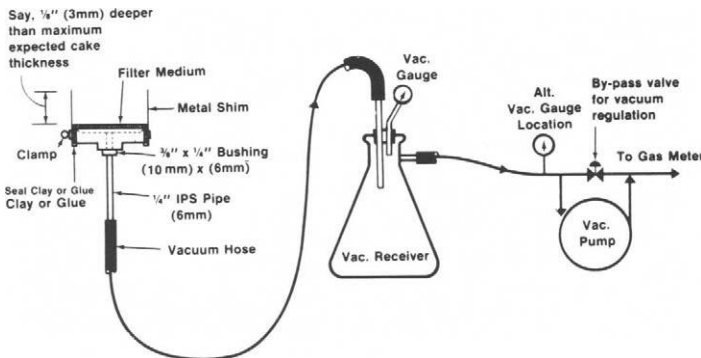*Figure 11.17. A typical single-pass test circuit for pressure filtration.*

Figure 11.18. A basic set up for conducting leaf tests on slurries in relation to continuous vacuum filters.

4572, now specifies on-line sampling and analysis as mandatory, recognizing that efficiency will tend to change as the filter progressively blocks; continuous monitoring with an analyser such as that illustrated in Figure 11.21 can provide a direct read-out of β ratio.

11.2.3.3 Filtration of gas/air

All gas-phase filtration tests are of single-pass format, but there is considerable variety both in the nature of the suspension of particles used to challenge a filter
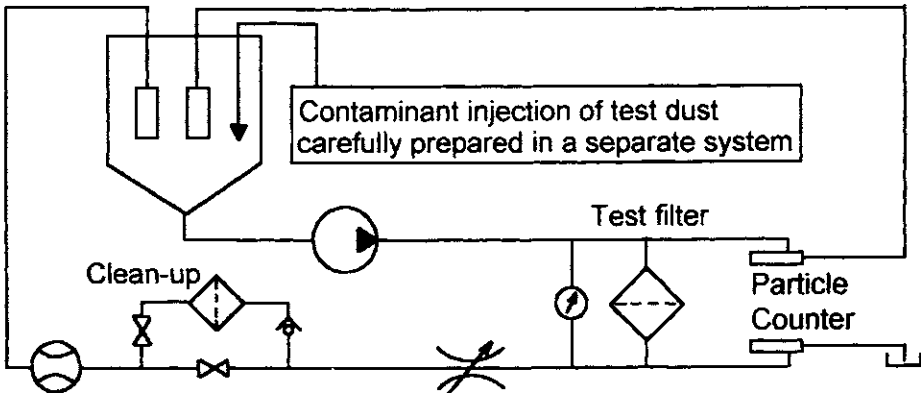

Figure 11.19. A typical multi-pass test circuit for pressure filtration.
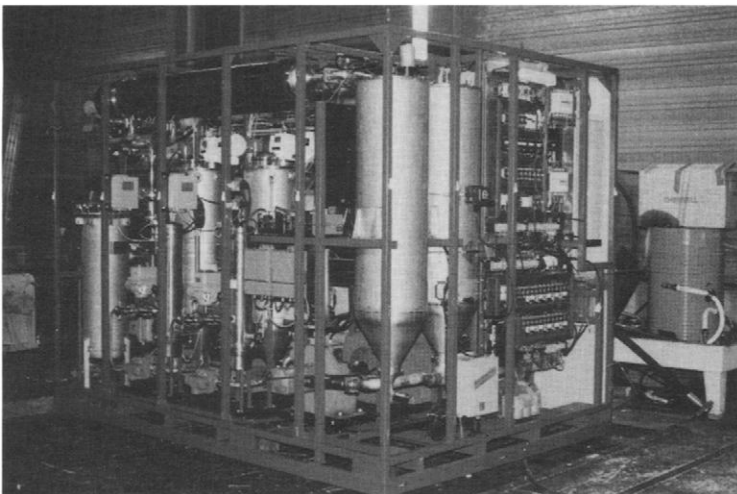

Figure 11.20. A skid-mounted multi-pass test rig handling flows up to 400 l/min at pressure differentials up to 40 bar.

and in the analytical methods whereby performance is assessed. Three types of test can be distinguished, respectively identified as:

- staining tests;
- weight arrestance; and
- particle concentration efficiency.

Atmospheric dust spot efficiency is the first of the *staining tests*. This test is a standard procedure for air filters used in air conditioning and general ventilation. It is described in detail in Part 1 of BS 6540[11], which is based on a EUROVENT standard[12]; this in turn is based on an ASHRAE standard[13] that originated in 1968.

The test is based on the intensity of staining of a 'target' filter paper caused by the flow through it of a quantity of atmospheric air. The staining arises from the natural contaminants in the local atmosphere. The intensity of the staining is monitored by an opacity metre and provides an empirical measurement of the concentration of the contaminants in the air drawn through the target.

Over a timed period, this technique is applied simultaneously to two parallel samples drawn at equal mass flow rates from the upstream and downstream sides of a filter under test; the downstream sampling, i.e. the filtrate, is continuous but the upstream sampling is intermittent and controlled by a percentage timer. Suitable adjustments of the timer results in the intensities of the stains on the upstream and downstream targets being approximately equal (i.e. they differ by no more than 20% of the higher value).

Expressed as a percentage, the atmospheric dust spot efficiency can then be calculated from the following relationship:

$$E = 100 \times (1 - Q_1 O_2 / Q_2 O_1)$$

where E = the atmospheric dust spot efficiency, %; Q_1 = the total volume of air drawn through the upstream target; Q_2 = the total volume of air drawn through the downstream target; O_1 = the opacity of the dust spot on the upstream target =
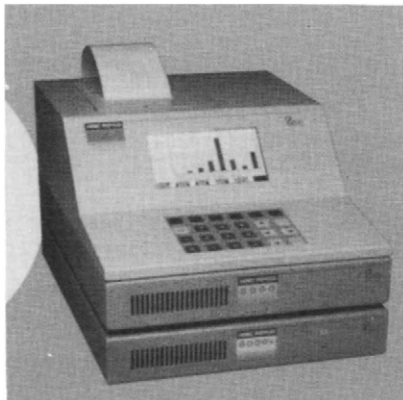

Figure 11.21. A Hyac-Royco BetaRatioMeter. (Photograph: Pacific Scientific Ltd)

$(T_{u1} - T_{u2})/T_{u1}$ (T_{u1} = the initial upstream light transmission, %; T_{u2} = the final upstream light transmission, %); O_2 = the opacity of the dust spot on the downstream target = $(T_{d1} - T_{d2})/T_{d1}$ (T_{d1} = the initial downstream light transmission, %; T_{d2} = the final downstream light transmission, %).

The *methylene blue staining test* was formerly used to characterize high-efficiency air filters in terms of the percentage penetration by submicrometre particles. With the methylene blue aerosol identified as 'Test Dust No. 1' (Nos 2 and 3 being fused alumina), it was included in both the 1957 and 1971 versions of the now obsolete BS 2831, which has been superseded by BS 6540. A short summary is provided by Dorman and Ward[14].

The aerosol is generated by atomizing a 1% aqueous solution into a constant stream of clean, dry air, which is then passed through the filter under test, the whole of the effluent being sampled by filtering again through an esparto paper for a known time. After intensification of the blue stain on the sampling esparto paper, estimation of penetration is based on comparison of this stain with a series of standard stains, either by eye or by a photoelectric densitometer.

The standard stains are previously prepared by drawing volumes of 12, 24, 36, etc., cm^3 of aerosol cloud through 125 mm^2 areas of esparto paper and intensifying the blue in steam. Identifying the nearest matching standard stain, or, if necessary, interpolating between two standard stains, defines the amount of blue dye collected on the sampling paper; for example, if the 24 cm^3 standard stain is the nearest match, then the amount of dye on the sampling paper corresponds to 24 cm^3 of unfiltered aerosol cloud. Hence, the percentage penetration is given by $100 \times 24/Q$, where Q is the total air volume filtered during the test.

Disadvantages reported for this test procedure are its increasing inaccuracy for penetrations below 0.01%, a simultaneous increase in the time required, and the need to utilize a high velocity (500 cm/s) for the esparto paper to achieve an adequate filtration efficiency.

The *synthetic dust weight arrestance* test is a standard procedure for air filters used in air conditioning and general ventilation. It is described in detail in Part 1 of BS 6540.

The essence of the procedure is to challenge a filter with a dispersion of test dust, the filtrate passing on through a second or final filter, which collects that part of the dust that penetrates through the filter under test. The dust dispersion is created continuously by a suitable combination of a dust feeder and a compressed air venturi ejector. The weight of dust passing through the filter under test is determined by re-weighing the final filter.

The full procedure, which is designed for testing complete air filters or filter panels (rather than simply a sample of filter medium), includes feeding a weighed quantity of dust in a series of equal increments, the first increment being restricted to 30 g, to permit determination of the initial synthetic dust weight arrestance. Between consecutive increments, measurement is made of the weight of dust passing the filter under test, the corresponding pressure loss across the test filter, and its atmospheric dust spot efficiency (as in the first of the staining tests described above).

The synthetic dust weight arrestance, A (%), for any particular period is given by:

$$A = 100 \times (1 - W_2/W_1)$$

where W_1 = the weight of synthetic dust fed, and W_2 = the weight of synthetic dust passing the filter under test. A typical plot of the resultant test data from this procedure is shown in Figure 11.22, reproduced from BS 6540:Part 1:1985. The data also allow calculation of the dust-holding capacity up to the maximum permissible pressure loss.

Particle concentration efficiency. For the various grades of high-efficiency air filters (HEPA, ULPA, etc.), particle concentration efficiencies are measured and expressed in terms of differences between upstream and downstream concentrations of submicrometer particles determined by continuous on-line monitoring.

Whilst the concept is simple, the practical reality tends to be complex because of the sophisticated technique and equipment required both to generate consistently suitable aerosols and to determine the size, size distribution and concentration of the particles.

Descriptions of the main techniques for the formation of aerosols are provided by Dorman and Ward[14]; they include use of pressure atomization of liquids,
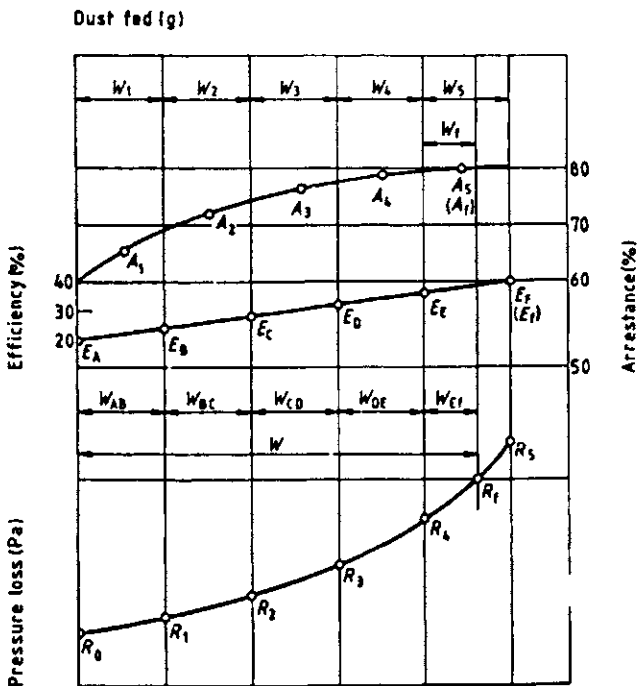

Figure 11.22. Example of plot combining data from test of atmosphere dust spot efficiency, E, and synthetic dust weight arrestance, A, based on BS 6540:Part 1:1985.

evaporation, condensation and classification. Aerosol particles are variously solid or liquid, and range from almost monosized to heterogeneous mixtures. For example, an aerosol of sodium chloride crystals can be generated by atomizing a 1% solution to produce fine droplets, from which the water is removed by evaporation; the particle size is determined by the atomization step. Alternatively, an aerosol of dioctylphthalate (DOP) droplets is formed by the condensation that occurs when warm air containing DOP vapour is quenched by dilution with cold air; the particle size is controlled by the temperature difference between the two air streams.

Successful monitoring of the concentrations of aerosol upstream and downstream of a filter requires careful integration of two separate operations. The first is the taking of representative samples, which must be done isokinetically with appropriate equipment and techniques; moreover, if the analytical device requires only a very small flow compared with that through the filter under test, then the sample must be withdrawn following a zone of thorough mixing.

The second operation is analysing the sample, the technique and parameter measured being dependent on the nature of the aerosol; with sodium chloride, the total mass concentration of all the particles is measured using flame photometry, with a photometer such as that illustrated in Figure 11.23. Liquid aerosols such as DOP are analyzed by light scattering particle counters (e.g. that in Figure 11.21), the reported sizes being related to the projected areas of the particles.

Definition of an appropriate reference particle size is complicated not just by the above parameters, but also by other factors that affect the filtration mechanism. Thus Figure 11.24 (from Wepfer[15]), which shows penetration
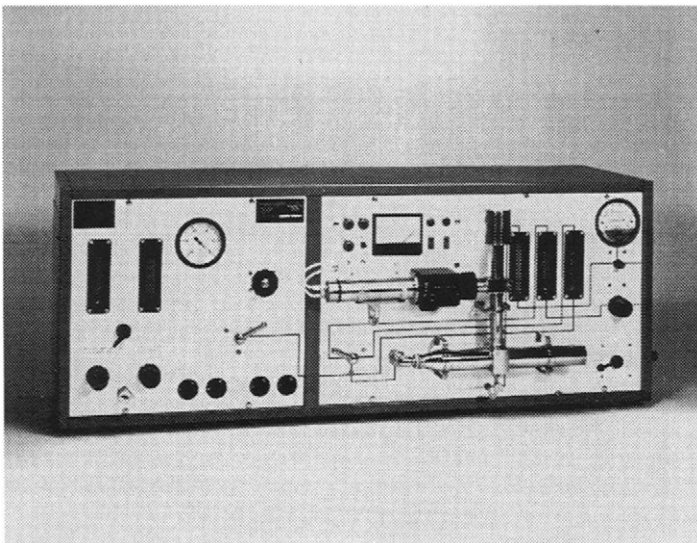

Figure 11.23. A bench rig sodium flame test meter. (Photograph: Moores Wallisdown Ltd)

maximizing in the particle size range 0.1–0.25 μm, illustrates that performance may depend on the air velocity through the medium as well as the nature of the medium; another significant variable is the nature of the aerosol. As indicated in Table 11.10, existing standards relate efficiency or penetration with their nominated aerosols to specific particles sizes (mostly 0.3 or 0.6 μm). By contrast, the new European standard[16] identifies the most penetrating particle size (MPPS) for a specific situation (especially for the most rigorous duties) and determines the penetration or efficiency for particles of this size.

Pierce (in a very good review of the history of HEPA filter testing[17]) notes that a value for MPPS of 0.3 μm has held since Langmuir first developed his theory of capture of small particles by fine fibres, even though it has long been recognized that the actual value was less than 0.3 μm. The paper presents strong support for the MPPS methods, expected to be about 0.13 or 0.15 μm for a HEPA filter.

11.2.4 Dirt-holding capacity

The dirt-holding capacity of a medium can conveniently be assessed as part of either the multipass liquid filtration test or the synthetic dust weight arrestance test for air filters, both described in the previous section.

11.2.5 Tendency to blind

Excepting under extremely unfavourable circumstances (i.e. where failure occurs very rapidly), little investigation of the tendency of a filter medium to blind is possible with small-scale, short-term tests.

11.3 Testing Mechanical Properties

Most filter media manufacturers have their own very specific mechanical property demonstrations. However, there are some generally accepted methods, which are reviewed here.

11.3.1 Strength

The strength of a material is typically characterized by generating stress/strain data using an extensometer such as the simple version shown in Figure 11.25, in which a strip of textile is stretched by a suspended weight. A linear relationship (Hooke's law) exists between applied stress and the amount of extension per unit length up to the elastic limit, beyond which stretching accelerates and then rupture occurs. This pattern provides a variety of parameters and definitions by which the material may be characterized, the most widely used being tensile strength; others are breaking, rupture or yield strength, yield point, elastic limit and ultimate elongation.

The extensometer in Figure 11.25 is not designed to test fabric as far as rupture, but only within the range of stress where both stretching and recovery

can occur, i.e. over the linear limits of Hooke's law. To be strictly correct, corresponding tensile strength figures should be expressed (e.g. as kg/cm^2), relating the applied force (e.g. the mass of the suspended weight) to the cross-sectional area of the strip. In practice, with sheet materials such as textiles and paper, it is customary to treat the sheet thickness as a constant and to relate the stress only to the width of the strip, i.e. as kg/cm.

The bursting strength is an empirical value that depends on the diameter of the disc tested in accordance with appropriate standards, such as BS 3137:1995 for paper and BS 4768:1991 for textiles. It is readily determined using apparatus such as the tester illustrated in Figure 11.26, which applies a hydraulic load (up to 70 bar) by the hand wheel, to a sample clamped over the base plate. A renewable rubber diaphragm beneath the base plate protects the sample from direct contact with the hydraulic fluid.
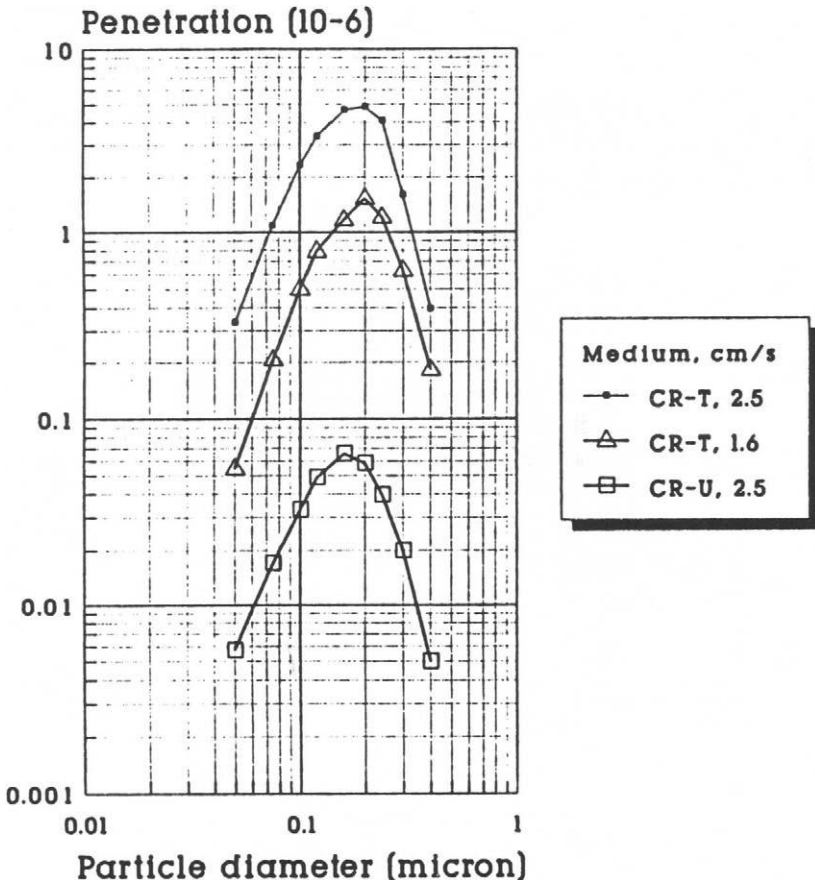

Figure 11.24. Plot of penetration versus particle size for two different media and velocities, showing Most Penetrating Particle Size (MPPS)[15].

Table 11.10   Aerosols and international standards relating to HEPA and ULPA filters

Standard or guideline	Country	Aerosol material	Average size of aerosol (μm)	Parameter measured
BS 3928	U.K.	NaCl salt	0.60	Mass related
Eurovent 4/4	Europe	NaCl salt	0.60	Mass related
AFNOR X44013	France	NaCl salt	0.60	Mass related
AFNOR X44011 uranine	France	Uranine salt	0.15	Mass related
DIN 24,184	Germany	Paraffin oil	−0.45	Area related
M 7605	Austria	NaCl salt	0.60	Mass related
SWKI 84-2	Switzerland	NaCl salt	0.60	Mass related
Mil Std 2282 (DOP)	U.S.A.	DOP oil	0.30	Area/quantity related
Mil Spec F-51068F	U.S.A.	DOP oil	0.30	Area/quantity related
IES-RP-CC001.3-93	U.S.A.	DOP oil	0.30	Area/quantity related
IES-RP-CC007.1-92	U.S.A.	DOS oil+	~0.18	Area/quantity related
prEN 1822 (Jan. 1995)	Europe	DEHS oil	MPPS	Area/quantity related
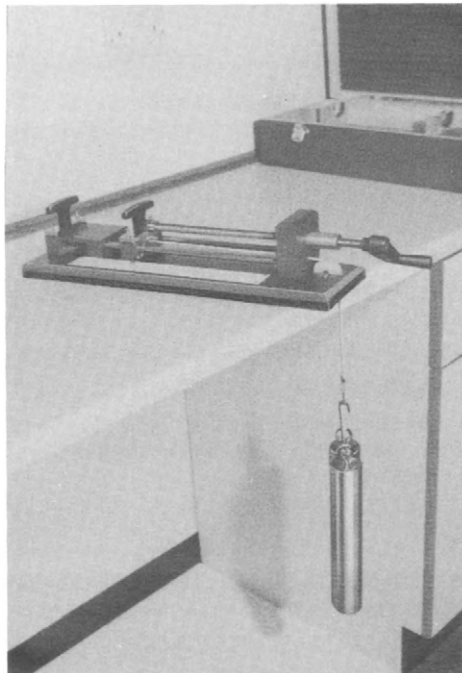
Figure 11.25. A Fryma Fabric Extensiometer.

11.3.2 Stiffness

The resistance to bending of paper is defined in BS 3748:1992 (ISO 2493:1992) as the force (in N or mN) causing deflection through 15° when applied at the free end of a bending length of 50 mm.

The apparatus illustrated in Figure 11.27 is an example of a tester that is applicable to a wide variety of materials, including paper, card, textiles and plastic, in thicknesses up to 6 mm. A sample to be tested is fixed in the clamp so that its bottom 6 mm overlap with the triangular vane attached to the top of the pointer, which is pivoted at its centre. During the test, the sample is moved against the vane, thus displacing it and the pointer until the sample bends and releases the vane; the range of force needed to achieve displacement can be selected to suit the stiffness of the sample by adjustable weights attached to the lower half of the pointer. The maximum displacement reading of the pointer is automatically set to force (mg) and hence to bending moment (g/cm or mN/m).

An empirical test of stiffness used in the textile industry (BS 3356:1990) measures the overhanging length of a horizontal strip necessary for it to bend through an angle of 41.5° under its own weight. With the simple apparatus shown in Figure 11.28, a specimen cut to size (25 mm × 200 mm) using a template is placed on the horizontal plate, with a ruler on top of the sample. The ruler and sample are then moved forward slowly until the bending fabric aligns with two engraved lines. The data may be reported as bending length, which is

Figure 11.26. A digital bursting strength tester with manual clamping.

half the overhanging length and relates to visual stiffness and drape in the material; or as flexural rigidity, G, that relates to tactile stiffness, and is given by:

$$G = 0.1ML^3$$

where M = cloth mass per unit area, g/m^2, and L = the overhanging length, cm.

11.3.3 Resistance to abrasion

Various devices are available whereby the resistance of textiles to abrasion can be quantified. Examples are the Frazier Schiefer Abrasion Tester, and the Martindale and Taber testers available from SDL International; these subject samples to continuous rubbing under a controlled pressure.

11.3.4 Thickness, compressibility and resilience

The Compressometer in Figure 11.29 permits the evaluation of the thickness, compressibility and resilience of a wide variety of materials (textiles, rubber, felt, non-wovens, paper, films, etc.) especially where observations are required at a range of compressive loads extending from 0.3 mbar to 1.7 bar. The sample to be tested is placed between the instrument base or anvil and the circular pressure foot that is fastened to the vertical spindle; three sizes of pressure foot are available (diameters 25, 75 and 125 mm). The lower dial indicates the thickness of the specimen, while the upper dial shows the pressure applied by a helical spring in the tube between them, this pressure being set manually using a rack and pinion device to compress or relax the spring.

11.4 Characterization of Other Media

The tests discussed above have dealt with the filtration and mechanical property tests for continuous media – sheets and rolls, and special cartridges. Although
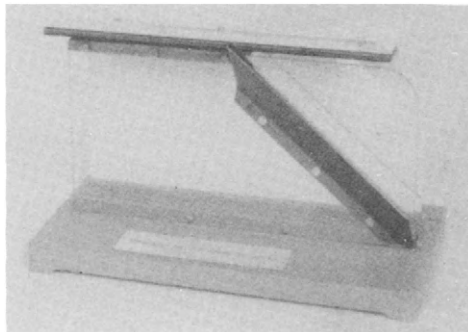

Figure 11.27. Shirley Stiffness Tester.

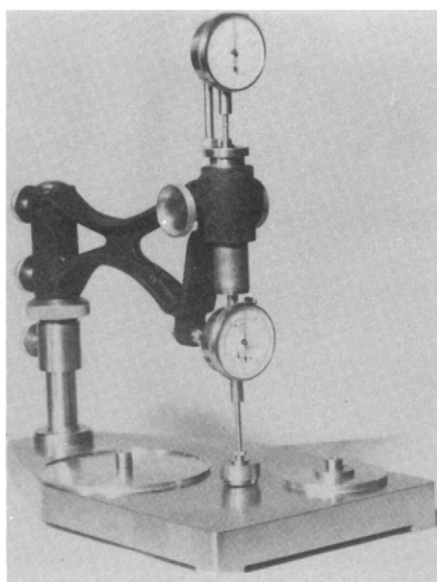*Figure 11.28. Model 4171-D Gurley digital bending resistance/stiffness tester.*

Figure 11.29. A Compressometer for evaluating thickness, compressibility and compressional resilience.

membranes are mainly of this type of material, there are still some special features of tests for membrane media, which are discussed now, together with those for loose granular media.

11.4.1 Membranes

The filtration action of micro- and ultrafiltration membranes is very similar in principle to that of other continuous media. Hence most of the testing methods already described have their equivalents in the testing of the properties of membranes. The delicacy and very fine pore structure of membranes, however, result in some major differences in test methods and procedures.

Characterization methods for porous membranes have already been introduced in Chapter 8. They can be divided into two areas: structure related parameters and permeation related parameters. Certain tests are also used to establish the integrity of membranes in specific applications. The direct measurement of pore statistics is routinely carried out by electron microscopy: by SEM (scanning electron microscopy) and TEM (transmission electron microscopy).

Table 11.11 summarizes the various test procedures used for micro- and ultrafiltration membranes, or for filters incorporating these membranes. It should be noted that the asymmetric structure of most ultrafiltration membranes, with top layer pore sizes in the range 20–1000 Å, means that many of the methods of characterization of microfiltration membranes and other continuous media cannot be applied. Bubble point and mercury intrusion

Table 11.11   Tests for characterizing membranes or membrane filters

Principle of test	Medium	Characteristic
Microfiltration membranes		
Air diffusion	Air	Integrity
Bubble point test	Air	Pore size
Cartridge retention test	Water	Filtration efficiency
Flow rate vs differential pressure	Water	
Particle shedding test	Water	
TOC tests	Water	
Resistivity test	Water	
Bacteria passage test	*Pseudomonas diminuta*	Sterility
Mercury intrusion test	Hg	Pore size and pore distribution
Latex sphere test	Latex sphere dispersion	Integrity
Water penetration test	Water	Integrity
Electron microscopy (SEM, TEM)		Pore size, shape, distribution, density
Permeation measurements		Water flux for pore size and distribution
Ultrafiltraton membranes		
Gas adsorption – desorption	N_2	Pore size and distribution
Thermoporometry	Water	Pore size and distribution
Permporometry	Gas	Pore size and distribution
Solute rejection	Various solutes	MWCO

methods require high pressures that could damage or destroy the membrane structure: SEM is generally not possible and TEM is not always applicable. The methods that can be used with ultrafiltration membranes include permeation experiments and test methods such as gas adsorption–desorption, thermoporometry, permporometry and rejection measurements.

A recent paper[18], basically describing PMI's porometer capability, has a good review of the main methods of membrane characterization, and advocates their use in combination to achieve the best definition of membrane performance.

11.4.1.1 Bubble point test

This standard test for determining the equivalent pore size and size distribution is described earlier in this chapter. It is a non-destructive test that does not contaminate the filter and thus can be used to test the integrity of a membrane as an alternative to destructive tests such as bacterial retention tests for sterilizing membranes.

11.4.1.2 Diffusion testing

A diffusion test is recommended in high-volume systems with final filter surface areas of 0.2 m^2 or greater. This test is based on the fact that gas will diffuse through the liquid in the pores of a fully wetted filter. The diffusion rate is proportional to the differential pressure across the membrane, and to its surface area. The flow of gas is limited to diffusion through water-filled pores at differential pressures below the bubble point pressure of the material under test.

In the diffusion test, pressure is typically applied at 80% of the bubble point pressure of the material. When there is liquid downstream of the filter, the volume of gas flow is determined by measuring the flow rate of displaced water. The rate of diffusion can also be measured by a gas flow meter.

In industrial settings, the flow rate is often measured on the upstream side of the filter, which does not require a tap into the sterile downstream side. The measurement technique used by many automated devices is pressure decay, after the gas on the upstream side is pressurized to the desired test pressure.

11.4.1.3 Mercury intrusion method

The mercury intrusion test, as already described for continuous media earlier in this chapter, relies upon the penetration of mercury into the membrane pores under pressure. The volume of mercury forced into the membrane is related to the pore size and pressure – the size is inversely proportional to the pressure. As with the bubble point test, a morphology or shape factor must be introduced.

In the test the pressure of mercury is gradually increased, and at a certain lowest pressure the largest pores will fill with mercury. The increasing pressure progressively fills the smaller and smaller pores, until a maximum intrusion of mercury is achieved. At high pressure, however, erroneous results may be obtained due to deformation or damage to the membrane material. In addition, the method also measures dead-end pores, which are not active in filtration. The size range of the test covers 5 nm to 10 μm pores, i.e. it covers microfiltration and some ultrafiltration membranes. Overall it gives pore size and pore size distribution.

11.4.1.4 Water integrity test

This test is relevant to sterilizing-grade hydrophobic filters that are used for the sterile filtration of air streams and gases in many pharmaceutical and biological applications. It is based on the same principles as the mercury intrusion test and may be performed *in situ* after sterilization without any downstream manipulations and can be directly correlated to the bacterial challenge tests. The upstream volume of the housing or filter must be completely flooded with water; pressure is then applied by air on the water volume and the rate of water permeation determined.

A water intrusion test has been developed by Sartorius[19] mainly for testing the integrity of sterile air filters, but also for membrane pore characterization. Its prime characteristic is that it can be used *in situ* to confirm the integrity of a sterile filter, because it is non-destructive, unlike a bacteria challenge test.

11.4.1.5 Bacterial challenge test

A bacterial challenge test system for the evaluation of the effectiveness of high-efficiency membrane filters, as illustrated in Figure 11.30, uses a nebulizer adapted from the original Microbiological Research Establishment (MRE) design for high-pressure operations. The device uses two impinger-type samplers in series upstream, and a silt sampler downstream of the test filter. A minimum challenge of 3×10^8 spores is recommended for filters operating 300 days per year with average flows of 850 dm^3/min.

The Health Industry Manufacturers Association (HIMA) regulations and the US FDA 'Guidelines on Sterile Drug Products Produced by Aseptic Processing' stipulate that a sterilizing filter, when challenged with a minimum concentration of 10^7 *Pseudomonas diminuta* organisms per cm^2 of filter surface,

Key
1. Nebulizer.
2. Test filter housing.
3. Impinger samplers.
4. Silt sampler.
5. Flowmeters.
6. Sterilizing prefilters.
7. Pressure meters.
8. Steam inlets.
9. Steam jacket.
10. Sterilizing effluent filters.
11. Optional prefilter housing.
12. Optional sampling ports.
13. Replenishment reservoir.
14. Draining ports.

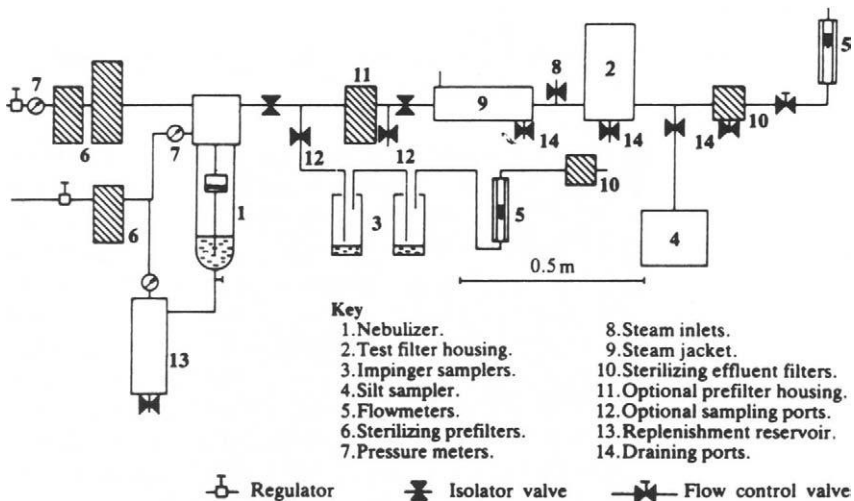Regulator   Isolator valve   Flow control valve

Figure 11.30. Diagram of bacterial challenge testing apparatus.

must produce a sterile filtrate. Filters that produced sterile effluents according to this test were accepted as 0.2 μm in size. In fact, the *P. diminuta* organism is much larger than 0.2 μm: a minimum of 0.3 μm in diameter and 1 μm in length (see Figure 11.31). This means that filters with 'pore sizes' of 0.3 μm and greater could retain the organism.

The bacterial challenge test is a destructive method and it therefore must be correlated with practical non-destructive integrity tests, e.g. bubble point and diffusion methods, to ensure filtration reliability. It has been proposed that a way of overcoming the variability of filter testing methods and to establish realistic, high challenge tests, is to use the log reduction value (LRV) as recommended for liquid sterilization filter tests. The LRV is defined as the logarithm of the ratio of the total number of micro-organisms in the challenge to the number in the filtered fluid. Generally, hydrophilic filters are totally retentive if they exhibit a bubble point of the order of 2.3 bar (i.e. the LRV is greater than 9). Similarly, for hydrophobic filters, a bubble point of 1 bar measured with methanol implies a totally retentive filter. As the bubble point falls, so does the value of the LRV. Such correlations are specific to particular membranes and filters. Filters that retain 100% of the challenge organism *P. diminuta* normally have water bubble point values of 3 bar or more.

Table 11.12   Sphere challenge in SDS

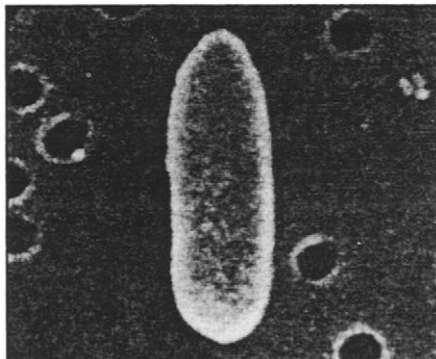| Membrane type	Sphere diameter, μm versus percent relation				
	0.149	0.198	0.220	0.300	0.398
PC	9	99	100	ND	ND
PTFE	31	81	98	100	ND
PVDF	ND	ND	37	60	100
PS	ND	29	46	62	100
N 66	ND	50	41	59	84

Figure 11.31. A Pseudominas diminuta *bacterium and a 0.2 μm hole.*

11.4.1.6 Latex sphere test

Latex spheres make up one of the variety of closely sized inert test dust materials that may be used in the challenge tests that were discussed previously in this chapter. The object of these tests is to characterize the pore size and the filtration efficiency of media.

Typical tests on a variety of membranes, as summarized in Table 11.12, revealed that, with the exception of the track-etched polycarbonate, the membranes all passed latex spheres of size equal to or greater than the manufacturer's rated pore size. Many 0.2 μm rated membranes actually passed spheres larger than 0.398 μm, despite the fact that they had been rated as sterilizing grade 0.2 μm 'absolute' by the industry standard HIMA bacteria challenge, using an organism with dimensions of 0.3 μm × 1 μm.

11.4.1.7 Gas adsorption–desorption

The use of gas adsorption–desorption is frequently practised for the measurement of pore size and size distribution of porous media. Typically nitrogen is used as the adsorbing medium, the method determining the quantity of gas adsorbed (and desorbed) at a particular pressure up to the saturation pressure. A model is required that relates the pore geometry to the adsorption isotherms. The method is limited generally to more uniform structures. Ceramic membranes have been satisfactorily characterized by this method. The method unfortunately includes the contribution made to the membrane structure by dead-end pores.

11.4.1.8 Thermoporometry

Thermoporometry uses the calorific measurement of solid–liquid transition in a porous medium. The method typically uses water as the fluid and is based on the fact that the freezing temperature in the pores of a membrane (i.e. the top layer) depends upon the pore size. The extent of undercooling is inversely proportional to the pore diameter. The method also measures the dead-end pores in the membrane. The material of the medium should have enough elasticity to resist the expansion of water as it freezes.

11.4.1.9 Permporometry

Permporometry is a method that characterizes only the active pores in the membrane. It is based on the blockage of pores by a condensable gas, linked with the measurement of gas flux through the membrane. The pore blockage is based on the same principle of capillary condensation as used in adsorption.

11.4.1.10 Flow porometry

A novel method of porometry[20] has been developed especially for ceramic membranes, although it could, in principle, be applied to other types. In this method, the membrane sample is soaked in a liquid that fills all the (through) pores in the sample spontaneously. One side of the sample is then pressurized with air, which slowly removes the liquid from the pores. The largest pore will become free first, followed by progressively smaller pores, and the air flow rate

can be related to the pore characteristics. Measured gas pressures and flow rates through wet and dry samples are used to evaluate all the required properties of the membrane: largest pore size, mean pore size, pore size distribution, gas and liquid permeability and surface area. A completely automated instrument is used to record gas pressure and flow rates.

11.4.1.11 Solute rejection of ultrafiltration membranes

Ultrafiltration membranes are generally characterized in terms of solute rejection, defined as the fraction of material of a specified size in feed that is retained by the membrane. The terminology of molecular weight cut-off, MWCO, is also often used; this is defined as rejection of 90% of a solute with a particular molecular weight.

The method measures the rejection (or passage) of selected solutes of different molecular sizes under controlled conditions. The solutes should cover the expected size range for 0–100% rejection, and they should not interact with the membrane. Thus solutes such as sodium chloride and glucose (MW 180) are used for the low end (i.e. 0% rejection), and large proteins such as immunoglobulins (MW > 900 000) or blue dextran are used for the upper size range, as these have almost 100% rejection with most membranes. In all, some five or six water-soluble solutes are required for adequate characterization of membrane rejection, as shown in Table 11.13.

There are no standard test conditions for solute rejection characteristics, although recommendations do exist. These include: a pressure of 100 kPa (although this does vary); a temperature of 25°C; a 0.1% (w/v) solution of the challenging solute in a 1% saline solution; and a maximum possible degree of agitation. In addition only a relatively small amount of solute should be filtered to avoid concentration effects. The membrane should be new, cleaned of preservative, and conditioned using a series of soaking, washing and pressurization steps, until a stable and reproducible water flux is achieved.

The results of a series of rejection tests will usually be expressed as a plot of rejection versus MWCO, such as Figure 11.32. Some membranes exhibit sharp cut-offs, with a narrow range of MWCO; others exhibit a diffuse cut-off, with a broad range of MWCO.
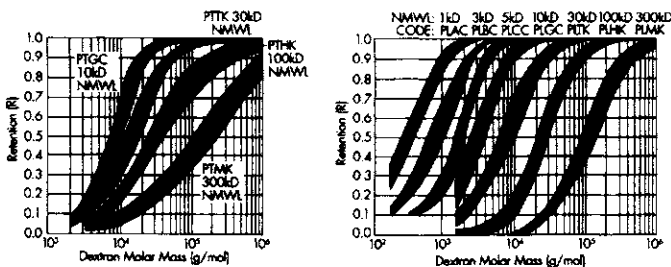

Figure 11.32. Typical rejection characteristics of polyethersulphone UF membranes.

Table 11.13   Typical solute rejection data and solutes used to characterize UF membranes

Solute	Molecular weight	UM05		UM2	DM5	UM10	PM10	YM10	UM20	PM30	XM		
		pH 5	pH 10								50	100A[b]	300[b]
D-Alanine	89	15	80	0	−	0	0	−	0	0	0	0	0
DL-Phenylalanine	165	20	90	0	−	0	0	−	0	0	0	0	0
Tryptophan	204	20	80	0	−	0	0	−	0	0	0	0	0
Sucrose	342	70	80	50	−	25	0	−	−	0	0	0	0
Raffinose	594	90		−	−	50	0	10	−	0	0	0	0
Inulin	5000	−		80	70	60	−	45	5	0	0	0	0
PVP K15	10 000	−		90	85	65	35	75	−	−	−	−	−
Dextran T10	10 000	−		90	90	90	5	−	−	−	−	−	−
Myoglobin	17 800	>95		>95	−	95	80	>90	60	35	20	−	−
α-Chymotrypsinogen	24 500	>95		>98	−	>95	>95	−	90	75	85	25	0
Albumin	67 000	>98		>98	−	>98	>98	>98	95	>90	>90	45	10
Aldolase	141 000	>98		>98	−	>98	>98	−	>98	>98	>95	−	50
IgG	160 000	>98		>98	−	>98	>98	−	>98	>98	>98	90	65
Apoferitin	480 000	>98		>98	−	>98	>98	−	>98	>98	>98	>95	85
IgM	960 000	>98		>98	−	>98	>98	−	>98	>98	>98	>98	>98

[a]   Measured at 55 psi (3.8 kg/cm^2), except where noted by footnote b.

[b]   10 psi (0.7 kg/cm^2).

Overall MWCO values of membranes are no more than a guide to a particular application. The arbitrary setting of the cut-off value, the variability in test procedures and solutes and the effects of other operating parameters mean greater precision is not as yet possible.

Other factors that affect the rejection measurements are the membrane material, the shape and flexibility of the macromolecule solute, its interaction with the membrane, the membrane configuration, concentration polarization phenomena, and interactions between different solutes or macromolecules. Secondary membranes can be formed on the membrane surface, which impede the permeation of lower molecular weight solutes. The adsorption of solutes by the membrane can also result in reduction in the apparent rejection of the ultrafiltration membrane. Overall, therefore, these membrane surface phenomena must ideally be taken into account in characterizing the true (intrinsic) membrane properties. This generally involves indirectly measuring the concentration of solute at the membrane surface using equations describing boundary layer phenomena, or alternatively eliminating these effects by experiments at low-pressure driving forces and low feed concentrations.

11.4.2 Granular media

The testing methods outlined in Sections 11.2–11.4 are concerned with sheet or roll materials, or with special cartridge formulations, and they do not apply (except those describing filter efficiency determination rather than specifically medium behaviour). The loose materials described in Chapter 10 need to be treated in a different way, and the methods of so doing are described in Section 10.3.1.

11.5 Organizations Involved in Filter Media Testing

The testing of filter media is undertaken widely, by a range of organizations:

- manufacturers of media have test rigs for their own products, and some provide a general testing service;
- some filter makers also have media test rigs available for use by others;
- most universities and research institutes that have an interest in filtration have filter or filter media test rigs, and some use them to provide testing services;
- there are a number of commercial suppliers of testing equipment who offer some kind of testing service, usually keyed to the company's range of equipment; and
- there is a very small number of fully independent testing companies, of which IFTS is the best known example.

As well as the makers of testing and particle characterization equipment, there are also the suppliers of test particulates (dust) most of whom at least give advice as to how their products should be used.

The following notes make absolutely no attempt to be exhaustive in their coverage – the 2002 *Filtration & Separation Buyers' Guide* lists nearly 60 organizations under the heading 'Testing Services'. The notes aim only to be indicative of the kinds of service to be expected.

11.5.1 Filtration equipment and media makers

Three-quarters of the names in the *Buyers' Guide* list mentioned above are makers of filters or filter media, all, apparently, offering testing services to outside companies. A not untypical example is that of MikroPul (Charlotte, NC), which provides testing services for bag filter media. These are based upon MikroPul's Filter Efficiency Media Analyzer, which puts media through 100 cleaning cycles, using a standard alumina dust. With PC control, it reports the residual pressure drop after cleaning, the optimum time between cleaning cycles, the weight gain of the medium, and the dust emission in the clean filtrate.

A similar service is offered by the Standard Filter Corporation (Carlsbad, CA), a large maker of filter bags. The company offers a complete in-house laboratory for bag and filter media testing.

11.5.2 Universities and research institutes

Wherever there is a university department with a strong filtration research programme, there is a reasonable chance that contract testing will be undertaken. This is especially true for the University of Sheffield, UK, where a new cartridge test rig for validation trials has been installed, and test facilities for membranes (micro- and ultrafiltration modules) are being installed in 2002, both available for outside contract work (www.shef.ac.uk/-cpe/staff1/burgoyne.html).

University College London (UCL), whose Civil Engineering Department was the home for the work reported in Chapter 10 by Ives, continues to have a testing service for granular media for use in water treatment deep bed filters (www.ucl.ac.uk/civileng/research/resenvir.html).

The Harwell Laboratory, UK, of AEA Technology has, for many years, been the headquarters for the Separation Processes Service, for which filtration, especially of gases, has been a key technology. The service no longer exists in its original research 'club' form, but test work is still undertaken for external clients.

There are several research institutes working in associated fields that have filtration interests, and which either produce testing equipment or undertake tests of specific media, such as the Shirley Institute for textiles, and a range of paper industry bodies, such as TAPPI.

11.5.3 Testing equipment suppliers

The key components of a filter test system are a source of fluid (usually air or water), a source of particles of known size and size distribution, a device that will measure the properties of the aerosol or slurry upstream and down stream of the

filter medium under test, and a pressure drop indicator across the medium. To these must be added the instruments used to characterize the macro- and microstructure of the medium. All of these are available from a number of companies, some specializing in filter test equipment, some parts of larger instrumentation companies. Within this range, there are several such companies that offer testing services to customers or potential customers, usually using their own equipment.

Typical of such companies is BIRAL (Bristol Industrial & Research Associates Ltd, UK), which supplies complete testing equipment for disposable respirators, filter cartridges, cabin air filters and associated media, and for HEPA and ULPA panel filters. The most recent addition to the range is a complete scanning test system[21] to enable the determination of MPPS data to match the needs of EN 1822 (www.biral.com).

Another example is offered by PMI (Porous Materials Inc) of Ithaca, NY. PMI is basically a maker of pore characterization equipment, whose porometers have been mentioned earlier in this chapter, and whose equipment can cover fluid flow rates, pressure drop, separation efficiencies, rates of clogging, absolute filter ratings and filter integrity (www.pmiapp.com).

11.5.4 Testing service organizations

Although Palas GmbH (Karlsruhe, Germany; www.palas.de) is also a testing equipment maker, it has a significant testing services business, based upon its general expertise in aerosol technology. It received *Filtration & Separation*'s Product Achievement Award in 1999 for 'Testing and Monitoring Equipment', its expertise in testing covering deep-bed filter media and surface filter media for gas or liquid filtration. It has a test rig designed to satisfy VDI guidelines 3926, Part 1.

IFTS (Institut de la Filtration et des Techniques Séparatives) is based at Agen in southwest France (www.ifts-sls.com). It is perhaps the only truly independent testing company. Its activities are very well known, and its managing director, Christophe Peuchot, is active in testing methods research, and is concerned in the development of many international standards. IFTS specializes in liquid filtration systems[22], and opened a new filter test facility in 2000. Much of the new expansion is concerned with filter testing, but there is an appreciable amount of media testing undertaken as well.

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