CLEANROOM TECHNOLOGY

Integrity testing of HEPA filters

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In situ aerosol filter testing is a black art to many, but the new ISOEN14644-3 Test Methods standard incorporates two in situ test methods that are discussed here by Neil Stephenson of DOP Solutions (Now ATI, Air Techniques International)

The industry is still subject to large costs through in situ filter test failures that can result from poor testing and poor application. Employing the correct test method and ensuring all the necessary hardware is in place for regular testing will go a long way to reducing the test failure rate. Good testing also makes it easier to identify where the leak is – rather than the scatter-gun approach that can occur where the filter or the filter housing is blamed and many hours of production time are lost in resolving disputes.

The definition of a cleanroom may be determined by the "standard" applied and ultimately the quality requirement of the sector. The pharmaceutical and electronics sectors have a number of standards against which a cleanroom is judged:

- Electronics – ISO EN14644-1 and 2 and failure of processes normally covered by in-house standards, which are not legislated.
- Pharmaceuticals and hospitals – the ISO EN14644 family of standards of which Parts 1 to 8 refer directly to clean air.

Additionally the pharmaceutical industry is legislated to ensure high standards are maintained. The ISO EN14644 standard parts are known as:

Part 1: Classification of air cleanliness

Part 2: Specifications for testing and monitoring to prove continued compliance with ISO 14644-1

Part 3: Test methods

Part 4: Design, construction and start-up

Part 5: Operations

Part 6: Vocabulary

Part 7: Separative devices (clean air hoods, glove boxes, isolators and mini-environments)

Part 8: Classification of airborne molecular contamination.

HEPA filters are capable of removing 100% of airborne particulate above 5µm. Below that, they are less efficient and typically will remove 95% to 99.9995% in the 0.15µm to 0.25µm particle size range. The grade of the filter chosen for the application determines the overall filtration capability.

Failure or incorrect application of the HEPA filter will lead to a degradation of the clean air. Thus if we test the HEPA filter to determine if it has passed, we can guarantee that the air we are introducing into the cleanroom is particulate-free for the chosen filter quality.

When the HEPA filter is tested in situ and it is leak-free, we can be sure that we are only introducing clean air into our facility. The installed HEPA filters require regular testing to ensure they remain leak-free. A good, leak-free HEPA filter is capable of providing many years of clean air but a leak, caused by damage to the filter, is a cause for concern.

The main reasons for testing the filter are:

1. Qualification – installation testing.

- Conformance to order specification
- Validate quality purchased

2. Periodic testing for leaks

- In situ testing – is the filter still in good operative condition over time?

3. Health & safety requirements.

To overcome the need to test the HEPA filters individually, particle counting to ISO EN14644-1 is often used to prove the facility is free of particulate and meets the standard according to Part 1. This does not prove that the HEPA filters are leak-free. Generally the particle counting is undertaken without processes running or personnel present. Thus there are no or very few particles to be counted. Very few cleanrooms are ever found to fail the particle counting test.

The risk of a leaking HEPA filters is real and data is coming to light that shows a correlation between increased micro-biological counts and leaking HEPA filters. The major risk in the facility is when it is in operation. At this time the filters are removing man- and process-generated particulates from within the facility.

New tests

ISO EN14644-3 was finally published in January 2006. Two methods for the in situ testing of HEPA filters are among a total of 13 possible different cleanroom tests. The two filter integrity test methods (Section B6), though set up in a similar manner, do not produce like-for-like results. Both methods require an evenly distributed aerosol challenge and the scanning of the filter gasket, filter frame and filter media downstream of the filter.

ISO EN14644-3 will for the first time give the international cleanroom community a common standard to work to. The standard is far from perfect and will require a considerable amount of interpretation but it provides a good starting point for most cleanroom metrology requirements.

The first in situ HEPA filter test method in the ISO EN14644-3 standard is the traditional aerosol photometer test method. This is commonly known as the DOP test and uses the aerosol photometer as the measuring device and an aerosol generator to produce an aerosol challenge. This method has been used since the 1950s and appears in many different standards throughout the world.

The aerosol photometry test method in the standard is quoted as being good for measurements to 0.003% by DOP scan testing. This meets most requirements in the pharmaceutical industry. In reality new instrumentation available today can achieve more than 10 times lower than this – typically 0.0003%.

The aerosol photometer uses a near forward scattered light chamber and a photomultiplier tube as its detection method. The forward scattered light is directly proportional to the aerosol mass concentration. The instrument is a continuous real-time detector and usually allows a pre-set alarm point to be set for easy detection of leaks.

The standard requires that the challenge aerosol is used as the 100% reference for the downstream measurement and must be homogeneously mixed resulting in an even challenge to the filter. The measurement downstream of the filter is then directly in percentage penetration. The filter gasket, frame and media are scanned for leaks. The maximum permissible leak is set at 0.01%, although lower limits may be used.

The aerosol photometry DOP scan test is truly non-destructive when carried out correctly. It is repeatable and may be used in all grades of pharmaceutical cleanroom – ISO3 through to ISO9 – dirty and clean. The method is well defined and returns repeatable results between testers.

The second method offered in the standard is the particle counting method. This method also requires the filter is evenly challenged with a known recorded concentration of aerosol, an aerosol diluter and a discrete particle counter.

This method is relatively new and uses a DPC (Discrete Particle Counter) test method. The standard claims this method is good for measurements to 0.000005% and better. This exceeds the requirements for the pharmaceutical industry and is really good only for cleanrooms ISO Class 4 and lower. This is because the particle counter is a batch measuring device and is not designed to make continuous measurements. Background particles in a non-unidirectional cleanroom will cause the detection of false counts, making the method difficult and less reliable.

The particle counter uses a discrete detector measuring the peak light scattered height of the individual particle passing through the detection chamber. It cannot count two particles in the chamber at a time (known as coincidence counting) and therefore a diluter has to be used to measure the upstream aerosol challenge.

The method, because of its reliance on a low challenge, incorporates relatively complex mathematics in order to ensure that the measurement is valid. The pass and fail criteria become less easy to determine and thus the measurement is more difficult to repeat between testers than the aerosol photometry test method.

Both test methods require that the aerosol challenge on the filter is evenly distributed and this should be validated the first time a system is tested. The limit of variance in ISO EN14644-3 is +15% across the challenge area. If this cannot be achieved then the standard requires that methods be employed to mix the aerosol at the point of introduction by the use of mixing methods. A common method for achieving this is the use of a sparge pipe and positive injection pump.

The tests require that an aerosol challenge be introduced upstream of the filter. This is to ensure that there is actually a challenge present against which we can make a downstream measurement. The common oils for this purpose are:

1. poly-alpha olefin (PAO) – Emery 3004 or Durasyn 162

2. shell ondina (EL) food quality mineral oil

3. dioctyl sebacate (DOS)

4. di-2-ethyl hexyl sebacate (DEHS)

5. dioctyl (2-ethyl hexyl) phthalate (DOP)

6. paraffin oil.

The challenge aerosol will traditionally be generated in one of two ways:

- Pneumatic generation by the shearing of the oil using compressed air and a Laskin nozzle.
- Thermal generation by the vaporising of the oil and re-condensing it in atmospheric conditions.

An inert gas is used to propel the oil through the generator.

The pneumatic generator system produces relatively small quantities of aerosol and uses large quantities of air to shear the oil. They are commonly used by filter manufacturers (generally there is no limit on the availability of compressed air) and the in situ testing of LAF and Microbiological Safety Cabinets (refer to BS ISO EN12469 for test methods). The aerosol produced by the pneumatic Laskin generator has a broad distribution – typically a GSD of 1.5 to 1.7 and a count mean diameter of about 0.35µm. This would be defined as a poly-dispersed aerosol. All the oil types given above have been used in the pneumatic aerosol generators.

Thermal generator

Dependent on the type, and with the correct oil, the thermal generator can be used for small to large systems. The aerosol produced is defined as a mono-dispersed aerosol as it has a GSD of 1.2 to 1.4 and a count mean diameter of nominally 0.2µm. This is well placed for measurements at the Most Penetrating Particle Size (MPPS) of the filter. Thus in situ measurements made with the thermal generator allow the determination of both filter leaks and media quality. It is not recommended that DOP, DOS or DEHS are used in thermal generators.

The leak test requires that the filter gasket, filter frame and filter media are scanned. The media is scanned at nominally 2.5mm from the filter face at a rate not exceeding that given in the standard.

ISOEN 14644-3 has made the particle counter and photometry scan test the same speed and this is defined as: “The probe traverse scan rate (Sr) should be approximately 15/WP cm/s. (WP is the width of the probe at 90° to the direction of the scan) For example, when using a 3cm square probe, Sr is 5cm/s.”

While this applies to scanning with a particle counter, the relationship between width of probe and scanning speed is different for an aerosol photometer. This is because the aerosol photometer test method uses a challenge concentration some 1,000 times higher than a particle counter. Additionally the aerosol photometer provides a real time continuous measure-ment. The traditional scanning speed for a “fish tail” scanning probe with an area of 6.45 cm2 (0.8cm x 8.1cm) is 5 cm/s.

A leak is defined as being >0.01% or >10-4 times the upstream challenge. The HEPA filters available today can easily exceed this requirement when ordered correctly; >0.01% may be considered, in some applications, to be a large leak and it may be a requirement in aseptic areas and in Microbiological Safety Cabinets (MBSC) to test to a lower set value – say 0.001%.

Another manner of describing a leak in a high grade HEPA filter, though not in the standard, can be defined as follows:

A leak may be considered to be five times the media penetration of the filter. The media penetration is measured when using a thermal generator and is the overall “background” penetration through the filter. Typically a good H14 filter (EN1822 classification) will provide an overall media penetration of 0.0005% or less. A leak of 0.0025% could thus be described as unacceptable. This definition is only applicable to high grade filters of H14 and above.

There are a number of in situ test methods – some withdrawn but still in daily use and others as used by mainly US-connected countries. Japan, Australia, South Africa, UK, Russia, France, Korea, Germany and US have all had or still have their own national standard in situ test methods. ISO EN14644-3 will, we hope, bring the aerosol filter test method to a common platform. These national test methods have small differences and thus the ISO standard will give all cleanroom testers some common ground.

ISO EN14644-3 now recognizes the need to document and provide the correct injection and sample measurement points.

Homogeneous mixing

Appropriate measures should be taken for the verification of the homogeneous mixing of the added aerosol to the supply airflow. The first time a system is tested it should be determined that sufficient aerosol mixing is taking place. For such validation all injection and sampling points should be defined and recorded.

The upstream aerosol concentration measurements taken immediately upstream of the filters should not vary more than ±15% in time about the average measured value. Concentrations lower than the average will reduce the sensitivity of the test to small leaks, while higher concentrations increase the sensitivity to small leaks. Further details as to how to conduct the air-aerosol mixing test should be agreed upon between customer and supplier. ASME N510-1989 and IEST-RP-CC034.1:1999 may also be of value.

The aerosol filter test method is greatly improved by:

1. Determining if there is sufficient mixing distance between the point of injection and the filters to be tested. Where the distance is insufficient then a sparge pipe (mixing manifold) should be used. This may be permanently installed or temporarily for the test. The manifold used should be dedicated to the installation even if it is removed between tests.

2. Installing and designating a specific injection port for the aerosol introduction.

3. Installing a suitable upstream aerosol sample port. Typical requirements are 10cm from the filter face and in the middle of the filter area. A suitable upstream sample port will have an ID not less than 5mm and not greater than 8mm.

4. Access to the filter on the down stream side of the filter so that it may be scanned. Where this is not available some engineering is required to enable the filter to be scanned. If it is not practical to manually access the area, a longitudinal scanning probe can be employed as a permanent installation. The longitudinal scanning probe is particularly useful for the scanning of filters in safe change housings.

5. The SOP should make reference to the ports used and the one-off test to prove even distribution of the aerosol mixing.

HEPA filter testing will, with the advent of ISO EN14644-3, become more evenly applied throughout the world. Correctly employed, this should result in fewer filter failures and improved clean air supply to cleanrooms. It is worth remembering that a cleanroom whose HEPA filters have been tested and passed will not fail a particle count unless the particles are generated within the facility being tested.

Long term, correct application to the HEPA filter test should reduce costs – particularly the costs not always counted and seen – and down time caused by having to repeatedly remove and re-insert HEPA filters which are apparently leaking.

This paper was presented at the 3C show held in Birmingham in February 2006.