Lessons Learned from Particulate Characterization Laboratory Anomalies*

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ABSTRACT

The White Sands Test Facility chemistry laboratory provides quality control for cleanroom operations including cleanliness verification of aerospace hardware by particulate counts and non-volatile residue determinations, particulate counts for liquid hypergolic propellants, gaseous helium and nitrogen propellant pressurizing agents used for ground support equipment and flight test article valve actuation, gaseous oxygen primarily used for component testing, and deionized water for refurbished propellant hardware flushing. Cleanliness verification includes particulate counts and non-volatile residue determinations to industry standard, NASA, and program specifications and levels. Particulate counts are typically to customer-specified specifications and levels including JPR 5322.1H (2016) Levels 50 and 100, Orion (MPCV 70156. Revision H (2018)) Level 100, RPTSTD-8070-0001 Revision 3 (2022), and IEST-STD-CC1246E (2013) Levels 50 and 100. The laboratory issues high pressure filter holders containing membrane filters to test operations personnel, who collect samples by flowing the required volumes of fluid through the filter holder, and the filter holder is returned to the lab for counting. A passing particulate count is required before testing may proceed. Rapid data reduction and issuing of reports indicating a pass or fail of the particulate specification are required. Corrective action and resampling invariably occurs if a sample fails. Consequently, the laboratory must maintain the highest degree of reliability to facilitate quality data used to decide if testing may proceed. Experience and continual improvements have enabled reliability. However, anomalies attributed to lab processes and hardware including filter holders, membranes, and Petri dishes have been encountered. This paper presents a summary of problems, solutions, successes, and lessons learned from particle counting experience for over 35 years.

INTRODUCTION

Reliable particulate counts are necessary for the verification of gas stream and liquid sample particulate specifications. There are causes a particulate count fails specification. One cause a particulate count fails specification is the system produces particulate and contaminates the sample media in excess of the allowable specification. Another cause is the sampling hardware (filter holder and/or membrane) is contaminated with or produces particulate resulting in a failed particle count of an otherwise clean sample. The cause of contamination cannot be revealed when contaminated sampling hardware is used to sample a contaminated system. Conversely, for a particulate sample to pass specification, neither the sampling hardware nor the system may contribute particulate outside of specification. System cleanliness is a variable that cannot always be controlled, such as in the case of newly assembled and unproven systems compared to existing systems with a history of passing specifications. Sampling hardware integrity and cleanliness thought to be controlled proved otherwise given a recent series of failures, coupled with knowledge that sampling hardware and processes related to processing and counting a system sample have failed. Consequently, lessons learned from this series of failures and related experiences are presented. This manuscript presents several lessons learned and

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recommendations for particulate sampling hardware and quality control measures that can be taken for proactive measures to prevent recurrence.

BACKGROUND

The White Sands Test Facility Chemistry (WSTF) Laboratory provides quality control for cleanroom operations including cleanliness verification of aerospace hardware by particulate counts and non-volatile residue determinations, particulate counts for liquid hypergolic propellant specifications or customer requirements, gaseous helium and nitrogen used for propellant pressurizing agents and ground support equipment valve actuation, gaseous oxygen primarily used for component testing, and deionized water for refurbished propellant hardware flushing. Cleanliness verification for cleanroom operations includes particulate counts and non-volatile residue determinations to industry standard, NASA, and program specifications. Particulate counts are to customer-specified specifications and levels including JPR 5322.1H (2016) Levels 50 and 100, Orion (MPCV 70156. Revision H (2018)) Level 100, RPTSTD-8070-0001 Revision 3 (2022), and IEST-STD-CC1246E (2013) Levels 50 and 100. Determination of non-volatile residue (NVR) is also performed routinely but this activity is outside the scope of focus on particulate counting addressed in this manuscript.

Definitions applicable to particulate include:
Particulate: Matter with observable length, width and thickness usually measured in microns; this definition includes fibers. Membrane filters are also occasionally scratched in handling, leaving pieces of filter material as particulate. Some of the propellant membrane filters can be observed with partially dissolved ink on the grid, leaving shiny, black pieces of ink as particulate. In case of uncertainty, the object in question must be counted as a particle. Source: JPR 5322.1H and WSTF.

Fiber: A particle whose length-to-width ratio is in excess of 10:1 (if not visible it may be referred to as a microfiber). Source: JPR 5322.1H.

Allowable particulate limits for JPR 5322.1H (2016), KSC-C-123J (2009), and RPTSTD-8070-0001 Level 50 are provided in Table 1 and for JPR 5322.1, KSC-C-123J, and RPTSTD-8070-0001 Revision 3 (2022) Level 100 are provided in Table 2.

Table 1. JPR 5322.1, KSC-C-123J, and RPTSTD-8070-0001 Particulate Limits for Level 50

Size Range (μm)	Maximum Number of Particles Allowed
<15	Unlimited*
15 to 25	17
>25 to 50	8
>50	0
* "Unlimited": Particulates of this size and smaller are not counted. The sample will be rejected if particulate accumulation/silting interferes with analysis. No silting permitted.	

Table 2. JPR 5322.1, KSC-C-123J, and RPTSTD-8070-0001 Particulate Limits for Level 100

Size Range (μm)	Maximum Number of Particles Allowed
<25	Unlimited*
25 to 50	68
>50 to 100	11
>100	0
* "Unlimited": Particulates of this size and smaller are not counted. The sample will be rejected if particulate accumulation/silting interferes with analysis. No silting permitted.	

Note the 0 allowable particulate in the >50 or >100 size ranges, respectively. Experience shows particulate samples rarely, if ever, fail based on particulate in the ranges where non-zero numbers of particles are allowed, whereas the most common failure is 1 or more particle in the 0 allowable range. Specification IEST-STD-CC1246E Levels 50 and 100 are not used for internal laboratory quality control purposes because these levels allow 1 particle greater than 50 or 100 µm. Although failures typically occur when particulate greater than the 0 allowable in the >50 or >100 µm range are found, an exception is when a filter is so contaminated with particulate, yet not silted, and it immediately fails the specification, but a statistical count is required to obtain the average particulate distribution. In this case, particulate in most if not all ranges fail the specification. Statistical counts are performed usually for engineering information only. A statistical counting technique involves counting the particulate area in representative squares on the membrane filter. For example, if a filter holder exposes an estimated 100 squares to the sample, the count on one square is multiplied by 100 to obtain a statistical count.

Particulate is sampled for 1.0 m^3 (35.31 ft^3) of gas and is constant for all gas samples. Particulate samples are collected for gas supplies such that 1.0 m^3 (35.31 ft^3) of effluent gas is sampled when a system is evaluated by purging at the maximum operational flow rate. Formulas exist for calculating the time required to sample based on the gas source pressure and the filter membrane pore size, e.g., 5 or 10 µm.

Cleaned surface areas are sampled for particulate with a flushing solvent on the basis of 100 mL of solvent used to flush 0.1 m^2 (1.1 ft^2) of cleaned surface area and is constant for all cleanliness verification samples. Areas of threaded surfaces and component geometry are considered such that 100 mL of solvent represents a surface area of 0.1 m^2 (1.1 ft^2). Formulas exist to calculate the surface area of batches of cleaned components so that a representative flush solvent volume is collected.

Liquid propellants are sampled for particulate using the minimum volumes specified by the corresponding military specifications to report results in mg/L. No particulate count requirements exist in the military specifications for propellant hydrazines and dinitrogen tetroxide oxidizers, although most if not all NASA programs have particulate requirements for liquid propellants. All NASA spacecraft or propulsion test programs impose particulate size distribution limits on the operational fluids, including propellants. All hypergolic liquid propellant samples submitted to the WSTF laboratory for specification analysis, typically a military specification, are subjected to a particle count. Particle counts of the propellant from a system help assure the overall cleanliness requirements of the system, including the test article, run tanks ready storage units, and interfaces, are met. Propellant sampling ports enable sample collection for verification of system cleanliness. Where hypergolic propellant engines necessitate stringent contamination controls, systems can sometimes be flushed with known high-purity propellant and sampled to verify system cleanliness, rather than disassembled, cleaned, and reassembled.

All membrane filter holders used at WSTF are high pressure, stainless steel with either a 4-bolt (of heritage vintage long discontinued by Millipore®†) or 6-bolt configuration with a 47mm filter screen where the filter membrane is placed. Filter holders are rated for 10,000 pounds per square inch (psi) of pressure. New filter holders are initially precision cleaned to customer specifications (Level 50, 50A, 100, etc.) and may be reissued and reused for the particulate cleanliness level last sampled and passed. For Level 50A, “50” refers to particulate level and “A” refers to NVR level expressed in mg per 0.1 m^2). Membrane filters used for particulate sampling are Millipore hydrophobic, polytetrafluoroethylene (PTFE), 47 mm. Millipore provides no claim of particulate cleanliness of membrane filters. Consequently, filter membranes must be pre-counted before use and if they do not pass user criteria or specification, they are discarded.

Particle count forms are used to record the number of particles in each size bin. Existing particle count forms provide columns for a blank (the pre-count), total (the total after sampling), and a net (the total minus the blank or the particulate normalized to 100 mL volumes). Since the 0-0-0 pre-count became standard, there was nothing to subtract. The origin of the 0-0-0 pre-count criteria predates any of the authors’ employment at WSTF and had been accepted as tradition.

† Millipore is a registered trademark of Merck KGaA, Darmstadt, Germany

OBJECTIVES

This manuscript presents an overview of lessons learned regarding particulate contamination in a contamination control quality assurance laboratory.

EXPERIMENTAL

All membrane filter handling, assembly and disassembly of filter holders were performed in a Class 100 flow bench equipped with a filtered nitrogen supply with an in-line 50-µm membrane filter and an illuminated magnifier. Filter holders in the flow bench were supported on metallic tripods. Membrane filters were installed and extracted from filter holders using particle free, visually clean when inspected at 50X magnification forceps. An adjacent work bench equipped with a vise was used to clamp filter holders when torquing to specification with a torque wrench set at 150 in-lb. Filter membranes were Mitex‡ Membrane Filter, 10-µm, 47-mm-diameter, 130-µm thick, hydrophobic, white, plain, polytetrafluoroethylene, screen filter; Millipore® part number LCWP04700 or Mitex™ Membrane Filter, 5-µm, 47-mm-diameter, 170-µm thick, hydrophobic, white, plain, polytetrafluoroethylene, screen filter; Millipore® part number LSWP04700. Falcon Petri dishes (Corning Falcon #351006) were 50-mm x 9-mm polystyrene bacteriological Petri dishes with tight-fit lids, disposable, and sterile and were used to contain membrane filters after pre-counting and when removed from filter holders.

One box each of twenty-five 10-µm (47-mm PTFE Mitex™ LCWP04700, Batch R1NB70369) and 5-µm (47-mm PTFE Mitex™ LSWP04700, Batch R1HB25417) Millipore filter membranes and Falcon Petri dishes (Corning Falcon #351006, 50 x 9 mm) were submitted to Component Services Section for rinsing with HFE-7100. Each rinsed filter was placed in a rinsed, dried Petri and bagged. Filters were not removed from the Petri dishes for counting. Membrane filters were then counted to JPR 5322.1 Level 50. Results are shown in Table 3.

A filter holder is built as follows. A cleaned high pressure filter holder or one showing a passing particle count of required cleanliness is disassembled in the Class 100 flow bench. Membrane filters were removed from their original container in the Class 100 flow bench, handled only with forceps, placed in lightweight, and pre-counted in Petri dishes with the lids on. Passing pre-count criteria was previously 0-0-0 for allowable particulate in countable ranges and changed later to passes specification. Any filter membranes failing pre-count criteria were discarded. Pre-counted membrane filters were stored in their Petri dishes with lids on until assembly into a cleaned or clean filter holder in the Class 100 flow bench.

Sampled filter holders were disassembled, the membrane filter removed to a Petri dish using forceps, the Petri closed and was transferred to an adjacent Class 100 flow bench containing optical microscopes. Size distributions and physical appearance, specifically metallic or non-metallic in appearance, were determined using an optical microscope equipped with a mechanical stage and a calibrated eyepiece scale.

RESULTS AND DISCUSSION

Table 3 results show over half of the of HFE-rinsed filter membranes in both 5- and 10-µm filter membranes failed a 0-0-0 pre-count and would be discarded using that criteria. The observation that 88% of the 5- and 10-µm filter membranes passed Level 50 led to revisiting the criteria for pre-counts and a decision was made that if a filter membrane passes specification, it is acceptable for use, and the 0-0-0 pre-count criteria was abandoned. And although the HFE-rinsing was not pursued at this time as a means of pre-cleaning the filter membranes at this time, it may be revisited.

‡ Mitex is a registered trademark of Merck KGaA, Darmstadt, Germany

Table 3. 5- and 10-µm Membrane Filters Rinsed with HFE-7100 Counted to JPR 5322.1 Level 50

Membrane Filter (25 each)	0-0-0 Pre-count Criteria		Pass Level 50 Criteria	
	Pass	Fail	Pass	Fail
5 µm	12 (48%)	13 (52%)	22 (88%)	3 (12%)
10 µm	10 (40%)	15 (60%)	22 (88%)	3 (12%)
Total out of 50	22 (44%)	28 (56%)	44 (88%)	6 (12%)

A Really Bad Day

The first anomaly (February 13-14, 2024) was a series of particulate samples taken from a Building 803 oxygen system in which 16 successive failures were encountered over two days for a system being sampled to JPR 5322.1 Level 50 using 10-µm filter membranes. This system required one passing sample. Late in the first day after 11 successive failures did the lab seriously consider the sampling hardware being issued might be contributing to the failures and that the failures might not be entirely, due to the test system. All the failures were due to 1 or more particles in the zero allowable >50 µm range. After a total of 16 failures were experienced, one filter holder, which failed with 2 particles >50 µm was rebuilt with a new, pre-counted filter membrane and it passed Level 50 and ended the sampling event. The overall pass rate for the 17 samples was 6%. Fifteen filter holders were submitted for precision cleaning to JPR 5322.1 Level 50A.

The second anomaly (February 15-16, 2024) was a set of particulate samples collected from nitrogen manifolds in Test Cell 110 high pressure oxygen panel onboard spacesuit control assembly test port samples being sampled to JPR 5322.1 Level 50. Five sequential sampling events using 10-µm filter membranes were required to pass 1 sample. The overall pass rate for the 5 samples was 20%. Four filter holders were submitted for precision cleaning to JPR 5322.1 Level 50A.

The third anomaly (March 1, 2024) was a set of particulate samples collected from nitrogen manifolds in the Pressure Calibration Laboratory being sampled to JPR 5322.1 Level 50. Two locations were sampled using the 10-µm filters that were supposed to have been taken out of service. The first location Manifold 328388 passed the first sample. The second location failed the first two samples. A 5-µm filter was used for the third sample and it passed using the filter holder rebuilt after the last failure with a 10-µm filter. The overall pass rate for the four samples was 25%. Two filter holders were submitted for precision cleaning to JPR 5322.1 Level 50A.

The fourth anomaly was a set of North High Bay Orion Orbital Maneuvering System Engine (OMSE) Clean Room samples taken and counted in late February and concluding in mid-March 2024. Thirty-two test ports supplying nitrogen or helium were sampled to Orion Level 50 or Orion Level 100. The first 4 test ports sampled failed specification. There was a pause in sampling and the customer was consulted and accepted a suggestion that reduced costs could be realized if the laboratory rebuilt and resubmitted failed filter holders for sampling rather than submitting them for precision cleaning before reissuing them. When this set of samples was concluded, the 32 test ports required 48 filter holders to pass. Twenty-three (23) or 72% of these passed specification on the first sample, 5 or 16% passed on the second sample, 2 or 6% passed on the third sample, 1 or 3% passed on the fourth sample, and 1 or 3% passed on the fifth sample.

Investigations

Experienced personnel-initiated investigations during and following these anomalies.

Filter membrane pre-counts to 0-0-0 were verified as accurate. Particulate counting technique was verified accurate by different technicians reproducing particle counts on the same membrane filter. It was considered contamination could be occurring within the filter holder as was once observed with ink shedding in a filter holder that was not sampled. A newly assembled filter holder was let sit for 10 minutes, then disassembled and the filter membrane examined under an optical microscope. The filter membrane showed delamination appearing at the O-ring compression area between the filter housing and the filter membrane and was producing what appeared to be clear fibers of various lengths.

Visual observation of failed 10-µm filter membranes showed distinct delamination of membrane fibers in the compression region of the O-ring. These were described as “clear fibers” and were characteristic of many of the non-metallic failures experienced. Clear fibers as long as 800 µm or more were observed and attributed to catastrophic membrane failures. Interim measures to not count these clear fibers in the compression region were implemented but failures were still experienced as clear fibers were observed well within the filter boundaries as well as random metallic particles. Selective counting criteria was abandoned in favor of not using the 10-µm filter membranes and using 5-µm filter membranes, which were not experiencing delamination. The batch of 10-µm filter membranes being used were taken out of service and since no other batches were available to evaluate, focus was turned to the available 5-µm filters.

Pre-counts showed a large failure rate of 10-µm filters with random clear fibers, yet gas streams sampled from the building 803 oxygen system, Test Cell 110 high pressure oxygen panel onboard spacesuit control assembly, Pressure Calibration Laboratory gas ports, and North High Bay Orion Main Engine Related Payload/Flight Hardware Processing (OMSE) Clean Room experienced random failures due to visually characterized random clear fibers, globs resembling grease, and metallic particles. Several filter holders had been fitted with 10-µm filters and were issued before a problem with the10-µm filters (Millipore Batch R1NB70369) was discovered, so the 10-µm filters were set aside and replaced with 5-µm filters. After the 10-µm filters were removed from service, customers were informed 5-µm filter membranes were being used so they could adjust their sampling parameters.

The Class 100 flow bench used for filter holder assembly, disassembly, and filter membrane handling was identified as a potential source of contamination in case of a failure since its last certification or monitor date. Flow bench failure was ruled out after a time-out in which the flow bench was tested and recertified with no problems observed. The flow bench in which particulate is A Class 100 (ISO Class 5) flow bench allows a maximum of 100 particles ≥ 0.5 µm and 0 particles ≥ 5.0 µm per 0.028 m^3 (1.0 ft^3). The Class 100 flow bench allowable particulate levels are well below the particulate size bins encountered in Level 50 and Level 100 particulate specifications. Particulate monitoring was also performed in the areas where personnel were performing manipulations and no spikes in particulate above baseline levels were observed.

A pre-counted (0-0-0) 5-µm membrane assembled in a 4-bolt filter holder sat undisturbed for 10 minutes after assembly then the filter holder was disassembled, and the filter membrane was examined by optical microscopy. Random metallic particulate and non-metallic particulate visually observed as globs were observed. The sizes of several of these particles failed the Orion Level 100 particulate specification. Visual examination of the filter housing showed significant metal abrasion, rough surfaces, grossly extruded O-ring, and suspected potential for particulate formation from metal surfaces and the O-ring. Extruded loose O-ring material was suspected to be a source of observed nonmetallic globs.

A pre-counted 5-µm membrane in a 6-bolt filter holder that sat undisturbed for 10 minutes after assembly showed was removed and examined by optical microscopy. Insignificant particulate was observed, and this passed the Orion Level 100 particulate specification.

Observations that pre-counted and passing specification filter membranes accumulated particulate without sampling led to suspicion that filter holders as well as membrane filters could be responsible for producing particulate. Since delamination of the 10-µm membrane filters was already established and the responsible batch had been taken out of service, the filter holders themselves were given careful attention.

Visual examination of the filter housing showed definite but less significant metal abrasion, definite but less significant rough surfaces, and no observable O-ring extrusion. However, there was suspected potential for particulate formation from metal surfaces.

Figures 1-5 are images of 4-bolt and 6-bolt filter holder inner surfaces.

Figure 1. 4-Bolt Assembly
Note surface wear, abraded metal, and the O-ring extruding from the gland.

Figure 2. Close-Up of 4-Bolt Assembly
Note surface wear, abraded metal, and the O-ring extruding from the gland.

Figure 3. 6-Bolt Assembly
Note reduced but visible surface wear and abraded metal compared to a 4-bolt assembly.

Figure 4. Close-Up of 6-Bolt Assembly
Note visible surface wear and abraded metal.

Figure 5. Side-By-Side Comparison of 4-Bolt and 6-Bolt Assemblies

The 4-bolt assemblies are nominally 60 years old and show more visible wear than the 6-bolt assemblies. Many of the 6-bolt assemblies are at least 20 years old. Nevertheless, all filter holder surfaces showed abraded metal surfaces with the potential to release metallic particulate and compressed O-rings with the potential to release PTFE particulate with both metallic and O-ring particulate capable of being trapped on the membrane filter surface, be counted as particulate, and contribute if not cause particulate specification failure. In addition, a 4-bolt design is preferred over a 6-bolt because with less bolts, there is less chance of producing metallic and grease particulate. Some of the oldest 4-bolt assemblies were fitted with pipe-threaded connections creating areas of entrapment for particulate, and these were taken out of service. The lab is currently looking at options to replace existing Millipore filter housings with a more practical design and with O-ring glands and O-rings that fit properly. In future applications, pressure rating might be reduced over existing 10,000 psi filter holders because high pressure is rarely encountered when sampling from a pressurized source to ambient.

Other enhancements included the installation of a desktop illuminated magnification lamp for enhanced visual observation capabilities and installation of a filtered nitrogen source into the flow bench used for filter holder assembly, disassembly, and filter membrane handling. A protocol for gaseous nitrogen purging of filter holder surfaces was developed and all personnel were trained and familiarized with the collection of particulate from the filtered nitrogen source using a filter holder, verifying they could collect a passing Level 50 particulate sample, and using this logic to purge a failed filter until it passed Level 50 validating it for reissuing.

After more about the problems and solutions were known and improvements were implemented, the first sample pass rate appeared to increase to previously baseline levels. This represented samples taken from around the site including the Propulsion areas, which included nitrogen and helium pressuring agent samples taken from propulsion ground support equipment systems. On April 4, 2024, an overnight even in the lab happened and a water leak from above the ceiling collapsed a ceiling tile onto the analytical balance. Images are provided in Figure 6. The ceiling tile not only temporarily disabled the balance, but the fiber material from the tile was foreign object debris (FOD) exceeding any reasonable amount acceptable for precision analytical work. This caused a temporary work stoppage as the flow blench for assembling and disassembling filter holders and handling had to be covered with a plastic tarp while scaffolding was erected, and facilities personnel accessed the roof area to find and repair the leak. The leaking component was identified as a hot water coil and since no replacement part was immediately available, the scaffolding was removed, and the lab was returned to service.

To ready the lab for precision particle counting, the entire lab was cleaned, the balance was restored, and both the flow benches were recertified. The filtered nitrogen supply enabled the lab to validate gas samples could be collected, and particulate counts could pass specification without contamination from ceiling tile and construction FOD and demonstrated the resilience of the lab with the new procedures despite the interruption.

Figure 6. Ceiling Tile Collapse Causing Temporary Disruption in Workflow
Note the flow bench to the right of the balance.

The next set of North High Bay Orion Main Engine Related Payload/Flight Hardware Processing (OMSE) Clean Room samples were taken and counted from May 13 to May 17, 2024. No anomalies were experienced. Thirty-three test ports supplying nitrogen or helium were sampled to Orion Level 50 or Orion Level 100. Thirty-one (31) or 94% of these passed specification on the first sample and the remaining 2 or 6% passed on the second sample. The two filter holders that failed were revalidated, rebuilt, and passed other test ports in this series. No filter holders were sent for precision cleaning to JPR 5322.1 Level 50A.

The laboratory process is one of confidence and continuous demonstration that quality control objectives can be achieved before assembled filter holders are issued. The laboratory can build a filter holder, sample it with filtered nitrogen, disassemble it, and count the membrane and pass it and not rely on customers bringing us sampled filter holds to be part of our quality control. The laboratory learned lessons are briefly described below and laboratory persons preparing, issuing, and receiving filter holders must be aware of these lessons.

- Open filter holders and filter membranes are always handled in a Class 100 flow bench.
- Successive particle count failures must be flagged, and the problems must be evaluated, and corrective measures taken before issuing further filter housings.
- A filtered nitrogen supply offers a quality control asset to evaluate filter holder and membrane suitability, to ensure personnel can successfully assemble, sample, disassemble, and pass a Level 50 particulate specification. Filtered nitrogen is also used to purge failed filter holders and to sweep filter holder surfaces free of particulate matter.
- An illuminated magnifier offers a quality control asset to evaluate filter holder surfaces including O-rings.
- Routine cleaning of filter holders to JPR 5322.1 Level 50A has been discontinued. Level 50A and other cleanliness levels for Millipore filter holders used for particulate sampling of gases were found to be tradition without basis and cleaning for routine sampling is only required to meet particulate level requirements and not NVR requirements. Requests for “A” level filter holders must be brought in advance to the attention of the laboratory to coordinate with the cleanroom.

- Previously unexperienced membrane filter failure due to delamination at the compression area of the inner filter holder O-ring showed the value of using the added quality control measures to evaluate built filter holders for sampling suitability. Batch-lot verification of membrane filters can also be performed using the filtered nitrogen supply to sample a filter holder fitted with a membrane filter from a new lot.
- A filter holder having failed particulate count can be swept clean with filtered nitrogen, rebuilt with a new filter membrane, sampled, and revalidated. Reusing a filter holder in such a manner avoids substantial cleaning costs.
- Filter holders used for sampling deionized water for particulate can also be dried and swept clean with filtered nitrogen and revalidated in the lab. Previously, filter holders used for sampling deionized water were submitted for precision cleaning to remove residual water and/or particulate regardless of whether they had passed or failed specification.

Protocol for Reuse and Revalidation of Filter Holders

The protocol for managing the filter holder of a membrane filter that failed specification was revised. When the count is complete, the results should be compared to the specification cleanliness level. If the filter holder was used for oxygen or an inert gas sample and passed specification, it may be considered clean and rebuilt with a membrane for reuse to the same particulate specification but not for any use requiring A-level cleaning. If the filter holder was used for oxygen or an inert gas sample and failed specification, it is rebuilt and reissued with a pre-counted membrane filter after being validated. Validation is accomplished by sweeping the internal surfaces of the filter holder with filtered nitrogen, rebuilding with a new membrane filter, sampling filtered nitrogen, allowing the filter holder to sit for 5-10 minutes to allow for equilibration, and disassembling and counting to Level 50. A filter holder that passing Level 50 is validated and may be reissued for particulate sampling without cleanroom cleaning. The general procedure is to examine filter holder interior areas including the O-ring perimeters with the illuminated magnifier and sweep with filtered nitrogen to remove particulate whether visible or not, then to install a filter membrane, connect the filter holder to the filtered nitrogen supply and sample to pass 1.0 m^3 (35.31 ft^3) of effluent nitrogen through the membrane filter. Immediately after sampling, the filter holder is recapped and allowed to sit for 5-10 minutes before disassembling and performing a particle count to JPR 5322.1 Level 50. If the particle count passes, the filter holder that passed Level 50 is validated and may be reissued for particulate sampling. If the particle count fails, the cause of the failure is investigated. If clear fibers are observed, the cause may be delamination of the filter membrane. If globules are observed, the cause may be shedding of the O-ring and using the illuminated magnifier to examine the O-ring and adjacent surfaces will reveal this. If metallic contamination is observed, the cause may be shedding of metallic particulate from inner surfaces. In any case of failure, the filter holder interior areas including the O-ring perimeters are examined with the illuminated magnifier and swept with filtered nitrogen to remove particulate whether visible or not. Measures are taken to correct the problem(s), then the revalidation steps are repeated until a passing result is obtained.

Previous Lessons Learned

Historical experience with gridded filters shedding ink that was counted as particulate and failing hydrogen particulate count specifications without sampling was considered. This occurred in the 1996-1997 timeframe. The gridded filters encountered during the hydrogen sampling experience not only shedding possibly by compression of the filter membrane on assembly but breaking up on impact with gas samples made for judgement calls whether particulate was ink or otherwise. In the ideal, judgement calls are not required because the variables have been reduced to where all particulate can be counted as particulate, and the use of gridded filters was discontinued.

Historical experience (740602, 740604) with contaminated Petri dishes used to hold pre-counted and sampled filter membranes was considered. In 2015, a series of Orion Level 50 particulate failures prompted an investigation. Technique, facility, flow benches, FOD control, clean workstation practices, filter housing cleanliness specifications, training, and other verifications were examined. The particulate matter common to the failing samples was characterized by optical microcopy and matched particulate matter found in the batch of Petri dishes being used to hold filter membranes after pre-counting and after disassembly of the filter holders. The Petri dishes being used were found to be contaminated with a material that was not visible to the naked eye or under ultraviolet (UV) light but was resulting in clear, amber/brown particulate deposited on the filter membranes and counted as particulate and failing the samples. Efforts were not made to chemically characterize the clear, amber/brown particulate. Cleaning and replacement options were examined, and the Petri dishes were able to be cleaned in the cleanroom by rinsing with HFE-7100, drying, and bagging to maintain clean. This solved the immediate problem and to the present Petri dishes are regarded as a source of FOD and are cleaned when necessary. Other changes made as a result of this investigation was to improve clean workstation principles and practices. All particulate shedding materials including fabric-covered chairs, absorbent mats under vacuum pumps, cardboard boxes, and other materials were removed from the laboratory. Tacky mats were installed inside the entrance and are changed daily at the beginning of the work shift. A tacky roller and UV light were obtained for periodic cleaning and wiping down the laboratory. Instituting "clean-as-you-go" as a routine practice. These practices continue through the present.

NASA Lesson Number 8018 (March 11, 2014) describes a related lesson submitted by Kennedy Space Center (KSC) for the Components Refurbishment and Chemical Analysis (CRCA) facility. Gas and liquid sample particle count analysis failures from the GSE community led to concerns regarding integrity of the CRCA facility sampling fixtures and/or laboratory techniques. This concern prompted an internal validation of sampling and cleaning processes of pre-cleaned sampling fixtures that were staged for use. Thirty-four of the unused sampling devices were inspected and seven (7) of the cleaned and staged fixtures had single particle particulate >100 µm. These findings of 20% sample failure rate supported the conclusion that contributory contamination was occurring in the sampling/cleaning process performed at CRCA. An Independent Investigation Team was stood-up on 01/29/2014 and initial team findings supported concerns that the cleanroom clean levels and maintenance were inadequate for the functions performed. CRCA Operations were suspended on Thursday, 02/06/2014 to re-establish the cleanroom baseline. Maintenance repairs were made to seal, adjust, and replace several defective items. The cleanroom underwent a thorough cleaning and mechanical systems were inspected, tested, and verified as meeting cleanroom requirements. Protocol procedures were reviewed and enhanced such as the processes for cleanroom entry and gowning and gloving. Refresher training was held with the entire staff. Tools and materials to perform the cleanroom work were reviewed and new items are being provided to both improve the efficiency and quality of the work performed. Cleanroom particle counts were taken at 60 locations to establish a baseline and ensure the room meets certification requirements. To help ensure cleanroom protocols are maintained, witness plates were placed at 10 locations throughout the room and were inspected frequently to determine whether any particle excursions are occurring. These witness plates were planned to be replaced with real-time particle monitoring stations equipped with local display and alarm capability to alert as contamination occurs. The CRCA cleanroom was reopened on 02/14/2014. The lesson(s) learned were that the particle contamination monitoring was insufficient to provide meaningful feedback to ensure operating protocols were successful in maintaining cleanliness levels.

CONCLUSIONS AND RECOMMENDATIONS

Lessons learned from assessing and recovering from a series of particulate count failures attributed to filter holders and filter membranes included a systematic evaluation of sources of contamination that contributed to particulate count failures. These included filter membranes, filter holders, Petri dishes, the particulate counting process, clean workstation principles and practices, assembly and disassembly practices and procedures, and use of filtered nitrogen to remove particulate from internal filter holder surfaces. Normalized procedures without documented requirements, including some possibly normalized deviances, for example 0-0-0 pre-counts rather than meets specification, sampling, and cleaning to 50A or any A-level where not explicitly required were identified, questioned, and changes were made. The capability to provide particulate count level assurance for filter holders by rebuilding after flowing filtered nitrogen and sampling to verify, rather than to depend on precision cleaning, was established. Implementation of quality control and validation capabilities and practices including the ability to validate lab readiness and individual's readiness to issue filter holders was accomplished. Recovery from the particulate count failures and improvements were accomplished.

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