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Superseding ARP743B

Procedure for the Determination of Particulate Contamination of Air
in Dust Controlled Spaces by the Manual Particle Count Method

RATIONALE

FED-STD-791 referred to in the document has been cancelled and superseded by ISO 14644-1 and ISO 14644-2. The procedure in ARP743 cannot be used to count the smaller particle sizes identified in the ISO documents. Accordingly, ARP743 cannot be used to demonstrate compliance with the ISO documents.

CANCELLATION NOTICE

This document has been declared "CANCELLED" as of May 2010. By this action, this document will remain listed in the Numerical Section of the Aerospace Standards Index.

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1. SCOPE:

1.1 This SAE Aerospace Recommended Practice (ARP) describes two procedures for sampling particles in dust controlled spaces. One procedure covers airborne dust above 5 μm . The other (and newly added procedure) covers particles of 25 μm and larger that "fall out" of the environment onto surfaces. In each case the particles are sized in the longest dimension and counted. Airborne particles are reported as particles per cubic meter (cubic foot) whereas particles collected in fall out samples are reported as particles per 0.1 square meter (square foot). This document includes English units in parentheses as referenced information to the SI units where meaningful.

These procedures may also be used for environmental analysis where the quality of the particles by visual or chemical analysis is intended.

1.2 Field of Application:

1.2.1 Airborne particle sampling (volumetric samples) - This procedure may be used in lieu of automatic particle counters for verifying conformance with FED-STD-209 for cleanroom classes above 10,000 unless the user or contracting agency specifies the counting of 0.5 μm particles. It may also be used for environmental analysis where the quality of the particles by visual identification is desirable.

1.2.2 This procedure can be used as a rough correlation check of particles greater than 5 μm when calibrating automatic particle counters.

1.3 Limitations and Errors:

1.3.1 This procedure is a slower alternative to the method by automatic particle counter, but necessary when the identification of particles is a concern.

1.3.2 This procedure is not appropriate when particles below 5 μm are of concern.

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- 1.3.3 The procedures herein are not recommended to evaluate the residues that may collect on residue sensitive instruments such as optics and sensors. These residues, sometimes called non-volatile residue (NVR) or volatile condensable material (VCM) are best sampled by other means.
- 1.3.4 One source of error in these procedures is a poor distribution of particles on the membrane filter surface (see 3.8.6) while another is from errors occurring during the microscopic analysis.

2. REFERENCES:

2.1 Applicable Documents:

- 2.1.1 SAE Publications: Available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096-0001.
- ARP598 Procedure for the Determination of Particulate Contamination of Hydraulic Fluids by the Particle Count Method
- 2.1.2 U.S. Government Publications: Available from DODSSP, Customer Services Desk, Building 4D, 700 Robbins Avenue, Philadelphia, PA 19111-5094. Fax requests to (215) 697-1462.
- FED-STD-209 Airborne Particulate Cleanliness Classes in Cleanrooms and Clean Zones

2.2 Definitions:

- 2.2.1 **CLEANROOM:** An enclosure or room built of and equipped with materials of low sloughing/dusting/linting potential. It is supplied with an over pressure of highly filtered air.
- The area is monitored with an automatic particle counter or the procedure herein to assure that the airborne particulate concentration is within prescribed limits. The most commonly used cleanroom requirements document is FED-STD-209.
- 2.2.2 **LAMINAR FLOW CLEANROOM:** A room, one wall or the ceiling of which, is a filter bank through which air flows laminarly across to the opposite wall or the floor which is an air return grating, or which is open to the uncontrolled environment ("tube" cleanroom).
- 2.2.3 **CONVENTIONAL FLOW CLEANROOM:** A fully enclosed room into which the air flows through a grating or gratings on one wall or around the room near the ceiling. The air exits through exhaust ducts near the floor.
- 2.2.4 **SEGMENTED SAMPLE TIME:** Sample time that is an accumulation of several sampling periods taken on the same membrane. Example a 24 h segmented sample may be an accumulation of three 8 h work shifts collected on one membrane.

3. TEST REQUIREMENTS:

3.1 Apparatus:

- 3.1.1 Aerosol Open Type Filter Holder, using 47 mm membrane filters, exposing $960 \text{ mm}^2 \pm 25 \text{ mm}^2$ of membrane surface to contamination (Note 1).
- 3.1.2 Air Flow Measuring Device (Note 2), accurate to $\pm 0.5 \text{ lpm}$ in the area of 10 lpm (0.35 cfm) at $50 \text{ torr} \pm 10 \text{ torr}$ below atmospheric pressure.
- 3.1.3 Membrane Filters, 0.45 to $1.2 \text{ }\mu\text{m}$ pore size, 47 mm diameter, having an imprinted grid on $3.10 \text{ mm} \pm 0.08 \text{ mm}$ centers. Each grid square is equal to approximately one hundredth of the total effective filtering area of the filter disc when used in the aerosol open-type filter holder (above). The membrane color shall be chosen to provide adequate contrast with the particulate contamination to be observed. The pressure drop through the membrane shall be $50 \text{ torr} \pm 10 \text{ torr}$ when sampling at 10 lpm (0.35 cfm).
- 3.1.4 Plastic Petri Dishes, 47 mm diameter, or other suitable device for mounting membrane filter specimen, capable of preventing contamination by atmospheric fallout during storage and examination.
- 3.1.5 Forceps with unserrated tips.
- 3.1.6 Rinse Dispenser (Note 3).
- 3.1.7 Vacuum Pump (Note 4), capable of moving 10 standard liters (0.35 standard cubic feet) of air per minute at observed operating pressure.
- 3.1.8 Particle Count Apparatus:
 - 3.1.8.1 Microscope, binocular or monocular (stereo microscopes shall not be employed with this procedure).
 - 3.1.8.2 Objectives and Oculars (eyepieces) in combinations to give magnifications of $50\text{X} \pm 10\text{X}$ and $100\text{X} \pm 10\text{X}$. The higher power objective shall have a minimum numerical aperture (N.A.) of 0.15 . The ocular shall not be greater than 15X .
 - 3.1.8.3 Ocular Micrometer, linear scale installed in one eyepiece. The smallest division shall not subtend a distance larger than the smallest particle to be counted at a particular magnification. A 10 mm scale with 200 equal divisions is known to be suitable when 10X and 4X objectives are used.
 - 3.1.8.4 Mechanical Stage, capable of traversing the entire area of the membrane filter. It shall have provision for holding a mounted membrane filter.
 - 3.1.8.5 Stage Micrometer, divisions of 0.1 mm and 0.01 mm .

3.1.8.6 Microscope Light, external, focusing. It shall be equipped with an external adjustable arm to give oblique incident light. It shall be capable of providing an illumination of 54,000 to 65,000 lux (5000 to 6000 ft-candles) at the counting surface.

3.2 Reagent:

Government regulations have eliminated the use of numerous rinsing agents used in the dispenser (Note 3) The user shall determine an appropriate material for this application. The material must be capable of filtration by a 1.2 μm , or finer, membrane filter and shall be compatible with the filter material. Hexane is one recommended reagent.

3.3 Samples:

3.3.1 Vacuum Assist Sample: The standard sample for the vacuum assist method shall be $0.28 \text{ m}^3 \pm 0.014 \text{ m}^3$ ($10 \text{ ft}^3 \pm 0.5 \text{ ft}^3$) air at ambient temperature and pressure, drawn at the rate of 10 lpm (0.35 cfm) for 28 min. For very clean areas, larger samples may be required. For determining conditions to which sensitive units are exposed, it is essential that the sample be taken in the immediate proximity of the sensitive component or system while it is being subjected to normal assembly or test procedures. Normally one sample should be taken for each 25 m^2 (250 ft^2) of floor space in the controlled environment area.

3.3.2 Fallout Sample: The standard sample is either 24 segmented hours or 48 continuous hours. Particle population within a room is usually not homogeneously distributed. It is, therefore, recommended that multiple samples be taken at key positions in the room.

3.4 Test Information:

3.4.1 Personnel performing loading, background count, or unloading after sampling shall be trained in contamination control technique and attired in clothing consistent with the cleanliness requirements of the atmosphere being sampled.

3.4.2 Samples are to be obtained in accordance with specified sampling procedures.

3.4.3 The aerosol holder for airborne particle counts is to be cleaned just prior to use by flushing with rinse solvent from the rinse dispenser.

3.4.4 Membrane filter mounts are to be cleaned prior to use by flushing with a compatible rinse solvent from the rinse dispenser.

3.4.5 Hazardous materials employed in this method shall be handled with suitable precautions.

3.4.6 All cleaning efforts shall achieve a contamination level consistent with the standard of the air particle sample or fallout sample being taken.

3.4.7 All related sampling and evaluation apparatus, or both, shall be maintained at a cleanliness level consistent with the requirements of the area being sampled.

3.5 Airborne Particle Sampling Procedure:

3.5.1 Blank Analysis:

- 3.5.1.1 Using clean forceps, remove one filter membrane from its container. Rinse it with solvent from the filtering dispenser, allow to dry, and place in membrane filter mount.
- 3.5.1.2 Perform a blank particle count in accordance with 3.8.8. The count shall not exceed 5% of the required maximum count for the area to be sampled.

3.5.2 Procedure for Samples - Airborne:

- 3.5.2.1 Using clean forceps, carefully transfer a precounted filter membrane from its mount and place it grid side up on the support of the aerosol holder. Secure in place with the inlet portion of the filter holder.
- 3.5.2.2 Locate the holder in accordance with the provisions of 3.3.1. Face inlet portion towards direction of principal air flow in laminar flow clean rooms. In conventional clean rooms the membrane filter should be in the vertical plane. Connect aerosol holder to flow measuring device and flow measuring device to vacuum source, using suitable vacuum tubing.
- 3.5.2.3 Apply vacuum and adjust flow to 10 lpm (0.35 cfm).
- 3.5.2.4 At the end of the 28 min sampling period, release vacuum. Remove aerosol holder inlet portion, and, using forceps, carefully remove the membrane filter from the aerosol holder and transfer it to the membrane filter mount. Close the mount to protect the filter membrane from further contamination. Identify sample.
- 3.5.2.5 Exercise care in handling the mounted filter prior to counting in order to avoid disturbing the distribution of the particles.

3.6 Fallout Particle Sampling Procedure:

- 3.6.1 Blank Analysis: Blank analysis samples are not necessary for fallout samples due to the large relative size of the target particles.

3.6.2 Procedure for Samples - Fallout:

- 3.6.2.1 Transfer a clean 47 mm filter membrane, gridded side up to a 47 mm petri dish.
- 3.6.2.2 Place the opened dish in the sample location. Sample locations can be at any spot from where data is desired but it is inadvisable to place it where it will be knocked over or off, say, a workbench.
- 3.6.2.3 Keep the petri dish cover in a place where it will not become contaminated while the sample is exposed.

3.6.2.4 Leave the dish out for 48 h of continuous exposure or 24 h of segmented exposure. Segmented exposure is where the membrane is exposed, then covered, then exposed again depending on when the activity of interest is going on. When the cumulative time reaches 24 h, the standard sample time is completed (Note 5).

3.6.2.5 Cover the completed sample and deliver the petri dish to the laboratory for analysis.

3.7 Microscopic Calibration:

3.7.1 Place the stage micrometer on the mechanical stage and adjust the light.

3.7.2 Place the required objective and oculars to obtain $100X \pm 10X$ magnification in the microscope and focus on the micrometer.

3.7.3 Calibrate the ocular micrometer located in one eyepiece (Note 6). Each operator shall perform this calibration when using a binocular microscope (Note 7). The calibration method requires that the length of the entire scale be measured rather than only a portion.

3.8 Particle Counting Procedure:

While certain details of the counting procedure depend somewhat upon the specific equipment used, the procedure specified herein must be followed exactly as stated to provide the required reproducibility (reference ARP598).

3.8.1 The particle size shall be determined by measuring the greatest dimensions visible under the microscope.

3.8.2 Place the membrane filter mount on the microscope stage. Secure it in the jaws of the mechanical stage.

3.8.3 Using a $50X \pm 10X$ magnification for fallout samples or a $100X \pm 10X$ magnification for aerosol samples, focus on the grid lines.

3.8.4 Turn the mount until the grid lines are aligned with the vertical and horizontal stage axes.

3.8.5 Focus the light; adjust angle (Note 8) and intensity to obtain maximum particle definition.

3.8.6 Examine the membrane by scanning the surface to determine that the particles have a random distribution. If the filter membrane shows evidence of nonrandom distribution, the statistical counting procedure shall not be used (random distribution should not be confused with uniform distribution). A new sample should be drawn or a total particle count performed.

3.8.7 For aerosol samples, count all particles $>5.0 \mu\text{m}$. For fallout samples, count all particles $>25 \mu\text{m}$.

3.8.8 Statistical Counting Procedure for Aerosol Membranes:

3.8.8.1 Count the number of particles on each field (Note 9) using $100X \pm 10X$ magnification by “gating” (Note 10) the membrane filtration area. As the particles pass by the ocular micrometer, measure (Note 11) and record the number of particles.

3.8.8.1.1 If the total number of particles in a size range is 100 or less, the entire surface of the membrane shall be examined and all particles in this size range counted.

3.8.8.1.2 If the number of particles in a specific size range is greater than 100, the following requirement shall be met:

$$F_n \times P_t = 500 \text{ or greater} \quad (\text{Eq. 1})$$

where:

F_n = Number of fields counted (minimum of 10)

P_t = Total number of particles counted in all the fields

3.8.8.2 If a particle lies on the upper or left boundary line of a counting area, count this particle as if it were within the boundaries of the counting area. Particles on the lower and right hand boundary lines of the counting area shall not be counted.

4. TEST RESULTS:

4.1 Particle Count Calculations:

4.1.1 The total particle count for each range shall be calculated using the following formula:

$$\text{Total Particle Count} = \frac{P_t \times A}{F_n \times F_a} \quad (\text{Eq. 2})$$

where:

P_t = Number of particles counted in F_n fields

A = Filtration area of membrane (normally 960 mm^2)

F_n = Number of fields counted

F_a = Area of each field in square millimeters