

PROCEDURE FOR THE DETERMINATION OF PARTICULATE CONTAMINATION
OF AIR IN DUST CONTROLLED SPACES BY THE PARTICLE COUNT METHOD

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Revised

1. SCOPE:

This test describes a self-checking procedure for the determination of particulate contaminant five microns or greater in size in air by the particle count method. A maximum variation of two to one (+33% of the average of two runs) in results should be expected for replicate counts on the same sample, providing that the procedure is followed closely and the precautions presented regarding check samples and self-checking aspects are observed.

2. OUTLINE OF METHOD:

Air from the controlled area is filtered through a membrane filter disc using vacuum to impinge the entrained contamination particles upon the surface of the filter. The filter disc is examined microscopically (using oblique incident lighting) to determine the amount of contaminant present in stated size ranges.

3. APPARATUS:

Aerosol Open-type Filter Holder, using a 47 mm filter.

Air Flow Metering device accurate in the range of 10 lpm (liters per minute).

Membrane filters, 0.45 to 1.0 micron pore size, 47 mm diameter, having an imprinted grid on 3.08 mm centers. Each grid square is equal to 1/100th of the total effective filtering area of the filter disc when used in the Aerosol Open-type Filter Holder (above).

Plastic Petri Dishes, 47 mm diameter.

Forceps with unserrated tips.

Vacuum Pump - Capable of 20-25" Hg.

Microscope with mechanical stage, capable of magnification of approximately 45 X and 90 X. For 90 X magnification, the recommended objective is 10 to 12 X but at least 6 X with a numerical aperture of at least 0.15. The optimum equipment is a binocular microscope with a micrometer stage. A Stereo microscope should not be employed with this procedure.

Measuring eyepiece - Ocular Micrometer - Baush & Lomb Cat. #31-16-01, or equivalent. (See illustration following 5.4.3).

Stage Micrometer, 0.1 to 0.01 mm calibrations.

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Microscope Lamp, high intensity, variable. This lamp is to be used as a source of oblique incident light; Leitz microscope lamp "MONLA"*, or equivalent; 5,000 - 6,000 candlepower at filter surface.

*"MONLA" - 6V - 5A microscope lamp with focusing illuminating lens on pillar stand with separate transformer.

4. SAMPLES:

- 4.1 A 28 minute sample at 10 liters/min (10 cubic feet) is to be used in this procedure.
- 4.2 Sampling Procedure: Samples for this test method should be as representative as possible of the area being sampled. Procedures for procuring such samples will, of necessity, have to be established by individual plants or laboratories. To assure reproducibility, the sampling program should be checked at the outset by the testing of replicate samples from the sampling area.

5. TEST PROCEDURE:

5.1 Test Information:

- 5.1.1 Personnel performing contamination analyses should wear lint-free laboratory coats, bonnets and shoe covers.
- 5.1.2 Samples are to be obtained in accordance with specified sampling procedures.
- 5.1.3 The filtration apparatus is to be cleaned just prior to use by acceptable cleaning procedures to remove random dust on the stainless inlet bushing of the filter holder.
- 5.1.4 The microscope and its accessories should be maintained in a state of maximum cleanliness. The microscope and accessories should be protected by a dust cover when not in use.
- 5.1.5 The processing and microscopic analysis of samples should be performed in as clean an area as possible within the confines of a modern, air-conditioned laboratory. A dust control room¹ is desirable, but not essential for validity and reproducibility. Smoking should be prohibited, both as a safety factor and to prevent the extra contamination of samples. The ingress and egress of personnel in the laboratory area should be limited.

5.2 Filtration Procedures:

- 5.2.1 Procedure for Blanks: Prior to each sample analysis, a blank analysis is to be performed on an unused filter as removed from the manufacturer's container. Identify the blank as to the sample number on an identification tag and attach it to the lid of the petri dish.²

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5.2.2 Procedure for Samples:

- 5.2.2.1 Using clean forceps, remove one filter disc from its container. Place the filter disc - printed grid side up - on the screen support of the filter holder.
- 5.2.2.2 Immediately lower and lock the inlet bushing and cover onto the base of the filter holder.
- 5.2.2.3 Connect the flow metering device into the vacuum line to the filter holder downstream of the membrane.
- 5.2.2.4 Apply vacuum to the filtering apparatus to sustain a flow of 10 lpm. At the end of a 28 minute sampling period, release the vacuum.
- 5.2.2.5 Using forceps, carefully remove the filter disc from the top of the filter holder support. Place the filter disc - grid side up - in a clean petri dish and replace petri dish cover.

Identify the petri dish using a sample identification tag. The test may be delayed overnight, if necessary, after completing this step.

5.3 Microscope Analysis Procedure: Particles are to be counted and tabulated in the following order: fibers, particles greater than 25 microns, and 5 - 25 microns. Particles smaller than 5 microns are not to be counted by this method. Fibers are defined as any particle whose length to diameter ratio exceeds 10 to 1 regardless of composition. Fibers are counted as particles and not differentiated unless length exceeds 100 microns. The size of a particle is determined by its greatest dimension. (See paragraph 5.3.4.8).

- 5.3.1 Place petri dish under the microscope dust cover and remove petri dish cover.
- 5.3.2 Adjust the microscope lamp intensity to obtain maximum particle definition.
- 5.3.3 A magnification of approximately 45 X shall be used for counting particles 25 microns or larger; approximately 90 X for particles smaller than 25 microns. The recommended objective to obtain the 90 X magnification is 10 to 12 X power in conjunction with the appropriate eyepiece.
 - 5.3.3.1 Using a stage micrometer, calibrate the measuring eyepiece (ocular micrometer) for each magnification.
- 5.3.4 Method of Counting Particles: Other statistical methods may be employed provided that the method shows agreement with the values of the certified standard samples as described in Section 6. The following described method is recommended:

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- 5.3.4.1 In obtaining the number of particles of a given particle size range, the number of particles on a representative number of grid squares on the filter disc are counted. From this count, the total number of particles, which would be present statistically on the total effective filtration area of 100 imprinted grid squares, is calculated.
- 5.3.4.2 If the total number of particles of a given particle size range is estimated to be between 1 and 50, count the number of particles over the entire effective filtering area.
- 5.3.4.3 If the total number of particles of a given particle size range is estimated to be between 50 and 1,000, count the number of particles in 20 randomly-chosen grid squares and multiply this number by 5 to obtain the total statistical particle count.
- 5.3.4.4 If the total number of particles of a given particle size range is estimated to be between 1,000 and 5,000, count the number of particles on 10 randomly-chosen grid squares and multiply this number by 10 to obtain the total statistical particle count.
- 5.3.4.5 If the estimated total number of particles of a given size range exceeds 5,000, count the particles within at least ten (10) randomly-chosen unit areas.³ To arrive at the total statistical count, the sum of the particles counted in the areas is multiplied by the calibration factor.⁴
- 5.3.4.6 Select unit areas so that there will be no more than about 50 particles of a size range in a unit area. See Figure 1 for the alternate unit areas.
- 5.3.4.7 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.
- 5.3.4.8 The largest dimension of the particle determines the size category into which the particle is placed.
- 5.3.4.9 Results shall be divided by ten (10) and reported in each size range as particles/cu.ft.

5.4 Calculation of Calibration Factor:

- 5.4.1 The calibration factor is the ratio of the effective filtration area (100 grid squares or 9.6 cm²) to the area counted.
- 5.4.2 To arrive at a calibration factor, start with the microscope adjusted for the power under consideration.

5.4.3 Using the stage micrometer, measure the length of the ocular micrometer scale which is used to define the width of the unit area. The length of the unit area is defined by the size of the grid square or 3.08 mm.

Figure 1

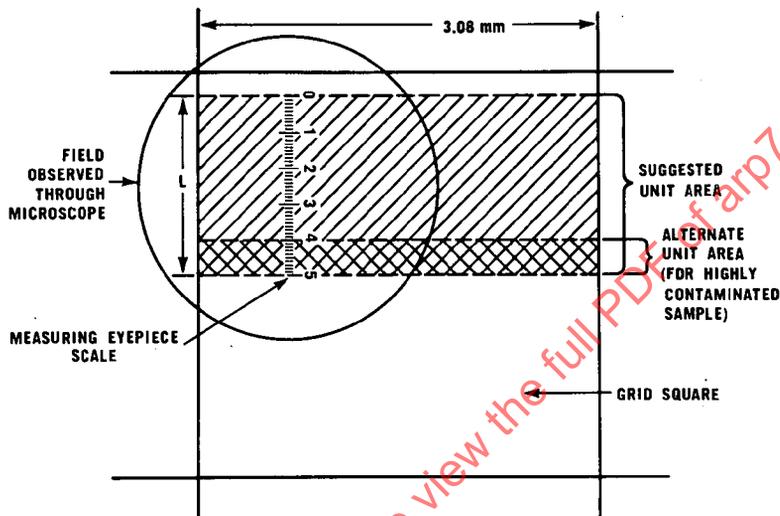


Figure 1 shows two possible unit areas within a grid square for statistical counting. The calibration factor is defined as the effective filter area divided by the total area counted and may be calculated from the following formula:

$$F = \frac{960}{3.08 \times L \times N} \quad \text{or} \quad \frac{312}{L \times x \times N}$$

where:

- F = the calibration factor
- N = the number of basic unit areas counted
- L = the calibrated length of the ocular micrometer scale or portion of scale used in defining unit area - in millimeters
- 3.08 = the side of a grid square in millimeters

for example: Using a 10 X objective and a 5 mm ocular micrometer, counting 10 basic unit areas, the calibration factor would be

$$F = \frac{312}{0.5 \times 10} = 62.4$$