Society of Automotive Engineers, Inc.
TWO PENNSYLVANIA PLAZA, NEW YORK, N.Y. 10001

AEROSPACE RECOMMENDED PRACTICE

ARP 598A

Issued 3-1-60
Revised 8-1-69

THE DETERMINATION OF PARTICULATE CONTAMINATION IN LIQUIDS BY THE PARTICLE COUNT METHOD

1. SCOPE

This method describes a procedure for the sizing and counting of particulate contamination in liquid samples by membrane filtration. The procedure will allow measurement of particulate contamination five microns or greater in size with a maximum variation of ±20% in results over an average of two runs. This procedure can be used for all samples where the membrane filter is compatible with the sample liquid and rinse liquid.

2. OUTLINE OF METHOD

A known volume of liquid is filtered through a membrane filter. The particulate contamination is deposited on the surface of the membrane filter. The residual contamination is then sized and counted by microscopic analysis.

3. MATERIALS

3.1 Membrane filter, pore size less than 1.0 microns. The filter shall have an imprinted grid on 3.10 ± 0.02 mm centers. The color shall be chosen for maximum contrast with the particulate contamination to be observed.

3.2 Petri dishes, plastic or glass.

3.3 Glass bottles, small mouth, screw capped, permanently marked to indicate sample volume. (1)

3.4 Plastic film, 0.002 in. minimum thickness. (2)

4. APPARATUS

4.1 Filtration Apparatus

4.1.1 Funnel, filtration - The lower hole shall have a diameter of approximately 35.0 mm ID. The effective filtering area shall be calibrated by filtering a contrasting particulate pigment through a membrane filter. The diameter of the residual pigment shall be measured at quadrature diameters. If the receiver is to be used for measuring the sample volume, the funnel shall be calibrated within ± 2% of the required volume.

4.1.2 Membrane filter support - Either a fritted glass, sintered metal or stainless steel screen may be used. The support shall be so designed as to enable attachment to a vacuum source.

4.1.3 Vacuum flask

4.1.4 Funnel holding device - A provision should be made for the dissipation of static electricity from the funnel.

Note: (1) The standard sample volume shall be 100 ± 5ml to allow complete use of fluid and to allow the bottle to be rinsed with solvent.

(2) Plastic film must be compatible with sample and rinse liquids.

4.1.5 Funnel cover - to prevent extraneous contamination.

4.1.6 Vacuum source - minimum vacuum of 18 inches of mercury.

4.1.7 Forceps - unserrated tips

4.1.8 Rinse dispenser (3)

4.2 Particle Count Apparatus

4.2.1 Microscope - Binocular or monocular (Stereo microscopes shall not be employed with this procedure).

4.2.2 Objectives and oculars (eyepieces) in combinations to give magnifications of 50 ± 10x and 100 ± 10x. The higher power objective shall have a minimum Numerical Aperture (N. A.) of 0.15. The ocular shall not be greater than 15x.

4.2.3 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.

4.2.4 Mechanical Stage - capable of traversing the entire area of the membrane filter. It shall have provision for holding a membrane container.

4.2.5 Stage micrometer - divisions of 0.1mm and 0.01mm.

4.2.6 Microscope light - external, focusing - It shall be equipped with an external adjustable arm to give oblique incident light. It shall provide an illumination of 5000 to 6000 ft-c at the counting surface.

5. REAGENTS

5.1 Liquid detergent solution which leaves no solid residue.

5.2 Isopropyl alcohol, acetone free.

5.3 Petroleum naptha - minimum initial boiling point of 85 F and a maximum end point of 220 F. Halogenated hydrocarbons may be used where petroleum naptha presents a safety hazard, providing the substitute solvent is compatible with the membrane filter and adequate precautions are taken to avoid inhalation of the vapors.

6. FILTRATION OF REAGENTS

The filtration of reagents shall be performed with the apparatus described in paragraph 4.1.8.

7. PREPARATION OF APPARATUS

The apparatus used in the filtration of samples shall be prepared as follows:

7.1 The apparatus shall be thoroughly washed in a solution of liquid detergent and hot water.

7.2 Rinse with hot distilled or de-mineralized water.

7.3 Rinse with 1 micron filter isopropyl alcohol to remove water.

7.4 Rinse with 1 micron filtered petroleum naptha.

Note: (3) A pressurized container equipped to pass rinse liquid through membrane filter having a pore size of 1.2 microns or finer.

7.5 Sample Bottle Preparation - Repeat paragraph 7.1 through 7.4, then allow the bottles to drip dry. Place a piece of plastic film, which has been rinsed with filtered liquid, over the mouth of the bottle. Hold the film while screwing on the cap to prevent the film from rotating (4).

8. LIQUID SAMPLES

8.1 The standard sample should be 100 ± 5ml except in the following cases: When the particle count from this volume is greater than 100,000 or less than 500 particles total, the sample volume may be altered. For counts less than 500 particles, the volume should be a minimum of 200 ml. For counts greater than 100,000 particles, the volume may be decreased to allow proper particle differentiation. In all cases, the sample volume shall be recorded.

8.2 Sampling Procedure - Samples shall be as representative of the system as possible. Procedures for sampling shall be established by individual plants or laboratories. To assure reproducibility, the sampling program should be checked by testing replicate samples from the sample port.

9. SAMPLING PREPARATION PROCEDURE

9.1 Blank Analysis Filtration

9.1.1 Remove a membrane filter using forceps from the container and rinse the filtered liquid.

9.1.2 Place the filter on the support, lower the funnel, and secure with the holding device. Cover the funnel.

9.1.3 Place an amount of membrane filtered liquid from the rinse dispenser, equivalent in volume to the fluid to be tested, in a bottle (5) and agitate.

9.1.4 Remove the cover and pour the contents of the bottle into the funnel.

9.1.5 Pour approximately 50 ml of rinse liquid into the bottle and agitate.

9.1.6 Pour the contents of the bottle into the funnel and replace the funnel cover.

9.1.7 Turn on the vacuum and allow the sample to filter until approximately 50 ml remain.

9.1.8 Remove the cover, rinse the funnel walls and replace the cover (6).

9.1.9 Allow the sample to filter until dry.

9.1.10 Remove the cover, holding device and immediately turn off the vacuum.

9.1.11 Remove the membrane filter using the forceps and place in a petri dish and label.

9.1.12 Perform a particle count as specified in paragraph 11.

9.1.13 The blank count shall not exceed 10% of total allowable sample count. If the blank analysis exceeds 10% of the count of an acceptable sample, or one particle, whichever is greater, and the total count exceeds the count of an acceptable sample, the apparatus shall be recleaned to produce a lower blank and the procedure should be rerun.

Note: (4) It is important to hold the film when applying and removing to prevent serration.

(5) This procedure is designed to utilize glass bottles. If other containers are to be used, this procedure should be deviated and proper identification given to the containers used.

(6) When the fluid filtration rate is excessive, causing a vortex, the vacuum should be released to allow adequate rinsing of the funnel walls and to eliminate the possibility of upsetting the particle distribution by the rinse liquid.

9.2 Sample Filtration

9.2.1 Repeat steps 9.1.1 through 9.1.2.

9.2.2 Thoroughly agitate the sample and then remove the cap.

9.2.3 Remove the funnel cover and pour the contents into the funnel.

9.2.4 Pour approximately 50 ml of rinse liquid in the bottle and agitate.

9.2.5 Pour the rinse liquid into the funnel and cover.

9.2.6 Turn on the vacuum and allow the sample to filter until approximately 50 ml remain.

9.2.7 Lift the cover and carefully wash down the funnel walls with rinse liquid (6).

9.2.8 Replace the cover and filter until the membrane is dry.

9.2.9 Remove the cover, clamp and funnel and then release the vacuum immediately.

9.2.10 Using the forceps, transfer the membrane filter to the petri dish.

9.2.11 Label the petri dish giving the sample volume and identification.

9.2.12 The filter is now ready for microscopic examination.

10. MICROSCOPIC CALIBRATION

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

10.2 Place the required objective and oculars in the microscope and focus on the micrometer.

10.3 Calibrate the ocular micrometer located in one eyepiece (7) at each magnification to be used. Each operator shall perform this calibration when using a binocular microscope (8). The calibration method requires that the length of the entire linear scale be measured rather than only a portion.

10.4 The operator shall calculate the number of linear divisions required to measure each range at all magnifications. For example: If an ocular micrometer with 100 divisions measures 250 microns at 50x, then each division would equal 2.5 microns. By calculating the ranges you would measure as follows: over 100 microns equals 40 division, 50 to 100 microns equals 20 to 40 divisions, 25 to 50 microns equals 10 to 20 divisions.

11. 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 accuracy and reproducibility.

Note: (6) When the fluid filtration rate is excessive, causing a vortex, the vacuum should be released to allow adequate rinsing of the funnel walls and to eliminate the possibility of upsetting the particle distribution by the rinse liquid.

(7) Do not place the eyepiece containing the ocular micrometer in an adjustable draw tube eyepiece because the calibration will change as the ocular is adjusted.

(8) When a binocular microscope is used, the focal length and calibration will change when the interpupillary distance changes.

11.1 The particle size shall be determined by measuring the largest dimension.

11.2 The size ranges shall be as follows: 5 to 15 microns, 15 to 25 microns, 25 to 50 microns, 50 to 100 microns, over 100 microns and fibers (9).

11.3 Remove the container lid and place the sample on the mechanical stage.

11.4 Select the required magnification and focus on the membrane grid lines.

11.5 Turn the petri dish until the grid lines are aligned with the vertical and horizontal stage axis.

11.6 Focus the light, adjust the angle (10) and intensity to obtain maximum particle definition.

11.7 Examine the membrane by scanning the surface to determine that the particles have a random distribution. If the membrane shows evidence of spotty distribution or rings of heavier particle concentration around the outside edge of the filtration area, the statistical counting procedure shall not be used. The liquid sample should be rerun or a total particle count performed.

11.8 This procedure defines a method of sizing and counting particles 5.0 microns and greater.

11.8.1 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.

11.8.2 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.

11.8.3 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.

11.8.4 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.

11.8.5 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. To arrive at the total statistical count, the sum of the particles counted in 10 or more unit areas is multiplied by the calibration factor.

Note: (9) A fiber is defined as a particle greater than 100 microns whose length exceeds the width by at least 10 times.

(10) The recommended angle is 15 - 45 deg from the horizontal.

11.8.6 Count the number of particles in each field (11) for each size range by "Gating" (12) the membrane filtration area. As the particles pass by the ocular micrometer measure (13) and record the number of particles in each size range. (14).

11.8.7 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. Particles on the lower and right-hand boundary lines of the counting area shall not be counted.

12. PARTICLE COUNT CALCULATIONS

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

$$\text{Total count} = \frac{A}{F_n \times F_a} \times P_t$$

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

F_n = Number of fields (unit areas) counted.

F_a = Area of each field (unit area) mm^2

P_t = Number of particles in F_n fields or unit areas.

12.2 Particle counts shall be expressed in particles per 100 milliliters. If the volume is other than 100 ml, the results shall specify the sample volume.

13. COUNTING PROFICIENCY

13.1 Check Samples - Membrane filters containing contamination, representative of the system to be counted, should be permanently mounted between glass. These samples should be counted by all operators to determine the mean particle count. This sample may then be sent to outside laboratories for additional counting. Care must be taken with these slides because the particle count may change as a result of handling.

13.2 Competence - Individual laboratories, with experienced counters, should be able to count with a 10% mean deviation thus proving operator and optical accuracies.

14. DISPUTE CLAUSE

In case of dispute, this clause shall be used to test laboratory competence in filtering and counting clean fluids.

Note: (11) Select a field size so that there are no more than about 50 particles of the size to be counted in the field. Optional fields are: a grid square; a rectangle defined by the width of a grid square and the calibrated length of the ocular micrometer scale; a rectangle defined by the width of the grid square and a portion of the length of the ocular micrometer scale as shown in Figure 1.

(12) Gating is the technique where one starts at a reference point and traverses the entire filtration area in a systematic manner.

(13) For particles improperly oriented relative to the ocular micrometer, an estimate shall be made. The eyepiece containing the ocular micrometer should not be rotated to size specific particles.

(14) More than one size range can be counted simultaneously providing the magnification is the same.