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AEROSPACE RECOMMENDED PRACTICE

ARP 598

PROCEDURE FOR THE DETERMINATION OF PARTICULATE CONTAMINATION OF HYDRAULIC FLUIDS 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 hydraulic fluids 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 on pages 10 and 11 of the procedure, regarding manipulation, check samples and self-checking aspects, are observed.

2. OUTLINE OF METHOD:

A fluid is filtered through a type HA Millipore filter disc using vacuum to impinge the contained 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:

Pyrex filter holder, Millipore Cat. #XX 10047 00, or equivalent, which includes:

A fritted glass base and rubber stopper.
A holding clamp.
A 250 ml. Pyrex glass funnel.

A filter cover for the glass funnel to minimize contamination from the air passing through the funnel during the vacuum filtration process. The optimum cover would incorporate a Millipore filter in the cover device.

0.45-micron Membrane Filter, type HA Black Grid 047 mm diameter Millipore, or equivalent. These to have 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 Millipore Pyrex filter holder (above).

Vacuum flasks.

Aspirator or Vacuum Pump, capable of pulling a minimum 26" of mercury.

Plastic Petri Dishes, disposable, Millipore Cat. #PD 10 047 00, or equivalent.

Forceps with unserrated tips.

Sample Bottles, small mouth, glass, etched or otherwise permanently marked to indicate 100 ml. sample size.

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 Catalog #31-16-01, or equivalent. (See illustration following 8.4.3.)

Stage Micrometer, 0.1 to 0.01 mm calibrations.

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.

Wash bottles, Pyrex glass.

Mylar plastic films, 2" x 2" x .002".

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

4. REAGENTS:

Distilled water.

Acetone free, reagent grade isopropyl alcohol.

Petroleum ether 30°-60° boiling range (Freon TF or equivalent may be substituted where explosive vapors are not accepted.)

5. FILTRATION OF REAGENTS:

5.1 Filtration Process: (for initial establishment of clean conditions for filtration of reagents.)

5.1.1 Clean all apparatus as follows:

5.1.1.1 Wash with self-rinsing type detergent and water.

5.1.1.2 Thoroughly rinse with hot soft tap water.[2]

5.1.2 Assemble Pyrex filter holder with filter disc in place.

5.1.3 Filter 100-200 ml. of isopropyl alcohol into the filter flask. Remove entire Pyrex Filter assembly and rinse the filter flask with the filtrate.

5.1.4 Repeat step 5.1.3 three times.

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[2]When filtered distilled water, filtered isopropyl alcohol, and filtered petroleum ether have been obtained, the apparatus should be cleaned as outlined in Para. 6.

5.1.5 Filter a 100-200 ml. volume of petroleum ether into the filter flask. Remove the funnel assembly and rinse the filter flask with the filtrate.

5.1.6 Repeat Step 5.1.5 three times.

5.1.7 Filter the desired volume of the solvent.

5.2 Control Analysis for Reagent Cleanliness:

5.2.1 Clean a Pyrex filter holder and a wash bottle using the process outlined in Paragraph 6.

5.2.2 Place 500 ml. of the filtered reagent in the cleaned wash bottle.

5.2.3 Rinse the upper surface of a filter disc with the filtered reagent from the wash bottle. Place the disc on the fritted glass base and complete the assembly of the filtration apparatus.

5.2.4 Pour a 150 ml. portion of the filtered reagent into the funnel directly from the filter flask. Filter the sample using the method for processing a blank described in Para. 8.2.1, except that the filter disc is to be rinsed only once and with a 50 ml. volume of the filtered reagent from the wash bottle. If the desired cleanliness level is not obtained,[3] repeat the cleaning and filtration process using the filtered reagents on hand for the cleaning process.

6. CLEANING METHOD FOR APPARATUS AND SAMPLE BOTTLES:

6.1 General Instructions: Lint-free coats should be worn by personnel performing cleaning operations to preclude excessive fiber contamination.

6.2 Cleaning Methods:

6.2.1 Each item of filtration apparatus will be cleaned before each run of samples and each sample bottle and cap will be cleaned before each use by the following method:

6.2.1.1 Rinse with two successive rinses of petroleum ether.

6.2.1.2 Wash thoroughly in a solution of detergent and hot water. Rinse twice in hot tap water (soft).

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[3]Contamination content of a 150 ml. sample of filtered reagent, so processed, should be no greater than the values specified for a blank analysis. See Footnote 6, Page 5 for blank analysis specifications.

6.2.1.3 Rinse with filtered distilled water twice.

6.2.1.4 Rinse with filtered isopropyl alcohol to remove water.

6.2.1.5 Rinse with filtered petroleum ether.[4]

6.2.1.5.1 Filtration Apparatus: After rinsing with petroleum ether, hold in an inverted position for 15 seconds to allow drainage and evaporation of the petroleum ether.

6.2.1.5.2 Sample Bottles: After rinsing with petroleum ether, allow a small quantity of petroleum ether to remain in the bottle since the vapor pressure will help preclude contamination when the bottle is opened. Rinse a previously cleaned 2" x 2" plastic film with filtered petroleum ether. Place the plastic film over the top of the bottle and install the bottle cap.

7. SAMPLES:

7.1 A 100 ± 5 ml. sample is to be used for this procedure.

7.2 Sampling Procedure: Samples for this test method should be as representative as possible of the fluid being sampled. Procedures for procuring such samples will, of necessity, have to be established by individual plants or laboratories. Extreme care should be taken to preclude the introduction of external contamination at this point. To assure reproducibility, the sampling program should be checked at the outset by the testing of replicate samples from the sampling port.

8. TEST PROCEDURE:

8.1 Test Information:

8.1.1 Personnel performing contamination analyses should wear lint-free laboratory coats.

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

8.1.3 The filtration apparatus is to be cleaned just prior to use by the method outlined in paragraph 6.

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[4] When high humidity conditions exist, Step 6.2.1.5 may be followed by an additional isopropyl alcohol rinse to prevent condensation.

8.1.4 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.

8.1.5 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 room[5] 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.

8.2 Filtration Procedures:

8.2.1 Procedure for Blanks: Prior to each sample analysis, a blank analysis is to be performed on 50 ml. of filtered petroleum ether contained in a regular, clean sample bottle. The procedure used will be identical with that described below, with the exception that Step 8.2.2.6 will be omitted. (The total amount of petroleum ether used in the blank analysis should be 200 ml., which is approximately the volume used in the filtration of a sample.) The blank analysis is performed to determine the amount of contamination being introduced by the sample bottle, filtering process, filtering equipment, filter disc, and the filtered petroleum ether. Identify the blank as to the sample number on an identification tag and attach it to the lid of the petri dish.[6]

8.2.2 Procedure for Samples:

8.2.2.1 Using forceps, remove one filter disc from its container. Rinse the top surface of the filter disc with a stream of filtered petroleum ether from a wash bottle. Place the filter disc-printed grid side up on the fritted glass base.

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[5] A hood pressured with filtered air is especially advantageous for both microscopy and filtration. At a minimum, a dust cover such as a polyethylene bag, with appropriate openings, pulled over the barrel of the microscope, is required to preclude fall out of dust from the air.

[6] The maximum particle count value of a blank analysis shall be no greater than 10% of the count of an acceptable sample for a specific laboratory.

8.2.2.2 Immediately lower the filter funnel onto the fritted glass base, secure with the holding clamp and place cap on top of the filter funnel. (Do not slide filter funnel over the filter disc during this process.)

8.2.2.3 Thoroughly agitate the sample bottle to assure that all solid particles are in suspension.

8.2.2.4 Remove the sample bottle cap and plastic film. Remove the filter cap and pour sample into the filter funnel. Replace the filter cap.

8.2.2.5 Pour 100 ml. of filtered petroleum ether into the sample bottle; replace the plastic film and bottle; agitate and proceed as in 8.2.2.4.

8.2.2.6 Rinse funnel walls with approximately 50 ml. of filtered petroleum ether from wash bottle.

8.2.2.7 Apply vacuum to the filtering apparatus. When the filtration is approximately one-half complete, release the vacuum.

8.2.2.8 While some liquid still remains in the funnel, using the stream from a wash bottle, carefully wash down the sides of the funnel with filtered petroleum ether (approximately 50 ml.). Replace the filter cap.

8.2.2.9 Apply vacuum and allow to operate until the filter disc is completely dry. Do not rinse the funnel walls further after the filter has become dry, as this will upset the distribution of particles on the filter surface. Turn off the vacuum and simultaneously remove the holding clamp and filter funnel so that the filter disc remains on the fritted glass base.

8.2.2.10 Using forceps, carefully remove the filter disc from the top of the fritted glass base. 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.

8.3 Microscope Analysis Procedure: Particles are to be counted and tabulated in the following order: fibers, particles greater than 100 microns, 50-100 microns, 25-50 microns, and 15-25 microns, and 5-15 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 their length exceeds 100 microns. The size of a particle is determined by its greatest dimension. (See Para. 8.3.4.7.)

8.3.1 Place petri dish under the microscope dust cover and remove petri dish cover.

8.3.2 Adjust the microscope lamp intensity to obtain maximum particle definition.

8.3.3 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 eye-piece.

8.3.3.1 Using a stage micrometer, calibrate the measuring eye-piece (ocular micrometer) for each magnification.

8.3.4 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 9.

8.3.4.1 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.

8.3.4.2 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.

8.3.4.3 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.

8.3.4.4 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.

8.3.4.5 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.[7] To arrive at the total statistical count, the sum of the particles counted in the areas is multiplied by the calibration factor.[8]

8.3.4.6 In no case shall the total number of particles in a unit area exceed 50 particles of a size range. See Figure 1 for the alternate unit areas.

8.3.4.7 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.

8.3.4.8 The largest dimension of the particle determines the size category into which the particle is placed.

8.4 Calculation of Calibration Factor:

8.4.1 The calibration factor is the ratio of the effective filtration area (100 grid squares or 9.6 cm^2) to the area counted.

8.4.2 To arrive at a calibration factor, start with the microscope adjusted for the power under consideration.

8.4.3 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 side of the grid square or 3.08 mm.

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[7] The basic unit area for the statistical count not based on the grid markings on the filter, when using the ocular micrometer, will be the area defined by scanning the length of an individual grid square with the length of the ocular micrometer scale or any appropriate portion of the scale.

[8] Calibration factor defined in 8.4.