AEROSPACE RECOMMENDED PRACTICE

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(R) Procedure for the Determination of Particulate Contamination in Hydraulic Fluids by the Control Filter Gravimetric Procedure

RATIONALE

The document has been significantly updated to make it reflect current practices, and two new appendices have been introduced to detail procedures that are referenced in the document.

TABLE OF CONTENTS

1. SCOPE 3

2. REFERENCES 3
2.1 Applicable Documents 3
2.1.1 SAE Publications 3
2.1.2 ISO Publications 3
2.2 Definitions 3

3. OUTLINE OF METHOD 3

4. SOLVENTS/REAGENTS 4

5. APPARATUS 4
5.1 Expendable Materials 4
5.2 Nonexpendable Equipment 4

6. PRECAUTIONS TO REDUCE THE EFFECT OF ELECTROSTATIC DISCHARGE 5

7. COMPATIBILITY OF THE FILTER MEMBRANE WITH THE CHOSEN SOLVENT AND TEST FLUID 5

8. PREPARATION OF EQUIPMENT 5
8.1 Sample Bottles and Glass Funnel (Borosilicate Glass or Stainless Steel Funnel) 5

9. SAMPLING 6
9.1 Method of Sampling 6
9.2 Sample Volume 6

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10. TEST PROCEDURE 6
10.1 Preparation of Test and Control Filter Discs 6
10.2 Double Membrane Method 6

11. CALCULATIONS 8

12. NOTES 8
12.1 Revision Indicator 8

APPENDIX A COMPATIBILITY TESTS FOR MEMBRANE FILTER DISCS WITH SOLVENTS AND WITH HYDRAULIC FLUIDS 9

APPENDIX B STABILIZATION OF CONTROL AND TEST MEMBRANE FILTER DISCS 12

Table 1 Presentation of test results 8

1. SCOPE

This ARP describes a gravimetric method for the determination of particulate contaminant in hydraulic fluids by the control filter technique.

NOTE: With this method, detectable contamination levels down to 0.2 mg (7.0×10^{-6} ounces) per sample can be obtained with a standard deviation of ±0.1 mg ($\pm 3.5 \times 10^{-6}$ ounces).

2. REFERENCES

2.1 APPLICABLE DOCUMENTS

The following publications form a part of this document to the extent specified herein. The latest issue of SAE publications shall apply. The applicable issue of other publications shall be the issue in effect on the date of the purchase order. In the event of conflict between the text of this document and references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific examption has been obtained.

2.1.1 SAE Publications

Available from SAE International, 400 Commonwealth Drive, Warrendale, PA 15096-0001, Tel: 877-606-7323 (inside USA and Canada) or +1 724-776-4970 (outside USA), www.sae.org.

ARP5376 Methods, Locations And Criteria For System Sampling And Measuring The Solid Particle Contamination Of Hydraulic Fluids

2.1.2 ISO Publications

Copies of these documents are available online at http://webstore.ansi.org/.

ISO 384 Laboratory glass and plastics ware - Principles of design and construction of volumetric instruments

ISO 3722 Hydraulic fluid power - Fluid sample containers - Qualifying and controlling cleaning methods

ISO 4021 Hydraulic fluid power - Particulate contamination analysis - Extraction of fluid samples from lines of operating system

ISO 4405 Hydraulic fluid power - Fluid contamination - Determination of particulate contamination level by the gravimetric method

ISO 4788 Laboratory glassware - Graduated measuring cylinders

2.2 Definitions

GRAVIMETRIC PROCEDURE: Gravimetric analysis describes a method for the quantitative determination of contaminant in a system based on the mass of contaminant measured.

TARE WEIGHT: The tare weight of a filter is its initial weight as received after equilibration to ambient room conditions.

3. OUTLINE OF METHOD

Total particulate contamination, including both organic and inorganic material, is determined by filtering a quantity of hydraulic fluid through a pre-weighed, 47.0 mm (1.85 inches diameter), 0.8 µm (3.14×10^{-5} inches) membrane filter; solvent flushing to remove filter retained fluid; oven drying followed by equilibration with ambient room conditions; then a final weighing.

In addition, a control filter is carried through the analysis in order to control and correct for test variables and possible sources of error. The control filter is subjected to the same procedures as each test filter with the exception of actual contaminant filtration, thereby serving as an indicator of any changes in balance calibration, changes in filter weights resulting from moisture content change (humidity variation), desorption of filter extractable, or absorption of hydraulic fluid constituents. A further function of the control filter is to provide a blank analysis run concurrently with the actual test sample.

4. SOLVENTS/REAGENTS

Solvents/reagents should be used with gravimetric contamination levels less than 0.1 mg/L (1.2×10^{-5} oz/gal) and compatible with the fluid being analyzed and filter membrane disc. It is important that solvents and hydraulic fluid do not react with filter membrane disc which would provide erroneous results using this procedure.

The following solvents are suggested (or other suitable solvents with similar properties) for hydraulic fluids, but they should be verified and documented using the test procedure in Appendix A before use:

1. Analytical Grade Petroleum Ether or Hexane (analytical grade with less than 0.1 mg/L (1.2×10^{-5} oz/gal) of solid contaminants) 30 to 60 °C (86 to 140 °F) Boiling Point or Analytical Grade Hexane or VM&P Naphtha should be used. The text should use the term "Petroleum Ether." It is a regulated chemical.

   CAUTION: These fluids are flammable and not environmentally friendly.

2. Reagent Grade Isopropyl Alcohol (Acetone Free - Analytical grade with less than 0.1 mg/L (1.2×10^{-5} oz/gal) of solid contaminants). It is a regulated chemical.

   CAUTION: This fluid is flammable.

3. Heptane (analytical grade with less than 0.1 mg/L (1.2×10^{-5} oz/gal) of solid contaminants). It is a regulated chemical.

   CAUTION: This fluid is flammable.

5. APPARATUS

5.1 Expendable Materials

Membrane filters, 47 mm (1.85 inches diameter), 0.8 µm (3.14×10^{-5} inches) pore size or finer, water extractables 5% maximum. Industry standard membranes of 0.45 µm (1.77×10^{-5} inches) pore should be acceptable as long as they are compatible with the fluids used.

5.2 Nonexpendable Equipment

These are the following:

1. A borosilicate glass filter holder assembly, 47 mm diameter, (1.850 inches) with a funnel approximately 300 mL (10.144 fluid ounces) capacity - pre-washed.

2. A side arm filter flask for collecting samples 1 L (35 fluid ounces) volume.

3. A vacuum pump or aspirator, capable of drawing 25 in Hg (12.27 psi) vacuum pressure.

4. Stainless steel flat-bladed forceps with un-serrated tips.

5. An analytical or electronic balance capable of weighing to 0.05 mg (1.7×10^{-6} ounces) sensitivity and accuracy of 0.05 mg (1.7×10^{-6} ounces).

   NOTE: Place a porous or open container holding desiccant granules into the balance case to maintain low relative humidity.

6. Covered glass Petri dishes, 150 mm (5.90 inches) diameter.

7. A drying oven capable of maintaining a minimum temperature of 43 °C ± 3 °C (110 °F ± 3 °F).

8. An assembly for dispensing clean solvent.

9. Glass sample bottles, small mouth, with screw cap, scored at the 100 mL (3.38 fluid ounces) level.

10. A volumetric flask or graduated cylinders, 100, 500, and 1000 mL (3.38, 16.9, and 33.8 fluid ounces) in accordance with ISO 4788.

11. A pressurized solvent dispenser with a filter holder arranged to pass the solvent though a 0.45 µm (1.77×10^{-5} inches) pore membrane filter during dispensing.

12. A desiccator capable of being sealed from the atmosphere and containing a means for removing the moisture.

    NOTE: In laboratory rooms with good controlled environment, desiccator may not be necessary. See Appendix B for the conditions under which this equipment is desired.

13. A Static Dissipator Additive (SDA) to reduce electrostatic charge in fluid.

14. A three-axis shaker - any commercial three-axis shaker like a paint shaker is acceptable.

15. An ultrasonic bath rated at 0.3 to 1.0 W/cm^2 (1.9 to 6.45 W/in^2) of bottom area.

16. An electrically grounded clamp which bonds the filter holder and funnel.

6. PRECAUTIONS TO REDUCE THE EFFECT OF ELECTROSTATIC DISCHARGE

Sample bottles and beakers made of either glass or plastic have tendency to develop an electrostatic discharge when hydrocarbon fluid passes over them. This phenomenon of buildup of electrostatic charges will affect the test results. It also may result in hazardous conditions. The following methods are suggested for reducing the effect of electrostatic charges on the test results and ensure safety during the test.

1. Grounding the filter holder clamping device.

2. Add a static dissipator additive to the solvent to a minimum concentration of 100 ppm. Check with the manufacturer of the anti-static agents on the recommended concentration required for the test fluid. The anti-static agent should be the analytical grade. In addition, it is recommended the laboratory performing tests be aware of the shelf life of these anti-static agents.

7. COMPATIBILITY OF THE FILTER MEMBRANE WITH THE CHOSEN SOLVENT AND TEST FLUID

It is necessary that the solvent selected does not chemically react with the filter membrane and fail to act as fluid rinsing agent. Also, the test fluid being analyzed may chemically react/affect the membrane filter and react with solvent. It is very important that solvent and membrane should be selected for compatibility using the information from the membrane manufacturer. If the information is not available, follow the compatibility check procedure given in Appendix A.

8. PREPARATION OF EQUIPMENT

8.1 Sample Bottles and Glass Funnel (Borosilicate Glass or Stainless Steel Funnel)

Sample bottles should be calibrated and scored, or otherwise permanently marked, at the 100 mL (3.38 fluid ounces) level as determined by transferring 100 mL (3.38 fluid ounces) of water from a volumetric flask or graduated cylinder. Graduations on the funnel should not be used for sample size measurements since they are only approximate. The graduated cylinders need to conform to ISO 4788 standard.

Bottles and funnels should be cleaned by washing with liquid detergent in water, rinsing with hot water and finally rinsing with filtered isopropyl alcohol or solvent that has passed through 0.45 µm (1.77×10^{-5} inches) pore membrane. Sample bottles should be rinsed with filtered reagent that has passed through 0.45 µm (1.77×10^{-5} inches) pore membrane just prior to capping. Filter funnels should be rinsed with filtered reagent just prior to the filtration procedure.

9. SAMPLING

Variables can affect the outcome of the results obtained by using this procedure. Therefore, it is highly recommended to ensure that that sample volumes are validated and sampling procedures in obtaining the fluid samples are consistent with the applicable SAE and ISO standards as detailed in Section 2.

9.1 Method of Sampling

A specific technique for obtaining hydraulic fluid samples is not detailed in this procedure. Such methods should be established by the individual agency or laboratory - each should be in accordance with ARP5376. Extreme care should be taken to insure that the samples are representative of the system fluid being tested, and free of significant external contamination.

9.2 Sample Volume

The sample volume recommended is 100 mL ± 5 mL (3.38 fluid ounces ± 0.170 fluid ounces).

The hydraulic fluid being tested should be transferred to a clean sample bottle and filled to the 100 mL (3.38 fluid ounce) mark.

In the case where contaminant levels may be very low, a larger sample can be taken and analyzed. Very low contaminant levels would not provide much of a weight difference and therefore be difficult to weigh accurately. A larger sample size would provide more contaminant to weigh and may allow a more accurate measurement. On the other hand, a very contaminated fluid may cause too much of a buildup on the membrane. A smaller sample size may allow for a more accurate measurement in the case of a highly contaminated sample.

10. TEST PROCEDURE

There are two methods of conducting test. One is a double membrane method. The other is a single membrane method. It is recommended that only double membrane method is used in order to obtain more repeatable results. The single membrane method is discouraged for accuracy and repeatability reasons unless the laboratory has long standing experience and has a good quality membrane with acceptable very low variability from one to the other.

10.1 Preparation of Test and Control Filter Discs

The procedure should be as follows:

1. Using forceps, remove two membrane filter discs from the packages and place each in separate Petri dishes.

2. Identify with the letters “T1” the filter membrane with a ballpoint pen on the filter edge, and the corresponding Petri dish (T) designates the test filter membrane and “1” refers to the number of the test. Similarly, “C1” designates the control filter membrane and “1” refers to the number of the tests to be run concurrently on the particular fluid. It is optional to perform more than one test on one fluid sample if there is enough quantity of test sample fluid to start with, and if it is specified as a requirement.

   CAUTION: UNDER NO CIRCUMSTANCES SHOULD AN OPERATOR TOUCH THE FILTER MEMBRANE DISC BY HAND BECAUSE THIS WILL CONTAMINATE IT.

10.2 Double Membrane Method

The procedure should be as follows:

1. Select a membrane filter disc that has been determined to be compatible with the solvent and fluid being tested from the procedure in Appendix A.

2. Place the labeled Petri dishes with the appropriate membranes labeled “T1” and “C1” from 10.1.2, with covers partially open, in the oven at 38 to 65 °C ± 3 °C (100 to 150 °F) for 15 minutes.

3. Replace the covers and remove the Petri dishes from the oven.

4. Stabilize the membrane filter discs in accordance with Appendix B.

5. Remove the test membrane filter disc (T1) from the Petri dish and place it on the balance pan. Weigh the test filter membrane to the nearest 0.05 mg (1.7 x 10^{-6} ounces), and record the weights as initial weight of “T1.” Return the test membrane filter disc to its Petri dish and replace the cover.

6. Remove the control membrane filter disc (C1) from the Petri disc and place it on balance pan. Weigh the mass of the control membrane to the nearest 0.05 mg (1.7 x 10^{-6} ounces), and record its value as initial weight of “C1.” Return the control membrane filter disc to its Petri dish and replace cover.

7. Shake the hydraulic fluid sample bottle for 5 minutes along each axis until all contaminant is uniformly distributed.

8. Measure-out using graduated cylinders in accordance with 10.1, 100 mL (3.38 fluid ounces) of test fluid into a clean sample container.

9. Place the sample container in ultra-sonic bath for 2 to 3 minutes to remove entrained air. After all the air has been removed from the fluid sample, carefully remove the sample container from the bath and wipe the exterior of the container with a clean lint free cloth.

10. Place the pre-weighed membrane discs on the filter holder base with control disc (C1) on the bottom and (T1) on the top. Apply vacuum to hold the discs in place. Place the funnel over the discs and clamp the funnel and holder together. The clamp should be electrically grounded to prevent static buildup and discharge.

11. Pour the sample fluid from 10.2.8 into the funnel. The fluid may be diluted with clean laboratory grade solvent to reduce filtration time.

12. With the mouth of the cylinder or sample container over the inlet of the funnel, rinse the cylinder or the sample container with appropriate solvent which is pressurized and pre-filtered during dispensing. As the fluid is filtering, pre-filtered solvent can be added to reduce the filtering time.

13. After all the sample fluid has passed through the membrane discs T1 and C1, with the vacuum still applied, gently rinse with solvent the funnel walls in concentric circles and around the funnel periphery, removing all the contaminants from the funnel wall and on to the filter’s discs.

14. With vacuum still applied, remove the clamping device and funnel.

15. Rinse the membrane filter discs using solvents in concentric circles around the periphery of each towards the center. Continue rinsing until all visible fluid residue has been removed. The pressure and velocity of the solvent rinsing stream should be low enough to prevent washing contaminant particles from membrane filter discs.

16. Stop the vacuum and remove the test membrane T1, placing it on its Petri dish. Replace the Petri dish cover.

17. Restart the vacuum and re-rinse the control membrane filter disc (C1) in concentric circles around the periphery towards the center. Continue rinsing until the traces of visible fluid residue have been removed.

18. Stop the vacuum and remove the control membrane (C1), placing it in its Petri dish labeled C1. Replace the Petri dish cover and place in the oven at 43 °C ± 3 °C (110 °F ± 3 °F) for 15 minutes.

19. Place the labeled Petri dishes with the appropriate membranes labeled “T1” and “C1” from 11.1.2, with covers partially open, in the oven to dry at 38 to 80 °C ± 3 °C (100 to 176 °F ± 3 °F) for 15 minutes. Stabilize both the membrane filter discs T1 and C1 in accordance with Appendix B.

20. Remove the membrane filter discs C1 and T1 from their respective Petri dishes one at a time and weigh them to the nearest 0.05 mg (1.7 x 10^{-6} ounces). Record the values as final weights for C1 and T1 filter membrane discs.

21. Save the filter membranes C1 and T1 in their respective Petri dishes. Replace the cover on Petri dishes and place them in a dry place until completion of the gravimetric analysis.

11. CALCULATIONS

The calculations should be as follows:

1. Subtract initial weight from final weight of each filter membrane discs C1 and T1.

2. Determine gain or loss in weight of control filter by appropriate subtraction.

   NOTE: A control filter weight change (Delta Weight of C1) of 1% of initial weight of C1 may indicate an incompatibility between solvent and the fluid or membrane, or between the fluid and the membrane. It may also indicate inadequate solvent flushing, unclean environment when handling membranes or lack of drying time. Re-flush both membrane discs in accordance with 10.2.15 or revisit Appendix A for a compatibility check.

3. Apply control filter weight change as a correction factor to each test result, subtracting this factor when the control filter shows a weight increase or adding the factor when the control filter shows a weight decrease.

4. The reporting of data should be reported in accordance with Table 1 (which illustrates an example of three tests done on one fluid sample). If only one test was done on the test fluid, then there will be only one control filter “C1” and one test filter “T1”.

NOTE: Unless otherwise specified, only one set of test is done per one set of fluid sample.

Table 1 - Presentation of test results

Test #	Test T1	Test T2	Test T3	Control C1	Control C2	Control C3
Final Weight (mg):	65.30	63.10	68.10	66.20	66.20	66.15
Initial Weight (mg):	64.10	61.75	66.65	65.90	65.85	65.90
Δ Weight (mg):	1.20	1.35	1.45	+0.30	+0.35	+0.25
Control Factor:	-0.30	-0.35	-0.25			
Results: mg/Sample	0.90	1.00	1.20			
Sample Volume:	100 mL	100 mL	100 mL			
Results: mg/100 mL	0.90	1.00	1.20			

12. NOTES

12.1 Revision Indicator

A change bar (I) located in the left margin is for the convenience of the user in locating areas where technical revisions, not editorial changes, have been made to the previous issue of this document. An (R) symbol to the left of the document title indicates a complete revision of the document, including technical revisions. Change bars and (R) are not used in original publications, nor in documents that contain editorial changes only.

PREPARED BY SAE PANEL A-6C1, CONTAMINATION AND FILTRATION OF COMMITTEE A-6, AEROSPACE ACTUATION, CONTROL AND FLUID POWER SYSTEMS

APPENDIX A - COMPATIBILITY TESTS FOR MEMBRANE FILTER DISCS WITH SOLVENTS AND WITH HYDRAULIC FLUIDS

A.1 FOREWORD

It is important to check the compatibility of the solvent and hydraulic fluid with the membrane that is used for filtering out the solid contaminants before testing for gravimetric levels in the sample fluid. If the material is not compatible with solvent or hydraulic fluid under test, results can be misleading because the hydraulic fluid and/or the solvent may chemically react with filter membrane.

A.2 RECOMMENDED REQUIREMENTS

It is necessary to perform the procedure whenever new hydraulic fluid or membrane or solvent is used in the gravimetric analysis. If the same hydraulic fluid of the same chemical base is used, individual tests again with different fluid is not necessary because it is safe to assume that the fluids of the same chemical base will react with the membrane material the same way.

Make sure that manufacturer of part number and material it is made from is recorded in the test report, and also the date it was checked needs to be reported in the test report. It is recommended that this verification procedure in this Appendix A is performed and documented by the laboratory, at least once, to ensure integrity of the test results whenever new types of fluids, solvents, and filter membranes are used. It also serves as a good verification of the process used.

It is recommended that the user contact the manufacturer of the membrane or the fluid manufacturer to ascertain that both are compatible.

A.3 PREPARATION OF THE MEMBRANE FILTER DISCS

The procedure should be as follows:

1. Remove two membrane filter discs from the packages using forceps (without touching with hands), place them in separate Petri dishes.

2. Mark each Petri Dish with a letter “S” for solvent and the other with letter “F” for fluid.

3. Place both Petri dishes, with covers partially open, in oven at 43 °C ± 3 °C (110 °F ± 3 °F) for 15 minutes.

4. Replace the covers and remove the Petri Dishes from the oven.

5. Using procedure given in Appendix B, stabilize membrane discs.

6. Remove the membrane disc to be evaluated for compatibility with solvent “S” from the Petri dish and weight it to nearest 0.05 mg (1.76 x 10^{-6} ounces) or better. Return the disc to its Petri dish marked “S” and replace cover. Record the mass as initial mass of disc “S.”

7. Remove the membrane disc to be evaluated for compatibility with “F” from the Petri dish and weight it to nearest 0.05 mg (1.76 x 10^{-6} ounces) or better. Return the disc to its Petri dish marked “F” and replace cover. Record the mass as initial mass of disc “F.”

A.4 PROCEDURE FOR COMPATIBILITY CHECK OF FILTRATION MEMBRANE WITH SOLVENTS

The procedure should be as follows:

1. Always use forceps when handling the membrane discs.

2. Place the pre-weighed disc “S” to be evaluated for compatibility with solvent on the filter base. Turn on the vacuum to hold the disc in place. Place the funnel over disc and clamp the funnel and holder together.

3. Pass 500 mL (17.0 fluid ounces) of clean laboratory grade solvent (with gravimetric concentration guaranteed to be less than 0.1 mg/L (1.3 x 10^{-4} oz/gal) through membrane filter disc.