



Standard Test Method for Insoluble Contamination of Hydraulic Fluids by Gravimetric Analysis¹

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This standard has been approved for use by agencies of the Department of Defense.

^{ε1} NOTE—Editorial corrections were made throughout in April 2000.

1. Scope

1.1 This test method covers the determination of insoluble contamination in hydraulic fluids by gravimetric analyses. The contamination determined includes both particulate and gel-like matter, organic and inorganic, which is retained on a membrane filter disk of pore diameter as required by applicable specifications (usually 0.45 μm or 0.80 μm).

1.2 To indicate the nature and distribution of the particulate contamination, the gravimetric method should be supplemented by occasional particle counts of typical samples in accordance with Test Method F 312.

1.3 The values stated in SI units are to be regarded as the standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For a specific warning statement, see 6.1.

2. Referenced Documents

2.1 ASTM Standards:

A 555/A 555M Specification for General Requirements for Stainless Steel Wire and Wire Rods²

D 1836 Specification for Commercial Hexanes³

D 2021 Specification for Neutral Detergent, 40 Percent Alkylbenzene Sulfonate Type⁴

E 319 Practice for the Evaluation of Single-Pan Mechanical Balances⁵

F 302 Practice for Field Sampling of Aerospace Fluids in Containers⁶

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum and Petroleum Products and is the direct responsibility of Subcommittee D02.N0.02 on Industrial Applications.

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² Annual Book of ASTM Standards, Vol 01.03.

³ Annual Book of ASTM Standards, Vol 06.04.

⁴ Annual Book of ASTM Standards, Vol 15.04.

⁵ Annual Book of ASTM Standards, Vol 14.04.

⁶ Annual Book of ASTM Standards, Vol 15.03.

F 303 Practices for Sampling Aerospace Fluids from Components⁶

F 312 Methods for Microscopical Sizing and Counting Particles from Aerospace Fluids on Membrane Filters⁷

F 314 Test Methods for Identification of Metallic and Fibrous Contaminants in Aerospace Fluids⁷

2.2 Military Standard:

MIL-C-81302 C Cleaning Compound Solvent Trichlorotrifluoroethane⁸

3. Summary of Test Method

3.1 The insoluble contamination is determined by passing a given quantity of a fluid sample through a membrane filter disk and measuring the resultant increase in the weight of the filter. The fluid sample is drawn through the filter by a vacuum and the insoluble contamination is collected on the surface of the filter. In addition, the filter disk is microscopically scanned for excessively large particles, fibers, or other unusual conditions.

4. Significance and Use

4.1 This test method indicates and measures the amount of insoluble contamination of hydraulic fluids. Minimizing the levels of insoluble contamination of hydraulic fluids is essential for the satisfactory performance and long life of the equipment. Insoluble contamination can not only plug filters but can damage functional system components resulting in wear and eventual system failure.

5. Apparatus

5.1 *Microbalance*, accurate to 0.005 mg, and the zero shall not drift more than 0.005 mg during the test period. The rated accuracy shall be obtainable by personnel actually making the weighings, under actual conditions of use and shall be verified in accordance with Practice E 319.

5.2 *Membrane Filter Support*, fritted glass, sintered metal, or stainless steel screen, to support 25-mm or 47 to 51-mm membrane filters. The support shall be designed to enable attachment of a vacuum flask.

⁷ Annual Book of ASTM Standards, Vol 14.02.

⁸ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

5.3 *Filtration Funnel*, glass or stainless steel, minimum capacity 15 mL, designed to enable attachment to the membrane filter support by means of a suitable clamping device. The filter funnel is calibrated to indicate volume.

5.4 *Vacuum Flask*, 250-mL, with rubber stopper.

5.5 *Filtered Liquid Dispensers (2)*—Washing bottles or other dispensers capable of delivering liquid through a 0.45- μ m in-line membrane filter.

5.6 *Air Ionizer*, alpha emitter, 18.5 MBq, of polonium-210, with a useful life of 1.5 years to a final value of 1.1 MBq.⁹

5.7 *Membrane Filters (2)*, 25 mm or 47 to 51 mm in diameter, with pore diameter as required.

5.8 *Microscope*, capable of 35 \times magnification.

5.9 *Vacuum Source*, capable of pulling 550 mm Hg. An electrically driven vacuum pump must be explosion-proof.

5.10 *Drying Oven*, capable of maintaining a temperature of 80°C.

5.11 *Sample Bottles*, with contamination-proof caps. Aluminum-foil wrapped stoppers or polyethylene liners between cap and bottle have proved satisfactory.

5.12 *Graduated Cylinder*, 100-mL (May be replaced by volume-calibrated filter funnel.)

5.13 *Petri Dishes*, covered glass, 150 mm in diameter.

5.14 *Forceps*, with unserrated tips.

5.15 *Calibration Weights*, for microbalance, 10 mg and 20 mg, accurate to 0.005 mg and made from nonmagnetic material conforming to Type 310 in Specification A 555/A 555M. The accuracy is traceable to the National Bureau of Standards as discussed in their Technical Note 288 .

6. Reagents

6.1 *Commercial Hexane*, conforming to Specifications D 1836 (**Warning**-Flammable. Harmful if inhaled. Skin irritant on repeated contact. Eye irritant. Aspiration hazard.).

6.2 *Detergent*, free-rinsing. Material conforming to Specification D 2021.

6.3 *Isopropyl Alcohol*, acetone-free (**Warning**-Flammable. Harmful if inhaled. Skin irritant on repeated contact. Eye irritant. Aspiration hazard.).

6.4 *Refrigerant-113* (trichlorotrifluoroethane) conforming to U.S. Military Specification MIL-C-81302C (**Warning**-Flammable. Harmful if inhaled. Skin irritant on repeated contact. Eye irritant. Aspiration hazard.).

7. Sample

7.1 A 100 \pm 1-mL fluid sample shall be used for this method. Larger or smaller samples may be used, however, to meet problems of unusually high or low contamination levels.

NOTE 1—Methods for obtaining the fluid samples are not specified in this method. These methods should be established by the individual agencies or laboratories in accordance with their requirements. However, extreme care should be taken to ensure that the samples are representative and free of external contamination.

7.2 The following ASTM sampling test methods should be used when applicable: Practices F 302 and F 303.

8. Preparation of Apparatus

8.1 The filtration funnel, petri dishes, graduated cylinders, and sample bottles shall be cleaned before each use by the following method:

8.1.1 Thoroughly wash in a solution of detergent and hot water.

8.1.2 Rinse with hot tap water and finally with distilled or deionized water.

8.1.3 Rinse twice with filtered isopropyl alcohol (delivered through a filtered liquid dispenser).

8.1.4 Rinse twice with filtered commercial hexane (delivered through filtered liquid dispenser).

8.1.5 Leave approximately 1 mL of commercial hexane in the bottom of each sample bottle. Replace sample bottle cap (**Warning**-Flammable. Harmful if inhaled. Skin irritant on repeated contact. Eye irritant. Aspiration hazard.).

9. Procedure

9.1 Select two membrane filters of the pore diameter required. Allow the filters to stabilize to equilibrium with ambient room conditions. Record room temperature and relative humidity.

9.2 Take precautions to minimize apparatus contamination from airborne dust. Use a protective hood or cover.

NOTE 2—One of the filters mentioned in 9.1 is for a blank determination, which is necessary to provide a correction factor for adsorption of hydraulic fluid by the filter, desorption of filter constituents by the solvent, and variations of moisture content of the filter. This blank should be determined for each sample of a given composition and history. In a series of samples of similar fluids with similar history, the blank need be determined only once. However, with any change of sample volume, a new blank should be determined as the relationship of blank correction to sample volume cannot be extrapolated.

9.3 Using forceps, weigh each filter on a microbalance to the nearest 0.005 mg.

9.4 Place the filters, one above the other, in the filtration apparatus. Ground the clamp to reduce static buildup during filtration.

9.5 Shake the sample container for approximately 1 min.

9.6 Measure the desired sample volume with a graduated cylinder and pour into the filter funnel, or pour the required volume directly into a calibrated filter funnel. Apply a vacuum and filter the entire sample.

9.7 Use a stream of filtered commercial hexane or Refrigerant 113 from a filtered liquid dispenser to rinse the residual sample from the graduated cylinder into the filter funnel and to wash down the inside of the filter funnel. The outside of the filter unit should also be washed at the joint between the filter holder and filter base to remove fluid between the holder and the filter disk. Repeat the washing twice so that a total of approximately 100 mL of commercial hexane or Refrigerant 113 is used for the washing process.

9.8 With the vacuum still applied, remove the funnel, taking care not to disturb the filter. With a fine steady stream, wash any residual hydraulic fluid from the periphery of the filter with filtered commercial hexane. The washing action should be towards the center with care taken not to disturb the deposited contaminants on the filter surface. Continue washing until the total volume of filtered hexane equals 250 to 300 mL.

⁹ May be obtained from any source approved by U.S. Nuclear Regulatory Commission.

9.9 Dry the filters with aspirator action and release the vacuum.

9.10 Using forceps, remove the filters and place in a covered petri dish. Place the petri dish in an 80°C oven for 15 min. Other drying methods such as 5 min of continuous aspirator action or vacuum dessication may be substituted for the oven-drying method if such substituted method proves superior to the oven method in achieving a constant weight upon continued drying. The nature of the contaminant will dictate the drying method to be used.

9.11 Allow the filters to equilibrate to ambient room conditions for 5 min in the petri dish with cover ajar. Record the room temperature and relative humidity. When these values differ from ambient conditions recorded as described in 9.1, results will be invalid unless a blank filter is run concurrently (see Note 2). Remove static electricity by placing each membrane on the air ionizer for several seconds.

9.12 Weigh each filter to the nearest 0.005 mg.

9.13 Inspect the same filter (upper membrane) under a microscope at approximately 35×. The presence of large or unusual particles or fibers shall be sufficient cause for requesting another sampling and analysis even though the allowable contamination weight is not exceeded.

9.14 For further examination of the filter, reference may be made to Test Method F 314 (**Warning**-Flammable. Harmful if inhaled. Skin irritant on repeated contact. Eye irritant. Aspiration hazard.).

10. Calculations

10.1 Calculate the weight of the contaminant as follows:

10.1.1 Subtract the initial weight (9.3) from the final weight (9.12) of the test filter (upper membrane) and of the blank filter (lower membrane).

10.1.2 Subtract the weight change of the blank from the weight change of the test filter.

10.1.3 The contamination weight is expressed as milligrams/100 mL of fluid sample retained on the specified pore diameter membrane filter.

11. Precision and Bias

11.1 *Precision:*

11.1.1 *Repeatability*—Duplicate results by the same operator should be considered suspect if they differ by more than 3.6 % of the weight of contamination per 100 mL of the sample.

11.1.2 *Reproducibility*—Available data do not justify any statement.

11.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure for measuring insoluble contamination, no bias can be determined.

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