Standard Practice for Field Sampling of Aerospace Fluids in Containers¹

This standard is issued under the fixed designation F 302; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

- 1.1 This practice covers field sampling of fluids from hermetically sealed containers and other fluid containers of 55-gal (208-L) volume maximum. It may be utilized at manufacturing, storage, or use levels for obtaining representative fluid samples for chemical, physical, or particulate matter determinations.
- 1.2 Use of this practice depends upon variables such as fluid toxicity, restrictive fluid odors, fluid flammability, etc. It is suitable for most hydraulic fluids; however, care should be exercised in determining compatibility before use.²
- 1.3 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.
- 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 hazard statement, see 6.5.1.

2. Referenced Documents

- 2.1 ASTM Standards:
- D 1193 Specification for Reagent Water³
- D 1836 Specification for Commercial Hexanes⁴
- D 2021 Specification for Neutral Detergent, 40 Percent Alkylbenzene Sulfonate Type⁵
- F 311 Practice for Processing Aerospace Liquid Samples for Particulate Contamination Analysis Using Membrane Filters⁶
- F 313 Test Method for Insoluble Contamination of Hydraulic Fluids by Gravimetric Analysis⁷

F 314 Test Method for Identification of Metallic and Fibrous Contaminants in Aerospace Fluids⁶

2.2 Military Standard:⁸

MIL-C-81302 C Cleaning Compound Solvent Trichlorotrifluoroethane

3. Summary of Practice

3.1 The minimum requirements for container agitation, sample withdrawal, and sample transfer are given in this practice. Precautions to assure sampling reliability are included in the procedure to the extent required by normal processing conditions. The procedure involves agitating the container, withdrawing with a suitable instrument capable of creating a vacuum, a predetermined quantity of fluid, and immediately transferring it to a vessel, properly identified, to hold for analysis by a stipulated method.

4. Significance and Use

4.1 Samples obtained by use of this practice are intended for processing in accordance with Practice F 311, Test Method F 313, and Test Method F 314, and other chemical or physical methods of analysis.

5. Apparatus

5.1 *Pipet*, volumetric transfer or equivalent rubber-bulb type. A taper-jointed type, as shown in Fig. 1, 22 in. (560 mm) long, calibrated to deliver 100 mL at 20°C, is also acceptable. This type provides for ease of maintenance by being separable at the midpoint of the bulb.

Note 1—The volume capacities selected for the pipet and sample bottles shall be as required for the sample volume desired. Normally a 100 \pm 5-mL sample is standard, which would require a capacity of approximately 125 mL. Unless otherwise indicated, it is intended that a sample volume of 100 \pm 5 mL be used for accomplishing the methods defined herein.

- 5.2 Bottles, sample, wide-mouth type (Note 1).
- 5.3 Solvent Filtering Dispenser—An apparatus to dispense a stream of 2.0 µm or finer membrane-filtered fluid.
- 5.4 Vinylidene Chloride, Polyethylene Terephthalate, or Polyamide Sheet, 4-mil (0.1 mm) min.
- 5.5 Beverage Can Opener (Unplated), sharpened, deburred.

¹ This practice is under the jurisdiction of ASTM Committee E-21 on Space Simulation and Applications of Space Technology and is the direct responsibility of Subcommittee E21.05 on Contamination.

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² Where a special environment is required, a Proposed Laboratory Method for Sampling Aerospace Fluids in Containers is under development in the committee. For further information write to B. R. Hall, American Petroleum Institute, 2101 L St., N.W., Washington, D. C. 20037.

³ Annual Book of ASTM Standards, Vol 11.01.

⁴ Annual Book of ASTM Standards, Vol 06.04.

⁵ Annual Book of ASTM Standards, Vol 15.04.

⁶ Annual Book of ASTM Standards, Vol 14.02.

⁷ Annual Book of ASTM Standards, Vol 15.03.

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



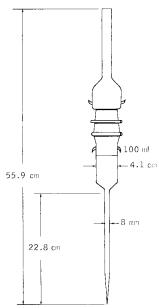


FIG. 1 Separable Pipet

6. Reagents

- 6.1 Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.
- 6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193.
- 6.3 *Detergent*, free-rinsing. Material conforming to Specification D 2021 is suitable.
 - 6.4 Isopropyl Alcohol, acetone-free. 10
 - 6.5 Ligroine (Petroleum Ether), 30 to 60°C.
- 6.5.1 **Warning**—Ligroine and hexane are highly flammable and should be handled with adequate precautions.

Note 2—Ligroine is suggested because of its high-evaporation rate and relatively negligible residue (0.001 %). Other solvents are acceptable as required by the sampling activity, when a comparable evaporation and residue is considered, such as commercial hexanes (see Specification D 1836), trichlorotrifluoroethane conforming to U.S. Military Specification MIL-C-81302 C, etc. In any case, reagent selected should not have a harmful effect on the sampling apparatus, sampled fluid or the equipment to be used in processing the sample, or both.

7. Preparations of Apparatus and Reagents

7.1 Apparatus used in this practice shall be prepared by a reliable process for assurance of essentially contamination-free surfaces.

Note 3—It is recommended that a process shall be used as described in Test Method F 313 or Practice F 311.

7.2 Reagents used in this practice shall be suitably filtered and stored to maintain a level of refinement equivalent to the highest attainable as required by the product evaluation method of determination.

8. Procedure

- 8.1 Select at random representative containers of fluid to be sampled. When defined, selection shall be as required by the test method. A suggested cube-root sampling quantity is shown in Table 1. Alternative plans may be based on the past experience and judgment of the sampling activity. Quantities exceeding those given in Table 1 should be determined by the cube-root method.
- 8.2 Having selected the containers to be sampled, prepare and sample each one individually.
- 8.3 Agitate the fluid container as required to assure safety and completeness of sampling of the particulate matter. The method used shall depend on the height of the container, the fluid viscosity, and the particle size, as related in Fig. 2. First, the container shall be inverted for a period of time sufficient for the particles of the predominant size to fall half the height of the container. This time shall be determined as follows: the fluid viscosity at the temperature of sampling shall be estimated and an appropriate diagonal line on Fig. 2 selected. The predominant particle size will probably be known from past experience; if not, 20 µm may be used as a preliminary estimate. The settling time shall then be read from Fig. 2 for a 1-qt (1-L) container, and the size factor applied if necessary. Any situation not covered by the chart may be calculated from Stokes' law. The time may turn out to be burdensome in some special cases; warming the container would be permissible in such cases provided it will not cause solution of plastic, etc., which might change the particle count. After the inversion period, the container shall be placed on its side and rolled slowly five turns. It shall then be rolled in the opposite direction for five turns and returned to an upright position. The sample shall be drawn within one half the inversion time, after agitation is completed.

Note 4—Hermetically sealed containers of 1 gal $(3.8\ L)$ or less may be shaken by hand or by mechanical vibration for a minimum of $30\ s.$

8.4 Utilizing the filtered ligroine (petroleum ether) (Note 2), dispensed from a washing bottle, carefully wash surface areas of the container that may be exposed to the sampling area selected. The container bung or other areas capable of adversely influencing the accuracy of the sampling apparatus which come in contact with the fluid to be sampled, shall be rinsed with filtered ligroine immediately prior to opening the container.

TABLE 1 Sample Plan

Quantity of Containers	Sample
1 to 10	1
11 to 30	3
31 to 70	4
71 to 150	5
151 to 210	6
210 to 530	8
531 to 1170	10

⁹ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, D. C. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, N. Y., and the "United States Pharmacopeia."

¹⁰ Material conforming to USP XVII, p. 995, is suitable.

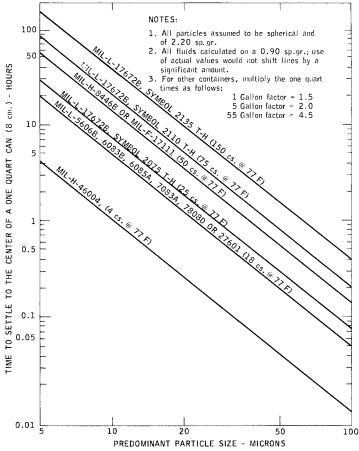


FIG. 2 Inversion Time Required to Distribute Particles in a Closed Container

8.5 To open a hermetically sealed container lay it on its side and quickly puncture the lowest point of the container. This is accomplished utilizing a beverage-type can opener (unplated), or equivalent. Allow approximately 300 to 500 mL of fluid to

flow from the opening prior to placing the container in an upright position. Cover the top of the container with a plastic sheet to preserve cleanliness. (The sheet shall be previously cleaned.) To obtain a sample, select a sample bottle prepared as described in Section 6. Remove the cap from the sample bottle and lay the container to be sampled on its side allowing free flow of the fluid. Place the bottle in the container effluent and collect the required sample volume. Immediately cover the sample bottle and return the container to an upright position and recover it. Identify the sample bottle.

8.6 Withdraw the sample from the drum-type containers as follows: Select a suitable volumetric pipet, prepared as described in Section 7, and withdraw sufficient fluid from the container opening to fill it. Cover the container opening, discard this volume of fluid, and withdraw an additional volume of fluid as required for the sample volume desired. Cover the container and transfer the fluid to a sample bottle prepared as described in Section 7. Identify the sample bottle as required. The method described in this paragraph is suitable as an alternative method for sample withdrawal from hermetically sealed containers.

Note 5—The method selected shall be accomplished sequentially and as quickly as possible to avert extraneous contamination of the container or sampling apparatus, or both.

9. Precision and Bias

9.1 The reproducibility and repeatability of the results obtained with these sampling techniques depend on the exercise of good workmanship and care. When performing the procedures described in this method, care must be taken to minimize the effects of the working environment and sampling apparatus contamination. Both precision and accuracy are fundamentally limited by the Poisson distribution of sampling, which causes the standard deviation of the number of particles collected to be a function of the square root of the total number of particles collected, in any given category.

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