

Designation: D4306 – 20

## Standard Practice for Aviation Fuel Sample Containers for Tests Affected by Trace Contamination<sup>1</sup>

This standard is issued under the fixed designation D4306; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

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

## 1. Scope\*

1.1 This practice<sup>2</sup> covers the types of and preparation of containers found most suitable for the handling of aviation fuel samples for the determination of critical properties affected by trace contamination.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use. For specific warning statements, see 5.1, 5.2, 5.3, 5.4, and 5.6.

1.4 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

#### 2. Referenced Documents

2.1 ASTM Standards:<sup>3</sup>

- D2624 Test Methods for Electrical Conductivity of Aviation and Distillate Fuels
- D3948 Test Method for Determining Water Separation Characteristics of Aviation Turbine Fuels by Portable Separometer

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4308 Test Method for Electrical Conductivity of Liquid Hydrocarbons by Precision Meter

D5452 Test Method for Particulate Contamination in Aviation Fuels by Laboratory Filtration

2.2 SAE Standard:<sup>4</sup>

MAP1794 Aircraft Recommended Practice, Ball-On-Cylinder (Boc) Aircraft Turbine Fuel Lubricity Tester

## 3. Significance and Use

3.1 General descriptions for the manual sampling of petroleum products are given in Practice D4057. However, a number of aviation fuel properties are established or affected by trace levels of polar or other compounds. Measurement significance therefore requires that the sample containers not add or adsorb any materials. This practice presents types and preparations of sampling containers found satisfactory for the determination of water separation, copper corrosion, electrical conductivity, thermal stability, lubricity, and trace metal content. The choice of construction materials is an important factor, particularly in the case of aviation turbine fuel, where thermal stability can be degraded by the presence of very low concentrations of copper. The use of copper or copper based alloys shall be eliminated from aviation sampling apparatus. An approval procedure for new containers is also given.

3.2 Two properties, particulate contamination and free water content, involve materials easily removed by any sampling container. These properties should be determined by placing the sample directly into the measuring apparatus and not using containers to transport the sample to the measuring equipment.

3.3 Recommendations in this practice provide guidance for immediate use and for storage of samples. Immediate use involves sample storage for periods less than 24 h.

## 4. Apparatus

4.1 Sampling Containers:

4.1.1 Epoxy-Coated Containers:

<sup>&</sup>lt;sup>1</sup> This practice is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.J0.04 on Additives and Electrical Properties.

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<sup>&</sup>lt;sup>2</sup> The detailed data on which this practice is based may be found in SAE Practice MAP1794 and three research reports. Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Reports RR:D02-1169, RR:D02-1142, and RR:D02-1504.

<sup>&</sup>lt;sup>3</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>&</sup>lt;sup>4</sup> Available from Society of Automotive Engineers (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001, http://www.sae.org.

4.1.1.1 While generally superior to other coatings, certain epoxy-coatings evolve plasticizers which can adversely affect critical fuel properties. Because no specification is known to describe a satisfactory epoxy-coating, 6.2 lists an approval procedure which can be used to identify a satisfactory coating.

4.1.1.2 For initial qualification of new container sources, coated cans should be examined closely to assure that the coating covers all inside surfaces. If not, the cans should be considered the same as tin-plated, exterior soldered side seam cans.

4.1.1.3 Epoxy-coated cans are generally considered satisfactory for sampling aviation gasoline.

4.1.2 *Borosilicate (Hard) Glass Bottles*—Amber colored or bottles covered with an opaque material such as aluminum foil are preferred to avoid possible reactions with sunlight.

4.1.3 *Polytetrafluoroethylene (PTFE) Bottles*—Black, carbon-filled bottles avoid possible reactions with sunlight.

4.1.4 Polyethylene Bottles, high-density, linear.

4.1.5 Steel Cans, tin-plated, exterior soldered side seam.

4.1.6 Soda Lime (Soft) Glass Bottles.

4.2 Closures:

4.2.1 Closures with a metallic inside surface are preferred. Closures with the same inside surfaces as suitable containers or PTFE are also suitable.

4.2.2 Where required by shipping regulations such as DOT 17C or 17E the closure should also include a metallic shipping seal.

Note 1—The use of improper or uncleaned closures or shipping seals will destroy all precautions used in selecting and preparing containers. The use of properly selected and cleaned closures or seals is essential.

## 5. Reagents and Materials

5.1 *Acetone*, CP Grade (**Warning**—Extremely flammable. Vapors may cause flash fire). (See Note 2.)

5.2 *Toluene*, CP Grade (**Warning**—Extremely flammable. Vapors may cause flash fire). (See Note 2.) When used to clean containers for conductivity, measure toluene conductivity according to Test Method D2624 or D4308 and use only if conductivity is less than 20 pS/m.

5.3 *Isopropanol*, CP Grade (**Warning**—Extremely flammable. Vapors may cause flash fire). (See Note 2.)

5.4 *Heptane*, CP Grade (**Warning**—Extremely flammable. Vapors may cause flash fire). (See Note 2.)

Note 2—Because these solvents are available at various purity levels, the use of CP grade is required to eliminates possible problems with residual impurities.

5.5 Detergent, heavy duty, water soluble, laboratory type.

5.6 *Jet A or Jet A-1*, used as reference fluid. (Warning—Combustible. Vapor harmful).

5.6.1 Reference fluid for approval testing with Jet A or Jet A-1 fuel is prepared in accordance with Test Method D3948, Appendix X1 on Preparation of Reference Fluid Base, and should have an electrical conductivity of 0.1 to 1.0 by Test Method D4308 (or give a reading of less than 1 according to Test Method D2624) and an MSEP rating of 98-100 by Test Method D3948.

5.6.2 *Compressed Air*, clean, dry, oil free and filtered, may be used to expedite air drying.

#### 6. Preparation of Apparatus

#### 6.1 Introduction:

6.1.1 Experience indicates no single container type to meets all desired requirements including size and cost. Certain container types have been found suitable for some test methods but not for others. Some containers are adequate if the samples are used immediately but are not suitable for sample storage. The procedure therefore designates the containers to be used for each test procedure and describes prior cleaning, if any. A summary of the procedures is found in Table 1. The detailed procedures follow below. However, the possibility that a fuel may contain an unusual contaminant, making a normally satisfactory container unsuitable should not be overlooked.

6.1.2 The largest sample meeting shipping rules, costs, availability, and other practical considerations should always be used to minimize surface effects.

6.1.3 It is not possible to describe some of the container materials by standard specifications or by suitable generic descriptions. Therefore, an approval procedure is outlined in 6.2.

6.1.4 Other sampling details such as sampling taps, labelling, shipping instructions, and so forth will be found in Practice D4057.

## 6.2 Approval Procedure (Stored Samples):

6.2.1 If internally coated the new container should be examined visually for coating integrity in accordance with 4.1.1.2 and closure suitability in accordance with 4.2.1.

6.2.2 Containers should be flushed three times with the container 10 % to 20 % filled with trisolvent (equal volumes of 5.1, 5.2, and 5.3), then three times with heptane. For each flush, the container should be closed and shaken for 1 min and the solvent replaced for the next flush. After the last flush is drained, the container should be air-dried.

6.2.3 Reference fuel as indicated in 5.6 should be used for testing.

6.2.4 The containers should be filled with reference Jet A, or A-1, closed, and stored for at least one month at room temperature. During this period the samples should be shaken strongly at least once a week. At the end of storage the sample should be tested for electrical conductivity and water separation. The final electrical conductivity should be no more than 2 pS/m greater than the original value. The water separation rating should decrease by no more than three MSEP units.

6.2.5 Supplemental testing is necessary if the fuel normally contains additives such as conductivity improvers which may be desorbed. In that case a large additive-containing sample which has been stored for a month or longer to equilibrate additive content should be used as the test fuel. Such fuel should have a conductivity above 50 pS/m if the additive is conductivity improver additive; and the MSEP value should also be determined. After similar storage for at least one month, the final electrical conductivity should not change more than the repeatability limits of Test Method D2624 or D4308,

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TABLE I Summary of Container Recommendations								
Type of Analysis:	Thermal Stability <sup>B</sup>	MSEP	Electrical Conductivity <sup>B</sup>	Lubricity	Trace Metals	Copper Corrosion <sup>B</sup>	Particulate	
Section	6.4	6.5	6.6	6.7	6.8	6.9	6.10	
Hard borosilicate glass								
Immediate use	S	P <sup>c</sup>	Р	S <sup>D</sup>	NR <sup>E</sup>	S	NE	
Storage	NE <sup>F</sup>	Р	Р	S	NR	S	NE	
Reuse	S	S	Р	S	NR	S	NE	
Soft soda lime glass (washed)								
Immediate use	NE	S	S	NE	NR	NE	NE	
Storage	NR	NR	NR	NR	NR	NE	NE	
Reuse	NR	S	S	NR	NR	NR	NE	
Aluminum containers								
Immediate use	NR	NR	NR	NR	NR	NR	NE	
Storage	NR	NR	NR	NR	NR	NR	NE	
Reuse	NR	NR	NR	NR	NR	NR	NE	
Epoxy-lined steel								
Immediate use	Р	Р	Р	Р	NR	S	Р	
Storage	Р	Р	Р	Р	NR	S	Р	
Reuse	Р	Р	Р	Р	NR	S	Р	
Polytetrafluoroethylene								
Immediate use	NE	S	NR	NR	Р	S	NE	
Storage	NE	NE	NR	NR	Р	S	NE	
Reuse	NE	NE	NR	NR	Р	S	NE	
Tin-plate exterior soldered side seam								
(Superclean only)								
Immediate use	S	S	S	S	NR	NR	NE	
Storage	NR	NR	NR	NR	NR	NR	NE	
Reuse	NR	NR	NR	NR	NR	NR	NE	
High-density linear polyethylene								
Immediate use	NR	S	NR	NR	Р	S	NE	
Storage	NR	NR	NR	NR	Р	S	NE	
Reuse	NR	NR	NR	NR	Р	S	NE	

<sup>4</sup> The containers listed in this summary should not be used without consulting the appropriate paragraphs of this practice for detailed advice.

<sup>B</sup> All transparent or translucent containers must be shielded from light by wrapping with opaque material such as aluminum foil, or enclosure in a dark box or cabinet. Amber bottles reduce photochemical effects.

 $^{C}$  P = preferred.

 $^{D}$  S = suitable.

<sup>E</sup> NR = not recommended.

<sup>F</sup>NE = not evaluated but may be suitable.

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whichever method is used to rate the fuel. The final MSEP rating should be within the repeatability limits for the initially obtained value.

6.2.6 The large container fuel sample should preferably be retained in its original container as a reference sample during the storage interval, and retested to determine whether a correction, equal to any change in the reference material, should be applied. Similar testing can be applied for other additives and properties.

## 6.3 Approval Procedure (Immediate Use):

6.3.1 All containers found suitable for storage are suitable for immediate use. The following procedure applies to circumstances where fuel samples will not be retained for longer than 24 h, preferably for shorter times.

6.3.2 The approval procedure is identical to that for storage except that the elapsed time interval between filling containers and testing should be not less than 24 h.

Note 3—Effects due to containers are sometimes variable depending on fuel sample properties especially if additives are present. Evaluations with several fuels or fuel types are helpful to verify conclusions.

6.4 Containers for Thermal Stability Testing:

6.4.1 Epoxy-lined containers in accordance with 4.1.1 are preferred for immediate testing or sample storage. New containers should be flushed in accordance with 6.4.1.2.

6.4.1.1 Used containers should be flushed three times with the container 10 % to 20 % filled with trisolvent (6.2.2) or heptane. For each flush the container should be closed and shaken for 1 min and the solvent replaced for the next flush. After the last flush is drained, the container should be air dried.

6.4.1.2 If the same fuel type containing the same additives is to be resampled, flushing with the product to be sampled is considered adequate preparation. Flushing shall be conducted immediately prior to sample collection and consists of flushing the container 3 times with the sample being collected. For each flush, the container should be 10 % to 20 % filled with new sample, closed, and shaken for a minimum of 5 s and the fuel replaced for the next flush. After the last flush is drained, the container may be filled (allowing safe ullage) for transport to the laboratory for testing.

6.4.2 New borosilicate glass bottles are satisfactory for immediate use if cleaned by rinsing with water, acetone, and air drying. Amber bottles are preferred. Clear bottles must be

shielded from light by wrapping with aluminum foil or enclosure in a dark box or cabinet.

6.4.2.1 Follow the instruction in 6.4.1.1 or 6.4.1.2 for reusing borosilicate bottles. Alternately, borosilicate glass bottles can be similarly flushed in accordance with 6.4.1.2 provided the same fuel type containing the same additives is to be resampled.

6.4.3 New tin-plated cans with exterior soldered side seams should only be used if the container is cleaned and the sample is used immediately.

6.4.3.1 *Cleaning Before Use*—Half fill the container with acetone. Alternatively, a mixture of equal volumes of acetone, toluene, and isopropanol may be used. Replace closure and shake vigorously for 1 min. Drain the solvent and air dry. Fill the container about <sup>1</sup>/<sub>4</sub> full with heptane, replace closure and repeat shaking, draining the solvent and air drying the container.

Note 4—Soldered cans often contain residues of soldering flux or roll-oils on inside surfaces. These materials may have low solubility in hydrocarbons but even at trace levels will adversely affect the properties discussed in this practice. The difficulties of completely removing these contaminants make the use of epoxy-lined containers preferable.

6.4.4 PTFE bottles have not been evaluated but should be satisfactory after cleaning with heptane in accordance with 6.4.1.1.

6.4.5 Other plastic bottles such as high-density linear polyethylene have not been evaluated and are not recommended.

6.5 Containers for Water Separation Testing:

6.5.1 Epoxy-coated containers, whether new or used, are preferred for immediate testing or sample storage. Immediately prior to sample collection, flush the container in accordance with 6.4.1.2.

6.5.1.1 Used containers can be reused after flushing in accordance with 6.4.1.1 or 6.4.1.2.

6.5.2 Borosilicate glass bottles are preferred for immediate use and storage, after rinsing with water, acetone, and air drying. Follow the instruction in 6.4.1.1 or 6.4.1.2 for reusing borosilicate bottles. Alternately, borosilicate glass bottles can be similarly flushed in accordance with 6.4.1.2, provided the same fuel type containing the same additives is to be re-sampled.

6.5.3 Tin-plated, exterior side-seam soldered cans are satisfactory for immediate use after cleaning according to 6.4.1.1 or 6.4.1.2.

6.5.4 PTFE bottles have not been evaluated but should be satisfactory after cleaning with heptane in accordance with 6.4.1.1. For re-cleaning, bottles should be filled with low-conductivity toluene and allowed to soak overnight (more than 16 h). The conductivity of the toluene should not have increased more than 20 pS/m following this soak; if it has, repeat soak. Empty and air dry.

6.5.5 Hard linear polyethylene bottles have been used satisfactorily for immediate use, but it is necessary to evaluate a particular manufacturer's product (bottles and closures) in accordance with 6.2, using Test Method D3948 to evaluate the containers.

6.5.6 Soft glass bottles (soda lime bottles) are satisfactory for some immediate use and storage if they have been soaked

overnight with deionized water, emptied, rinsed with acetone, and dried. Follow 6.4.1.1 and 6.4.1.2 for cleaning or reusing soft glass bottles.

6.5.7 Aluminum containers are not satisfactory.

6.6 Containers for Electrical Conductivity Testing:

6.6.1 Epoxy-coated containers, whether new or used, are preferred for immediate testing or sample storage. Immediately prior to sample collection, flush the container in accordance with 6.4.1.2.

6.6.1.1 Epoxy-coated containers can be reused after cleaning in accordance with 6.4.1.1 or 6.4.1.2.

6.6.2 Borosilicate glass bottles are preferred for immediate use or storage of samples. Prepare containers by rinsing with water, acetone, and air drying, or by rinsing with hot water followed by deionized water and air drying. Follow the instruction in 6.4.1.1 or 6.4.1.2 for cleaning or reusing borosilicate bottles. Alternately, borosilicate glass bottles can be similarly flushed in accordance with 6.4.1.2, provided the same fuel type containing the same additives is to be resampled.

6.6.3 PTFE bottles are not recommended.

6.6.4 Tin-plated, exterior side-seam soldered containers have been satisfactory after cleaning in accordance with 6.4.3.1.

6.6.5 Hard linear polyethylene bottles are not recommended.

6.6.6 Soft glass bottles (soda lime bottles) are satisfactory for immediate use if they have been soaked overnight with deionized water, emptied, rinsed with acetone, and dried. Alternatively, rinse with hot water, then deionized water or acetone, and air dry. Follow 6.4.1.1 and 6.4.1.2 for cleaning or reusing soft glass bottles.

6.6.7 Aluminum containers are not satisfactory.

NOTE 5—Although this practice attempts to minimize container effects wherever possible, electrical conductivity tests should be carried out directly on fuel in tankage or by drawing a sample for immediate testing.

Note 6—Conductivity of fuels is known to change during storage. Results obtained on shipped samples may not be a reliable indicator of the fuel's actual conductivity level. Therefore using shipped samples for conductivity measurements is not recommended. Supplemental testing as discussed in 6.2.5 is useful only to qualify containers. See Test Method D2624.

Note 7—Studies have shown that exposure to sunlight can cause dramatic, permanent loss in the conductivity of fuels containing conductivity improving additives. This has been demonstrated in borosilicate glass and in UV-transparent PTFE bottles, and probably occurs in any UV-transparent container. The following data were obtained for three kerosine jet fuel samples with conductivities of 385 pS/m to 550 pS/m, in 500 mL borosilicate glass or PTFE containers after exposure to Mid-Atlantic summer sunlight. Slower loss is expected from fluorescent lights or other less intense UV sources. Similar effects were noted with various additives. Amber glass bottles are less affected.

Container	Conductiv	Conductivity Loss at Exposure Inte				
	5 min	20 min	95 min			
Amber Glass	0	0 to 16	1 to 47			
Clear Glass	0 to 58	66 to 71	78 to 89			
PTFE	22 to 70	76 to 80	81 to 90			

#### 6.7 Containers for Lubricity Testing:

6.7.1 Closures for bottles, cans, or other types of containers that have wax or plastic coatings in contact with the sample are unsatisfactory for samples to be tested for lubricity.