



Standard Practice for Sampling Cryogenic Aerospace Fluids¹

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

1. Scope

1.1 This practice describes procedures for taking a sample of cryogenic aerospace fluid for analysis.

1.2 *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 requirements prior to use.* For hazard statement, see Section 5.

2. Referenced Documents

2.1 *ASTM Standards:*²

F311 Practice for Processing Aerospace Liquid Samples for Particulate Contamination Analysis Using Membrane Filters

G93 Practice for Cleaning Methods and Cleanliness Levels for Material and Equipment Used in Oxygen-Enriched Environments

G127 Guide for the Selection of Cleaning Agents for Oxygen Systems

3. Summary of Practice

3.1 *Dewar Flask Procedure*—A clean Dewar Flask is used to collect a sample of cryogenic aerospace fluid either from a sampling valve, or poured from a larger Dewar flask used for storage.

3.2 *Cryogenic Sampler Procedure*—The sampler is used to withdraw a small amount of liquefied gas from a large supply. The sampler is allowed to cool until a steady stream of cryogenic liquid exists in the sampler. Once the sampling valves are closed the trapped liquid will convert to a gas and pressurize the sampling vessel.

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

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² 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.

4. Apparatus and Materials

4.1 *Apparatus and Materials Common to Both Procedures:*

4.1.1 *Protective Clothing*, such as an apron, face-shield, and thermal gloves. Coveralls meeting safety requirements for static dissipation, and flammability may be required depending on local safety regulations or operating procedures. White coveralls are recommended while working with cryogenic oxygen as they will show dirt or oil which may react violently with cryogenic oxygen.

4.1.2 *Wash Bottle*, 1–L made of a material compatible with the solvent selected for the cleaning procedure, Teflon FEP is generally acceptable with the solvents and fluids used.

4.1.3 *Solvents*—must be selected to meet the performance, safety, cleanliness, and environmental requirements based on standardized procedures, local and international environmental regulations, and local procedures.

4.1.3.1 *Cleaning Solvents for Cryogenic Oxygen Sampling Equipment*—Solvents for use on oxygen sampling equipment should be selected in accordance with 5.6. Examples of solvents currently used for this purpose include, but are not limited to:

Hydrofluorochlorocarbons, such as Asahiklin AK 225,

Hydrofluorocarbons, such as DuPont Vertrel XF or Vertrel MCA,

Hydrofluoroethers, such as 3M HFE 7100 or HFE 71DE,

Water-based solvents, such as non-ionic detergents.

4.1.3.2 *Cleaning Solvent for Sampling Equipment Used with other Cryogenic Fluids:*

Ethyl acetate shall have no more than 1 $\mu\text{g}/\text{mL}$ residue after evaporation,

Cyclohexane shall have no more than 1 $\mu\text{g}/\text{mL}$ residue after evaporation,

The cleaning solvent will be an azeotrope mixture of ethyl acetate and cyclohexane, filtered in accordance with Practice F311. The mole fraction azeotropic mixture is 0.5286 ethyl acetate and 0.4714 cyclohexane. This is prepared by mixing 503 mL of ethyl acetate with 497 mL of cyclohexane to produce 1 L of cleaning solvent. A fluorocarbon wash bottle should be used with this cleaning solvent.

4.2 *Dewar Flask Procedure:*

4.2.1 *Dewar Flask*, 1-L capacity.

4.2.2 *Dewar Cover*, with provisions for venting.

4.2.3 *Stainless Steel Catch Bucket*—(hydrocarbon clean if used for liquid oxygen sampling).

4.2.4 *Tongs*, stainless steel (Type 300).

4.2.5 *Carrying Case for flask*.

4.2.6 *Polychlorotrifluoroethylene Bag*.

4.3 *Cryogenic Sampler Procedure*:

4.3.1 *Liquid Cryogenic Samplers*³

4.3.2 *Miscellaneous Fittings*, for adapting to the sample point.

4.3.3 *Flexible Hose*, pressure-rated at 3500 kPa (500 psig) gage suitable for the minimum temperature to be encountered and made of materials compatible with the fluid being sampled.

4.3.4 *Handtools*, needed to make the appropriate connections.

5. Hazards

5.1 When sampling cryogenic fluids, care should always be exercised to avoid contact with fluid, equipment, or cold gas to prevent painful frostbite.

5.2 During the chill-down process, caution should also be exercised as gas exiting from the sampling point is under high pressure.

5.3 Certain cryogenic gases such as nitrogen will displace the oxygen in the atmosphere from the ground upwards. Always sample these products in well ventilated areas to prevent asphyxiation.

5.4 Pressure buildup in cryogenic samplers from vaporizing liquid presents a potential hazard.

5.5 When sampling cryogenic oxygen all equipment must be cleaned to the oxygen cleanliness standards outlined in Practice G93 to prevent flash fires or explosions. Rust and dirt particles are known to cause flash fires within oxygen lines when the particles impact valves or bends in the sampling lines. Organic material such as solvents or even the oil from your skin can react violently with cryogenic oxygen leading to an explosion.

5.6 When cleaning cryogenic oxygen sampling equipment, only solvents that have been determined to be compatible with oxygen should be used due to the danger of fire or explosion. Practice G93, Table 5, provides a list of common solvents and Guide G127 provides guidance on the selection of an oxygen system cleaning solvent.

6. Dewar Flask Procedure

6.1 Clean all equipment to meet oxygen system cleanliness standards in accordance with Practice G93.

6.2 Clean the outlet of the sampling port with fluid from the wash bottle in 4.1.2 in accordance with Practice G93.

³ The sole sources of supply of the samplers known to the committee at this time are the following: Type TTU-131/E, Cosmodyne, LLC, 3010 Old Ranch Parkway, Suite 300, Seal Beach, CA 90740 and Model FCS 2001, Cv International, Inc., 2730 Monterey St., Suite 108, Torrance, CA 90503. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

6.3 Open the sampling valve and allow the chill-down to occur until liquid is flowing into the catch bucket.

6.4 Remove the cover from the Dewar flask.

6.5 Hold the flask in a stream of liquid and fill to approximately one-half full. Dump the liquid in the catch bucket.

6.6 Repeat the procedure described in 6.4 until the flask has been sufficiently chilled down.

6.7 Fill the Dewar flask approximately three-fourths full.

6.8 Close the sampling valve.

6.9 Replace the cover on top of the Dewar flask (**Warning**—The cover must be vented to avoid excessive pressure.)

6.10 Place a polychlorotrifluoroethylene or FEP bag on top of the Dewar flask (**Warning**—Do not seal bag, allow to vent.)

6.11 Label the sample.

6.11.1 The label shall identify the sample and remain with the sample.

6.11.2 The label shall include the following information:

6.11.2.1 Location where the sample was taken;

6.11.2.2 Date and time the sample was taken;

6.11.2.3 Fluid that was sampled; and

6.11.2.4 Person who took the sample.

6.12 Analyze the sample as specified in the applicable requirements documents.

7. Cryogenic Sampler Procedure

7.1 The approved procedure for the specified sampler being used and the fluid being sampled shall be followed.

7.2 Clean all equipment to meet oxygen system standards and certify as required by the specific procedure.

7.3 Verify certification for all equipment before using the sampler.

7.4 Inspect sample port for rust, dirt, water, and other contaminants.

7.5 Clean outlet of the sampling port with fluid from the wash bottle in 4.1.2 in accordance with the specified procedure.

7.6 Attach the sample inlet port to the sampling point using fittings as necessary, and flexible hose if necessary.

7.7 Remove the dust cover on the outlet, and open the sampling valve on the system.

7.8 Allow chill-down to occur until a steady stream of liquid is exiting from the outlet of the sampler.

7.9 Open the chamber valve on the sampler for the time specified in the procedure and then close; this allows the precooled reservoir chamber to fill.

7.10 Remove the sampler from the system, and place the dust cover on the inlet and outlet ports.

7.11 Tip the sampler to spill liquid into the outer chamber, the gasification will occur.

7.12 Relieve pressure in the cylinder, if it exceeds 3500 kPa (500 psi) as indicated on the gage of the sampler.



7.13 Label the sample in accordance with the procedure.

7.13.1 The label shall identify the sample and remain with the sample.

7.13.2 The label shall include the following information:

7.13.2.1 Location where the sample was taken;

7.13.2.2 Date and time the sample was taken;

7.13.2.3 Fluid that was sampled; and

7.13.2.4 Person who took the sample.

7.14 Analyze the sample as specified in the applicable requirements documents.

8. Precision and Bias

8.1 Neither the precision nor the bias for this practice has been determined.

9. Keywords

9.1 aerospace fluids; cryogenic fluids; fluid sampling

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