



National Aeronautics and  
Space Administration

**MEASUREMENT  
SYSTEM  
IDENTIFICATION  
METRIC SI (ENGLISH)**

MSFC-STD-3535  
REVISION A  
EFFECTIVE DATE: May 21, 2019

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**George C. Marshall Space Flight Center**  
Marshall Space Flight Center, Alabama 35812

EM01

MSFC TECHNICAL STANDARD

**STANDARD FOR  
PROPELLANTS AND  
PRESSURANTS USED FOR  
TEST AND TEST SUPPORT  
ACTIVITIES AT SSC AND  
MSFC**

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<b>MSFC Technical Standards EM01</b>		
<b>Title: Standard for Propellants and Pressurants Used for Test and Test Support Activities at SSC and MSFC</b>	<b>Document No.: MSFC-STD-3535</b>	<b>Revision: A</b>
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## FOREWORD

This standard was developed by the NASA George C. Marshall Space Flight Center (MSFC) and the NASA John C. Stennis Space Center (SSC) to provide uniform engineering and technical requirements across both Centers for contamination control of propellants and pressurants. This standard has been endorsed and approved for use at the MSFC and the SSC.

This standard establishes core requirements, practices, and methods to assure the quality of propellants and pressurants are maintained. The movement toward joint standardization is driven by past contamination experiences and different technical requirements and practices at both Centers. Non-standard Center practices complicate test interfaces and increase operational risks and costs.

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## **1. SCOPE**

### **1.1 Purpose**

The purpose of this standard is to specify the minimum requirements for the certification and maintenance of propellant and pressurant (P/P) systems used for the purpose of rocket engine testing, test operations, ground support operations, center laboratory operations, and center facility operations.

### **1.2 Applicability**

This standard is applicable to ground-based rocket propulsion test, test support, center sample analysis laboratory, and center operation facilities for P/P requiring system certification. P/P quality provided to the user interface points shall meet or exceed program, test article, center or laboratory requirements. Programs or customers may impose additional requirements. In such cases, process changes and supplemental procedures may be required.

Propellants and pressurants covered in this standard are high purity air (HPA), gaseous nitrogen (GN<sub>2</sub>), liquid nitrogen (LN<sub>2</sub>), gaseous hydrogen (GH<sub>2</sub>), liquid hydrogen (LH<sub>2</sub>), gaseous oxygen (GOX), liquid oxygen (LOX), gaseous helium, and liquid helium, breathing air, gaseous methane, liquid methane, Rocket Propellant-1 (RP1), Rocket Propellant-2 (RP2) and hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>).

This standard applies the following: All mandatory actions (i.e., requirements) are denoted by statements containing the term, “shall.” The terms: “may” or “can” denote discretionary privilege or permission; “should” denotes a good practice and is recommended, but not required; “will” denotes an expected outcome; and “are/is” denotes descriptive material.

## **2. APPLICABLE AND REFERENCE DOCUMENTS**

The documents listed in this section contain provisions that constitute requirements of this standard. The latest issuances of cited documents shall be used unless otherwise approved by the assigned Technical Authority. The applicable documents are accessible via the National Aeronautics and Space Administration (NASA) Technical Standards System at <https://standards.nasa.gov>, directly from the Standards Developing Organizations, or from other document distributors.

### **2.1 Applicable Documents**

MIL-DTL-25576, Detail Specification Propellant, Rocket Grade Kerosene

MIL-PRF-16005, Performance Specification Propellant, Hydrogen Peroxide

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MIL-PRF-25508, Performance Specification Propellant, Oxygen

MIL-PRF-27401, Performance Specification Propellant Pressurizing Agent, Nitrogen

MIL-PRF-27407, Performance Specification Propellant Pressurizing Agent, Helium

MIL-PRF-32207, Performance Specification Propellant, Methane

MPR 8715.1, Marshall Safety, Health, and Environmental (SHE) Program

MSFC-SPEC-164, Cleanliness of Components for Use in Oxygen, Fuel, and Pneumatic Systems

MWI 8715.1, Electrical Safety Program

NPR 1400.1, NASA Directives and Charters Procedural Requirements

NPR 1441.1, NASA Records Management Program Requirements

RPTSTD-8070-0001, Surface Cleanliness Standard of Fluid Systems for Rocket Engine Test Facilities of the NASA Rocket Propulsion Test Program

SPR 1440.1, Records Management Program Requirements

SPR 8715.1, NASA Stennis Space Center Safety and Health Program Requirements

## **2.2 Reference Documents**

ANSI/AIAA G-095, Guide to Safety of Hydrogen and Hydrogen Systems

ASME B 31.1, Power Piping

ASME B 31.3, Process Piping

ASTM MNL36, Safe Use of Oxygen and Oxygen Systems: Handbook for Design, Operation, and Maintenance

CGA G-5.3, Commodity Specification for Hydrogen

CGA G-7.1, Commodity Specification for Air

## **2.3 Order of Precedence**



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When this standard is applied as a requirement or imposed by contract on a program or project, the technical requirements of this standard take precedence, in the case of conflict, over the technical requirements cited in applicable documents or referenced guidance documents.

### 3. DEFINITIONS AND ACRONYMS

#### 3.1 Acronyms

ASME	American Society of Mechanical Engineers
C	Celsius
CO	Carbon Monoxide
CO <sub>2</sub>	Carbon Dioxide
CGA	Compressed Gas Association
CH <sub>4</sub>	Methane
F	Fahrenheit
GH <sub>2</sub>	Gaseous Hydrogen
GHe	Gaseous Helium
GOX	Gaseous Oxygen
GN <sub>2</sub>	Gaseous Nitrogen
He	Helium
H <sub>2</sub>	Hydrogen
H <sub>2</sub> O	Water
H <sub>2</sub> O <sub>2</sub>	Hydrogen Peroxide
HPA	High Purity Air
IPA	Isopropyl Alcohol
kPa	Kilopascals
L	Liters
LEL	Lower Explosion Limit
LH <sub>2</sub>	Liquid Hydrogen
LN <sub>2</sub>	Liquid Nitrogen
LOX	Liquid Oxygen
m	Meter
mg	milligram
MIL	Military
MPR	Marshall Procedural Requirement
MSFC	George C. Marshall Space Flight Center
MWI	Marshall Work Instruction
NASA	National Aeronautics and Space Administration
NPR	NASA Procedural Requirement
N <sub>2</sub>	Nitrogen
O <sub>2</sub>	Oxygen
OJT	On-the-Job Training
POC	Point of Contact
P/P	Propellant / Pressurant

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PPE	Personal Protective Equipment
ppm	Parts per million
psia	pounds per square inch absolute
psig	Pounds per Square Inch Gauge
RP1	Rocket Propellant-1
RP2	Rocket Propellant-2
scf	Standard Cubic Foot
scfm	Standard Cubic Foot per Minute
SPR	Stennis Procedural Requirement
UIP	User Interface Point

### 3.2 Definitions

Analytical Sample: Sample taken during a maintenance activity or after decertification of a system that is for engineering-based analysis only. The information will be used to assist in preparing a system for certification.

Buy Point: A sample point selected and used to verify results obtained by other evaluation personnel.

Certification: A process in which a written record is generated that demonstrates that the requirements of this standard have been met and verified.

Certification Sample: Any sample taken by certified personnel on a system that is currently certified or taken on a system under maintenance or a de-certified system for the purpose of certification.

Certified System: A certified system is comprised of clean components and subsystems initially verified through a multiple sample process as meeting the requirements of this standard. Only certified P/P's shall be used in this system which requires periodic sampling to maintain certification.

Condensable Hydrocarbons: Oily mist or droplets containing carbon and hydrogen components and other elements in a gas stream. These are expressed as the mass of oil (e.g., milligrams) per a specified volume of gas. (See Appendix C.)

Contaminant: Any material that could reduce the intended purity of a pressurant/propellant thereby allowing chemical reaction or mechanical interference with a cleaned component, system, or end item.

Dew Point: The temperature at which a gas becomes saturated with water vapor and condensation begins (usually atmospheric pressure).

Fiber: A flexible structure having a length-to-width ratio of 10-to-1 or greater.

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Hydrocarbon: Any compound containing carbon and hydrogen.

Micron: Dimension of length equal to 0.001 millimeter (0.0000394 inch).

Moisture: The residual liquid/gas/water measured in parts-per-million (ppm) by volume.

Non-Certified System: A utility or facility system not intended to be certified.

Particulate: Matter with observable length, width, and thickness usually measured in micrometers. This definition includes fibers.

Propellants/Pressurants (P/P): All cryogenic and non-cryogenic fluid products such as liquid or gaseous oxygen, hydrogen, helium, nitrogen, air, methane, hydrogen peroxide, and rocket propellants that are used to support combustion for propulsion, pressurize propulsion systems, purge systems, simulate high-vacuum space conditions, or create inert atmospheres.

Receiving Tank: Tanks used for receiving commodity at gas generation or storage facility.

Run Tank: Tank that feeds directly to test article.

Sample: A selected portion of material, usually from a larger amount, taken or provided for inspection, certification or verification.

Sample Point: The designated point in a system or component from which a representative sample may be taken.

Sampling Port: A location in the pressurant/propellant system configured to readily allow taking a representative portion of the fluid for evaluation.

Specific Impurities: The amount of predetermined contaminants contained within a fluid sample. Typically, the contaminants of interest are those that are more likely to be introduced into the facility's fluid system (moisture, total hydrocarbons, nitrogen, etc.). Also, specific impurity can be described as a subset of all impurities that have a high risk for freezing in the cryogenic liquid and could cause damage to sensitive hardware.

Storage Tank: (e.g., Barges) Tanks used to transfer commodity to run tanks, trailers, or test articles.

System Maintenance: A process whereby a closed propellant/pressurant system is opened for the purpose of preventive or corrective maintenance or system modification. This does not normally include connecting and disconnecting of test articles and equipment.

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Total Hydrocarbon: For the purpose of this document, the total hydrocarbon content will be gaseous hydrocarbons expressed as methane and defined by any compound containing carbon and hydrogen.

Total Impurities: The amount of all contaminants contained in a fluid. Typically, this term applies to the maximum allowable amount of contaminants in a sample taken from a vendor delivery.

User Interface Point: The point between the propellant/pressurant system operator (supplier) and the end user (customer) which is determined by contract, agreement or specification requirements.

Verification: Testing performed to determine whether previously certified gas system continues to satisfy specification requirements.

## **4. GENERAL REQUIREMENTS**

### **4.1 Responsibilities**

Responsibilities for the requirements and applications of this standard are as specified in the text herein. All users of this standard shall comply with its requirements and verify the latest version prior to use.

The NASA SSC Engineering and Test Directorate and the MSFC Engineering Directorate are responsible for approval of the content of this standard and have final authority for its interpretation. Concurrence from both Centers is required for any revision/clarification to this document.

NASA and contractor personnel have various responsibilities for planning, approval, supervising, monitoring, notifying, and documenting, as specified throughout this standard.

### **4.2 Certification Sampling**

Certification sampling requirements are identified in this standard. Once the process of certification or recertification of a system has begun, no analytical or other samples shall be allowed.

Samples shall be taken at predetermined gas or liquid sample points downstream of filters, regulators, valves, and any other equipment normally installed in a line.

#### **4.2.1 Common Sampling Team**

The common sampling team will consist of a dedicated group of personnel with common processes, equipment, and training/certifications to perform the assigned functions. Team

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members may consist of personnel from a single contractor or any combination of government and personnel from different contractors.

In order to achieve consistent and reliable results, all personnel on the team shall be trained or certified to the same requirements.

A NASA or contractor point of contact (POC) will be identified as the common sampling team lead. Some responsibilities of the team lead include:

- a. Assuring failed sample results are communicated to the appropriate parties.
- b. Prioritizing the sampling schedule when there are conflicts.
- c. Serving as the focal point for technical issues related to sampling.
- d. Verifying common sampling team personnel maintain updated training.
- e. Performing quarterly reviews of the sampling database.

See Appendix A for minimum training requirements.

#### **4.2.2 Common Sampling Equipment**

The common sampling team shall control the use of all approved portable sampling panels and related equipment, while maintaining records pertaining to preventive and corrective maintenance.

Note: Fixed pressure-reducing or sampling panels are the responsibility of the system operator. The sample analysis laboratory shall control the use of all sampling containers and related equipment and maintain records pertaining to preventive and corrective maintenance.

#### **4.2.3 Sampling Schedule**

The common sampling team shall create and maintain a sampling schedule for taking periodic gas and liquid samples. This sampling schedule should be created with inputs from fluid system operators, customers, and the sample analysis laboratory. The schedule should identify each designated sampling port, along with the fluid to be sampled and sampling frequency.

P/P system sampling frequency shall meet the requirements of Section 5.2.

#### **4.2.4 Sample Database**

The common sampling team shall create and maintain a sampling database to document gas and liquid samples. All samples taken, analytical, periodic (see Table I) and system certification, shall

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be maintained in the database, but shall be distinctively identified to prevent confusion. At a minimum, a quarterly data review shall be performed on the periodic data to verify overall system integrity.

#### **4.2.5 Sample Points**

Sample points are designated points in a pressurant/propellant system or component from which a representative sample may be taken and configured to provide representative system conditions for sampling operations. These discreet points are typically valves and are identified by each site and documented on applicable system drawings.

#### **4.2.6 Procedures**

The common sampling team shall work to approved procedures for all sampling and analysis. Acceptable procedures shall include technical steps required to safely perform and maintain accuracy and process control. Sampling practices/techniques noted in Appendix B shall be incorporated into approved procedures.

The owners/operators of the system to be sampled shall be responsible for preparing the system prior to any sampling activity.

#### **4.2.7 Safety**

Personnel involved in sampling processes specified herein shall be required to adhere to safety requirements as specified by their Center.

#### **4.2.8 Environmental**

The mitigation of possible impacts to the environment takes precedence over function, cost, or expediency. Environmental impacts and concerns should be reported to the appropriate Center's Environmental Officer.

#### **4.2.9 Training and Personnel Certification**

Personnel involved in sampling processes shall be instructed to exercise precautionary measures necessary to reduce introduction of contaminants. Minimum classroom training requirements are noted in Appendix A. On-the-job training (OJT) required to meet certification requirements will be documented at each given center.

#### **4.2.10 Quality Assurance**

Sampling processes shall be validated to demonstrate the ability of the process to achieve required results. The processes shall be approved by the cognizant sampling personnel management and other NASA/contractor organizations as required. Process procedures shall describe methods and

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techniques, which control critical parameters. Approved changes to the procedures shall be documented. Monitoring and measuring equipment shall be controlled per the Center's requirements to ensure accuracy.

## **5. GENERAL PROPELLANT/PRESSURANT SAMPLING REQUIREMENTS**

### **5.1 Propellant/Pressurant System Sampling Frequency Rationale**

Sampling frequency was determined based on such considerations as use of the system, commodity, adverse trends, customer requirements and risk.

A quarterly sampling frequency does not provide enough data points to establish identifiable trends within gas piping systems. Data gathered from sampling operations at SSC since August 2007 has provided adequate data points for trending capability. MSFC weekly sampling data supports the proposed frequency requirements.

Based on the benefits of a common sampling team, the samples supplied to the analysis lab will be consistent. Samples will be analyzed for all parameters as specified at the User Interface Points (UIP's). The database for sample results will be used to identify trends that could impact the integrity of site-wide systems.

### **5.2 Propellant/Pressurant System Sampling Frequency**

When used for testing, drying and preservation, gaseous systems that are used daily shall be tested with a frequency adequate to ensure the maintenance of system integrity as defined in Table I below. If intermittent sampling is elected due to extended system non-use, the system shall be sampled and verified prior to use.

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**TABLE I. PERIODIC SAMPLING REQUIREMENTS**

<b>Propellant / Pressurant</b>	<b>Gaseous Generation Facilities</b>	<b>Delivery Piping Systems/ User Interface Points</b>	<b>Trailers</b>
High Purity Air	Every 2 Weeks	Monthly	Each Every 6 Months*
Gaseous Nitrogen	Every 2 Weeks	Monthly	Each Every 6 Months*
Liquid Nitrogen	See Additional Requirement 4	Not Applicable	Each Every 3 Months
Gaseous Hydrogen	Every 2 Weeks	Monthly	Each Every 6 Months*
Liquid Hydrogen	See Additional Requirement 4	Upon Request	Upon Request
Gaseous Oxygen	Upon Request	Upon Request	Each Every 6 Months
Liquid Oxygen	See Additional Requirement 4	Not Applicable	Each Every 3 Months
Gaseous Helium	Every 2 Weeks	Monthly	Each Every 6 Months*
Liquid Helium	See Additional Requirement 4	Not Applicable	Not Applicable
Gaseous Methane	Every 2 Weeks	Monthly	Each Every 6 Months*
Liquid Methane	Every 2 Weeks	Monthly	Each Every 6 Months*
Rocket Propellant-1	Every 2 Weeks	Monthly	Each Every 6 Months*
Rocket Propellant-2	Every 2 Weeks	Monthly	Each Every 6 Months*
Hydrogen Peroxide	Every 2 Weeks	Monthly	Each Every 6 Months*

Additional Requirements:

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1. All samples are for in-service systems or components only. Specific sampling requirements for out-of-service or stand-by systems or components should be in accordance with program-specific plans for those items (\* SSC gas trailers shall be sampled per load.).
2. Liquid Hydrogen and Liquid Oxygen barges shall be sampled prior to delivery.
3. Sampling downstream of the user interface point shall occur as requested by user.
4. Cryogenic tanks shall be sampled at the discretion of the operator or per agreement between the operator and the customer.

### **5.3 Gaseous Sampling Requirements**

Gaseous P/P's typically include oxygen, nitrogen, hydrogen, helium, methane, and the high purity air system (missile grade air). These P/P's are sampled at designated sample points to verify the sample analysis requirements of Table II in Section 9.

Note: Designated sample points may include truck tube banks, sampling points near maintenance or system break points, and pressure vessels.

### **5.4 Particulate Sampling in Gaseous Propellants/Pressurants**

Particulate sampling equipment shall be verified/certified clean prior to use. High-pressure or low-pressure filter holders may be used depending on the systems sampled. The filter holder will contain a filter disc (0.45 micron).

### **5.5 Cryogenic Liquid Sampling Requirements**

Liquid P/P systems are sampled at designated sample points in Section 5.3 to verify the sample analysis requirements of Table II in Section 9.

Only Cryogenic Samplers with a proven history of accurate sampling are allowed. Currently, the Cosmodyne™ brand sampler is utilized by both MFSC and SSC. The sampler shall be verified/certified clean for specific P/P, LOX, LH<sub>2</sub> or LN<sub>2</sub>, prior to liquid sampling.

Insulated flex hoses, cleaned to the specific level for the P/P, will be used to connect the Cryogenic Samplers to the sample point.

### **5.6 Sampling for Condensable Hydrocarbons in Propellant/Pressurant Systems**

Gaseous P/P sampling shall be performed on the nitrogen, helium, methane, and high purity air systems to verify that these systems are free of condensable hydrocarbons.

Sampling for condensable hydrocarbons is only performed on fluids that are pressurized to system operating pressures by compressors that contain hydrocarbon lubricants (i.e., oils). These sample points must be near the point where the gas is introduced into the distribution system.

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See Appendix C for condensable hydrocarbon sample analysis procedures.

## **6.0 PROPELLANT/PRESSURANT SAMPLING EQUIPMENT**

### **6.1 General**

Portable sample panels shall receive proper preventative maintenance on a scheduled basis and corrective maintenance when required. The design of portable sample panels shall incorporate applicable standards for fail-safe use and personnel safety, along with considerations for ease of operations.

Prior to use, hand tools, materials, and equipment that may come into contact with significant surfaces or the service media shall be verified/certified and identified as cleaned, maintained clean or field cleaned to the required system or component level where it is being used.

### **6.2 Detailed Sampling Equipment**

#### **6.2.1 Cryogenic Liquid Sampling Equipment**

All equipment identified as verified/certified “clean” shall be cleaned, maintained clean or field cleaned as appropriate to the system level where it is being used. Only Cryogenic Samplers with a proven history of accurate sampling (such as Cosmodyne CS-4.4 or TTU-131) are allowed. An insulated clean flex hose (Cosmodyne™ part number 2601191-1 or equivalent) shall be used.

#### **6.2.2 Gas Sampling Equipment**

A gas sampling cylinder (hoke, watermelon, or equivalent) rated for the appropriate maximum pressure shall be used for gas samples.

#### **6.2.3 Particulate Sampling Equipment**

Particulate sampling shall be performed with an appropriately pressure-rated filter holder containing a verified/certified filter disc (0.45 micron).

#### **6.2.4 Moisture Sampling Equipment**

A moisture meter approved by the gas analysis laboratory is required for moisture content determinations in the field.

#### **6.2.5 Condensable Hydrocarbon Sampling Equipment**

A condensable hydrocarbon sampler is a filter holder that contains a sintered porous disc that has a 10-micron nominal pore size.

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### 6.3 Propellant/Pressurant Sampling Panels

To assist in the collection of high-pressure gas samples, a pre-fabricated “sample panel” may be used in lieu of individual components. These pressure-reducing sample panels may be permanently installed at the sample location or be portable in nature. See Section 6.3.1 for advantages and disadvantages.

The panels shall be designed per the ASME B31.3 and in a manner to prevent over pressurization, damage to hardware, and injury to personnel. The hand-operated pressure regulator should have a flow rate as low as possible to reduce the size of the relief valve, or burst disk protecting the sample bottle. The range of the calibrated gauge shall be such that the normal operating pressure of the panel reads approximately mid-range of the gauge.

If the panels are used to obtain particulate data, the hand-operated regulators must be those without built-in filters. The panel must also have either a calibrated flow-meter or a calibrated orifice, or it must be controlled by an approved written procedure detailing flow rate requirements.

Panels shall include labeled inlets and outlets, pressure limits, P/P’s for which it is suitable and appropriate valve identifiers and cautions.

Note: Personnel collecting samples must follow all appropriate safety practices related to the fluid being sampled.

#### 6.3.1 Advantages and Disadvantages of Types of Sample Panels

The permanent installed pressure-reducing panels have the advantage of eliminating possible cross-contamination of the sample taken from previous locations and the individual components can possibly be smaller in size and cheaper. The disadvantage of the permanent panels is that one is required for each sample point, and therefore may be cost prohibitive if several sample points are required.

The portable sample panels have the advantage in that “one panel serves all,” or a few panels will serve all locations or different gases. The disadvantages are the possible cross-contamination of samples and if panels are designed for different pressures, a safety issue arises when using a panel not suited for the pressure being sampled.

#### 6.3.2 Sample Container Preparation

##### Sample Container Preparation

All new sample containers shall be verified/certified clean prior to initial use.

The following processes are acceptable for preparing cylinders to measure the maximum allowable contaminants in P/P systems for routine samples:

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- a. Vacuum – using a pump system, the vessel shall be pulled to a vacuum of 50 microns or less to assure there is no residual gas left in the vessel; or
- b. Purge/Vent – purge each vessel by flowing sample gas from the sample point through the sample vessel for a minimum of three minutes to ensure that all residual gas has been removed.

## **7. SAMPLE ANALYSIS REQUIREMENTS**

### **7.1 General Propellant/Pressurant Sample Analysis**

Samples taken may be gas and/or liquid, but the impurities in all P/P samples shall be analyzed in a gaseous state. All analyses shall be performed to approved procedures. Particulate samples shall be evaluated in either a verified/certified clean room or at verified/certified clean work stations (flow benches).

### **7.2 Propellant/Pressurant Sample Analytical Methods**

Unless otherwise stated, samples shall be analyzed using examples in Appendix C. Calibration gas standards are required to calibrate analytical instruments used for sample analysis.

### **7.3 Hydrocarbon Content**

Hydrocarbon Content performed on both gaseous and cryogenic liquid samples shall be expressed as methane. A hydrocarbon analyzer capable of detecting one part per million of methane shall be used to determine hydrocarbon content. The equipment shall be operated in accordance with the manufacturer's instructions.

### **7.4 Condensable Hydrocarbon**

Condensable hydrocarbon content shall be determined in accordance with Appendix C.

### **7.5 Moisture Content**

Moisture content shall be determined using a moisture analyzer capable of detecting one part per million of water. The equipment shall be operated in accordance with the manufacturer's instructions.

The following approved test equipment or equivalent items are required for moisture content determinations:

- a. Electrolytic hygrometer
- b. Aluminum Oxide Hygrometer
- c. Tunable Diode Laser Absorption Spectroscopy

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## **7.6 Sample Analysis Laboratory Identification of Failed Samples**

When a sample does not meet requirements as specified by the requester, the sample analysis laboratory shall immediately notify the requester and the sampling team POC that the sample has failed. Should the requestor not be available, the requestor's management shall be notified immediately. See Section 10.1 for the notification process for failed samples.

## **8. CERTIFICATION REQUIREMENTS**

### **8.1 Initial Certification**

During the initial certification process, if any sample fails to meet the requirements of Table II (Section 9) the initial certification process shall be restarted. The initial certification process shall be repeated until the system is certified. (Note: The nonconformance process will not take effect until the system has been certified.)

Two consecutive samples at each gas sample point in the system, taken 8 to 72 hours apart, shall comply with the user interface point requirements identified in Table II (Section 9).

Two additional samples taken consecutively at 5-to-10 day intervals from each gas sample point shall comply with the user interface point requirements identified in Table II (Section 9).

Example: S1= 0 hrs, S2= 8 to 72 hrs, S3= 5 to 10 days, S4 = S3 + 5 to 10 days

#### **8.1.1 Certification Requirements for User Interface Points (Usage Points)**

UIP's shall be certified in accordance with the initial certification requirements and periodically verified in accordance with requirements in Table I.

### **8.2 Systems Under Maintenance**

To maintain certification while under maintenance, approved contamination control procedures must be followed. Sampling may or may not be required to verify continued certification.

Examples when sampling is not required:

- a. The removal and replacement of a relief valve, pressure indicator, vent valves, or other instrumentation at the end of a line.
- b. The change-out of an in-line component with an end-to-end purge.

If contamination control procedures are not followed, sampling is required. Engineering can request sampling at any time.

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### 8.3 Recertification Requirements for Propellant/Pressurant Systems

When recertification is required, two consecutive samples at each affected sample point in the system shall be taken 8 to 72 hours apart and both shall comply with the UIP requirements identified in Table II (Section 9). If one sample fails to meet the requirements, the process shall be repeated until the system is recertified. (Note: The nonconformance process will not take effect until the system has been certified.)

Example: Sample 1 = 0 hours, Sample 2 = 8 to 72 hours

### 8.4 Certification Requirements for Cylinder Supplied Gas

A gas system may consist of a cylinder, or a bank of cylinders, equipped with one or more user interface points. Note: For the purposes of this standard, k bottles are not considered gas cylinders.

a. Initial certification shall require a sample obtained from the user interface point downstream from the cylinder gas system. This sample must meet the contamination level user interface point requirements specified in Table II. The certification sample shall be taken with all bottle valves in the open position.

b. Periodic certification is not required; however, following cylinder replacement or maintenance, a recertification sample shall meet the requirements of Table II.

## 9. PROCUREMENT AND DELIVERY REQUIREMENTS / MAXIMUM ALLOWABLE IMPURITIES

Liquids used in testing and gases used in the testing, drying and preservation of components, assemblies and support/test equipment shall meet all of the user interface point requirements specified in Table II. In the tables below, “total impurities” refers to all impurities whereas “specific impurities” refers to a subset of only the impurities of interest to the user.

**TABLE II. PROPELLANT/PRESSURANT REQUIREMENTS**

PROPELLANT/PRESSURANT PARAMETER	REQUIREMENTS	
	PROCUREMENT	USER INTERFACE POINTS
<b>HELIUM, GAS (He) Ref MIL-PRF-27407 (Grade A)</b>		
Purity	99.995% He (min.)	99.994% He (min.)
Total Impurities (max ppm)	50	N/A
Specific Impurities (max ppm)	N/A	60
H <sub>2</sub> (max ppm)	1	N/A
N <sub>2</sub> and Argon (receiving only)	14 ppm maximum	N/A

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<b>PROPELLANT/PRESSURANT</b>	<b>REQUIREMENTS</b>	
<b>PARAMETER</b>	<b>PROCUREMENT</b>	<b>USER INTERFACE POINTS</b>
O <sub>2</sub> and Argon (max ppm)	3	10
N <sub>2</sub> (max ppm)	N/A	40
H <sub>2</sub> O (max ppm)	9 (-61.1°C Dew Point)	9 (-61.1°C Dew Point)
Volatile Hydrocarbon (max ppm)**	5	5
Neon (max ppm)	23	N/A
CO (max ppm)	1	N/A
CO <sub>2</sub> (max ppm)	1	N/A
Particulate (gas)	N/A	30μ-100μ: 25; >100μ: 0
Particulate (liquid)	N/A	N/A
Condensable Hydrocarbons	N/A	0.25 mgs (max)
<b>METHANE, GAS (CH<sub>4</sub>) (Grade C) Ref MIL-PRF-32207</b>		
Purity (min % volume)	99.97	N/A
Water (max ppm volume)	0.5	N/A
Oxygen (max ppm volume)	1	N/A
Nitrogen (max ppm volume)	100	N/A
Carbon Dioxide (max ppm volume)	50	N/A
Other Gas Impurities(max ppm vol)	125	N/A
Ethane (max ppm volume)	100	N/A
Propane (max ppm volume)	100	N/A
Other Volatile Hydrocarbons	1 ppm volume maximum	N/A
Total Volatile Sulfur	0.1 ppm volume maximum	N/A
Non-volatile Residue & Particulates	1 mg/L maximum	N/A
<b>HYDROGEN, GAS OR LIQUID (H<sub>2</sub>) Ref CGA G-5.3 QLV "A"</b>		
Purity	99.995% H <sub>2</sub> (min.)	99.993% H <sub>2</sub> (min)
Total Impurities (max ppm)	50	N/A
Total Liquid Impurities	50	N/A
Specific Impurities (max ppm)	N/A	70
Parahydrogen	95.0% (liquid H <sub>2</sub> only)	N/A
Selected Impurities: N <sub>2</sub> , Hydrocarbon**	9 (-61.1°C Dew Point)	N/A
H <sub>2</sub> O	N/A	9 (-61.1°C Dew Point)
O <sub>2</sub> and Argon (max ppm)	1	5
N <sub>2</sub> (max ppm)	N/A	15
Volatile Hydrocarbons (max ppm)	N/A	5
He (max ppm)	39	45
CO <sub>2</sub> plus CO (max ppm)	1	N/A
Particulate (gas)	N/A	N/A

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PROPELLANT/PRESSURANT	REQUIREMENTS	
PARAMETER	PROCUREMENT	USER INTERFACE POINTS
Particulate (liquid)	Fill, 10 $\mu$ nom. 40 $\mu$ abs	User Defined Level
<b>NITROGEN, GAS OR LIQUID (N<sub>2</sub>) Ref MIL-PRF-27401 (Grade B)</b>		
Purity	99.99 % N <sub>2</sub> (min.)	99.989 % N <sub>2</sub> (min.)
Total Impurities (max ppm)	100	N/A
Specific Impurities (max ppm)	N/A	111
O <sub>2</sub>	50	100
CO (max ppm)	5	N/A
CO <sub>2</sub> (max ppm)	5	N/A
Argon (max ppm)	20	N/A
Volatile Hydrocarbons ** (max ppm)	5	5
H <sub>2</sub> O (max ppm)	6 *** (-64.0°C Dew Point)	6.0 *** (-64.0°C Dew Point)
Particulate (gas)	N/A	30 $\mu$ -100 $\mu$ : 25; >100 $\mu$ : 0
Particulate (liquid)	1 mg/liter	N/A
Condensable Hydrocarbons	N/A	0.25 mgs (max.)
<b>OXYGEN, GAS (GOX) OR LIQUID (LOX) Ref MIL-PRF-25508 (Grade A)</b>		
Purity	99.6% O <sub>2</sub> (min.)	99.6% O <sub>2</sub> (min.)
Total Impurities (max ppm)	4000	N/A
H <sub>2</sub> O (max ppm)	3 (-69.0°C Dew Point)	20 (-55.2°C Dew Point)
Volatile Hydrocarbons ** (max ppm)	50	50
Particulate (gas)	N/A	30 $\mu$ -100 $\mu$ : 25; >100 $\mu$ : 0
Particulate (liquid)	1 mg/liter	N/A
<b>HIGH PURITY AIR (HPA)</b>		
O <sub>2</sub>	N/A	19.5-23.5%
H <sub>2</sub> O (max ppm)	N/A	24 (-53.9°C Dew Point)
Volatile Hydrocarbons ** (max ppm)	N/A	15
Particulate (gas)	N/A	30 $\mu$ -100 $\mu$ : 25; >100 $\mu$ : 0
Particulate (liquid)	N/A	N/A
Condensable Hydrocarbons	N/A	0.25 mgs (max.)
<b>ALCOHOL (IPA)</b>		
Purity	87.4 – 87.9	N/A
H <sub>2</sub> O (% by weight)	Balance	N/A
NVR (% by weight)	0.002	N/A

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PROPELLANT/PRESSURANT	REQUIREMENTS	
PARAMETER	PROCUREMENT	USER INTERFACE POINTS
<b>BREATHING AIR Ref CGA G-7.1 (Grade D)</b>		
Oxygen, balance is predominantly nitrogen	N/A	19.5-23.5%
Carbon dioxide (max ppm)	N/A	1000
Carbon monoxide (max ppm)	N/A	10
H <sub>2</sub> O	N/A	24 ppm
Volatile Hydrocarbons as CH <sub>4</sub>	N/A	15 ppm
Condensable Hydrocarbons	N/A	5 mg/m <sup>3</sup>
<b>METHANE, LIQUID (CH<sub>4</sub>) (Grade C) Ref MIL-PRF-32207</b>		
Purity (min % volume)	99.97	N/A
Water (max ppm volume)	0.5	N/A
Oxygen (max ppm volume)	1	N/A
Nitrogen (max ppm volume)	100	N/A
Carbon Dioxide (max ppm volume)	50	N/A
Other Gas Impurities(max ppm vol)	125	N/A
Ethane (max ppm volume)	100	N/A
Propane (max ppm volume)	100	N/A
Other Volatile Hydrocarbons	1 ppm volume maximum	N/A
Total Volatile Sulfur	0.1 ppm volume maximum	N/A
<b>ROCKET PROPELLANT-1 (RP1) Ref MIL-DTL-25576</b>		
Sulfur, Total (max mg/Kg)	30	N/A
Mercaptan-sulfur (max mg/Kg)	3	N/A
Aromatics (max % volume)	5.0	N/A
Olefins (max % volume)	2.0	N/A
Particulates (max mg/L)	1.0	N/A
<b>ROCKET PROPELLANT-2 (RP2) Ref MIL-DTL-25576</b>		
Sulfur, Total (max mg/Kg)	0.1	N/A
Mercaptan-sulfur (max mg/Kg)	3	N/A
Aromatics (max % volume)	5.0	N/A
Olefins (max % volume)	1.0	N/A
Particulates (max mg/L)	1.0	N/A
<b>HYDROGEN PEROXIDE (H<sub>2</sub> O<sub>2</sub>) (Type 98) Ref MIL-PRF-16005</b>		
Purity (minimum % by weight)	98.0	N/A
Chlorine (max mg/Kg)	0.5	N/A
Nitrate (NO <sub>3</sub> ) (max mg/Kg)	5.0	N/A

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<b>PROPELLANT/PRESSURANT</b>	<b>REQUIREMENTS</b>	
<b>PARAMETER</b>	<b>PROCUREMENT</b>	<b>USER INTERFACE POINTS</b>
Phosphate (PO <sub>4</sub> ) (max mg/Kg)	0.2	N/A
Sulfate (SO <sub>4</sub> ) (max mg/Kg)	0.5	N/A
Ammonium (NH <sub>4</sub> ) (max mg/Kg)	3.0	N/A
Stability (max % loss active oxygen)	2	N/A
Residue (max mg/Kg)	20	N/A
Total Carbon (max mg/L)	40	N/A
Aluminum (max mg/L)	0.35	N/A
Tin (acceptable range mg/L)	1.0 – 4.0	N/A
Chromium (max mg/L)	0.03	N/A
Lead (max mg/L)	0.03	N/A
Manganese (max mg/L)	0.03	N/A
Iron (max mg/L)	0.03	N/A
Copper (max mg/L)	0.03	N/A
Nickel (max mg/L)	0.03	N/A
Antimony (max mg/L)	0.03	N/A
Arsenic (max mg/L)	0.03	N/A
Gold (max mg/L)	0.03	N/A
Zinc (max mg/L)	0.03	N/A
Titanium (max mg/L)	0.03	N/A

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\* Unless otherwise specified, requirement levels are total ppm by volume.

\*\* Total volatile (gaseous) hydrocarbons expressed as methane (CH<sub>4</sub>).

\*\*\* The maximum H<sub>2</sub>O content is 6 ppm verified from the delivery manifest. This is less than the 11.5 ppm allowed in MIL-PRF-27401; however, historical data documents receipt of product within the lower limit.

**Notes:**

- 1) High purity air should not be used as breathing air in a confined area for long periods of time. This air is typically very dry and could cause respiratory problems. If you use high purity air through a self-contained breathing apparatus, you are required to follow guidelines and requirements for grade (L) in document CGA G-7.1.
- 2) Procurements are certified by the vendor and the certification results are sent to the procuring organization. These results shall satisfy the procurement requirements unless analytical verification tests are requested by engineering.
- 3) Purity is determined by subtracting specific or total impurities from 100% or by direct analysis. (For H<sub>2</sub>, N<sub>2</sub> and He, purity is normally calculated by subtracting the specific impurities from 100 %.)
- 4) A filter with no more than a 10-micrometer nominal and 40-micrometer absolute rating shall be installed between the manufacturer's plant system (e.g., manifold) used to fill the gas or liquid containers for delivery.

## 10. QUALITY ASSURANCE REQUIREMENTS

The NASA and/or contractor quality assurance (QA) organization shall determine the appropriate level of monitoring, surveillance, or inspection based on such considerations as customer requirements, contract requirements, and positive or negative trends. NASA QA may request assistance from, or delegate this responsibility to, the contractor QA. NASA QA or their designee may perform scheduled or unscheduled audits of specified buy points verifying the process. At the discretion of each Center's Chief Engineer, Safety and Mission Assurance (S&MA) Manager, and, for certified samples, NASA QA or their designated representatives may be required to be present during sampling operations for specified buy points (verify, witness, or inspect) in the approved procedures. They may also be required to verify sample analysis results at a specified buy point in approved procedures.

### 10.1 Nonconformance Reporting

NASA and Contractor QA shall be notified of failed sample results per the applicable Center procedure. The Center's nonconformance documentation, investigation, and disposition process shall be implemented.

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The safety and critically of the test hardware for a given system should be considered when deciding what type of nonconformance reporting process should be used. At a minimum, a “flash” report shall be sent to concerned/involved parties documenting the nonconformance. The reporting process will begin after a maximum of two failed samples have been declared by the lab.

The two samples taken for analysis can be taken at the same time using two different sampling containers at each sample point, a primary and a backup where one sample container is for analysis, and the second for analysis only if the first one fails.

Note: The purpose of the backup sample is to verify sampling technique associated with a failed primary sample.

A second approach would be to take two independent samples at different times or days. Unless specific gas analysis requirements are submitted to the lab, the analysis lab will verify that a sample has passed or failed based on the requirements within this standard.

## **10.2 Waivers and Deviations**

Any substitutions, deviations, or modifications to this standard shall be made only with the approval of the cognizant authority for waivers and deviations.

## **11. RECORDS AND REPORTS**

### **11.1 Records**

Logs or databases shall be used to document all sampling at each sample point or user interface point in order to monitor trends and/or failures. The data from these logs or databases will be maintained by the sampling team and may be used to determine the adequacy of the sampling frequency. All records generated for equipment maintenance shall be maintained per NPR 1441.1, NASA Records Retention Schedules.

### **11.2 Reports**

Data gathered as a result of this standard shall be documented by the performing organization as designated by NASA Quality Assurance, and shall include pertinent information recorded in logs or databases. All test results shall be made available upon request.

Forms shall be the latest edition unless otherwise specified.

For Quality Records, refer to the Master Records Index.

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## APPENDIX A. MINIMUM TRAINING REQUIREMENTS

Note: On-the-job-training (OJT) will be Center-specific.

### A.1 Sampling Certification

Common sampling team lead will be responsible for verifying the certification of team members. Each person is responsible for taking site-specific classes that are required by the Safety, Health and Environmental (SHE) Group and any classes required by employer.

### A.2 Minimum Requirements for Sampling Certification

Annual Physical

LO/TO --- (Lock Out/Tag Out) Training

O2/LEL --- (Oxygen/Lower Explosion Limits) Meter Training

Hearing Conservation

High Pressure Safety Training

Cryogenic Safety Training

### A.3 Minimum Requirements for OJT for Certification in Fluid Sampling

OJT requirements for certifications are developed into specific OJT training packages with the necessary lesson plans, instructional material, qualification tests, and OJT training required to perform a specific critical task or hazardous operation. Details of these OJT requirements for specific certifications will be maintained by the individual's Supervisor/Team Lead. These packages must be approved by the respective site Certification Boards.

Demonstrate knowledge of tests and monitoring equipment.

Review and become knowledgeable of procedures and work documents.

Buddy System Training.

System Cleanliness Training.

Area Access Control Training (as required).

Gas Sampling Training.

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Cryogenic Sampling Training.

Pass a written exam covering the contents of sampling fluid systems.

The common sampling team's certification will be recognized for sampling at either Center as long as there is at least one representative from the specific site accompanying the team.

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## APPENDIX B. COMMON SAMPLE PRACTICES/TECHNIQUES

### B.1 System Interfaces with Different Cleanliness Levels

When fluid systems are designed or modified, cleanliness breaks shall be established to enable connecting of systems that have different cleanliness levels.

1. Use a filter for any cleanliness level breaks that have different particulate levels and the same nonvolatile residue (NVR) level or no NVR level.
2. Use dual check valves for any cleanliness level breaks that have different NVR requirements. Add a filter if downstream particulate requirements are more stringent than upstream requirements. An upstream system must have the same or higher NVR cleanliness level requirements than the downstream system.

### B.2 Gas Samples

1. Perform sampling using detailed step-by-step procedures that includes all tooling, equipment and personal protective equipment (PPE) requirements as well as Cautions, Warnings, etc.
2. Purge-out/flow through sample port prior to connecting sample panel.
3. Prior to sampling, purge the sample point valve and regulator/flex hose at the rate of 283 liters per minute (plus or minus 10) (10.0 (plus or minus 0.35) scfm) or at an audible purge for a minimum of 2 minutes.
4. Avoid the use of ball flow meters for gas sample panel; instead, use calibrated orifices and flow for specified time in order to enhance safety and process consistency.
5. Ensure all equipment is relief protected (gauges, etc.)
6. Access to sampling area should be restricted to involved personnel.
7. No welding, heat or spark producing operations are allowed in an area where H<sub>2</sub> and O<sub>2</sub> sampling is to take place.
8. Maintain slight positive purge during panel (or Millipore) hook-up and disconnect of inert gas samples.
9. Prior to collecting GH<sub>2</sub> samples, purge the panel and sampling lines to remove air and moisture from the sampling system.
10. Monitor by "sniff check" sampling panel, hoses, container and other equipment during GH<sub>2</sub> sample taking operations.

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11. Avoid sampling during rain or high moisture/fog conditions.

12. Particle level determination for gas systems shall be made using a minimum 850 liter (30 scf) sample. Gas flow rate through the sampler, while accumulating the sample, shall be 283 liters  $\pm$  10 liters per minute ( $10.0 \pm 0.35$  scfm) or maximum flow rate for systems not capable of this flow rate. The sample size for gases from a cylinder or system of cylinders shall be 283 liters (10 scf) minimum. A positive flow of at least 14.2 liters per minute (0.5 scfm) shall be maintained during the installation and removal of the sampler.

Note: Although particles should be imbedded in the filter disc surface, handle the filter holder so as not to jar particles from the filter. Hold the filter holder with the inlet port up.

### **B.3 Cryogenic Liquid Samples**

Once the Cryogenic Sampler is connected to UIP of the vessel and the system is ready for sampling, the instrument must be chilled properly. Open the sample valve, flow through the Cryogenic Sampler inlet and outlet port and chill the instrument until a steady stream of liquid is coming out the outlet port. This could be 5 to 10 minutes. On liquid hydrogen samples, the vent hose should show signs of liquid air. To guarantee liquid has made it to the sampler, this could take up to 60 minutes depending on the location of the sample point. Once the Cryogenic Sampler is adequately chilled, open the sample-collecting valve on the instrument for 1 to 2 minutes and close. Verify between 1,380 kPa-gauge to 2,760 kPa-gauge (200 psig to 400 psig) pressure on the Cryogenic Sampler gauge for adequate sample.



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## **APPENDIX C. CONDENSABLE HYDROCARBON SAMPLE ANALYSIS**

### **C.1 Scope**

This specification establishes the procedure for the determination of entrained, condensable hydrocarbons in high-pressure, greater than 690 kPa (100 psi) gas supply systems. The procedures required by this specification shall not be used for the sampling of combustible gases.

### **C.2 Detail Requirements**

Gas Sample Volume – During sample collection, the minimum total volume sampled shall be 850 liters (30 scf). The minimum total volume of gas sampled from systems of cylinders shall be 283 liters (10 scf). Volume requirements are met when sampling collection procedure per Table III is followed.

### **C.3 Procedure**

#### **C.3.1 Sample Collection**

- a. Before connecting sampler, apply purge to the sample line.
- b. Remove the sampler from the sealed bag. A positive flow shall be maintained during installation and removal of the sampler. Remove the protective AN plug from the inlet and attach the sampler directly to the standard AN fitting at the console or sampling port.

Note: Do not add pressure line extensions between sampling port and the system being sampled.

- c. Open the system valve to achieve a pressure drop reading above 3,450 kPa (500 psi) and sample for 1.2 minutes. If a pressure drop of 3,450 kPa (500 psi) cannot be obtained, refer to Table III for the required sampling time. No sampling shall be conducted below a pressure drop of 1,380 kPa (200 psi).

#### **C.3.2 Condensable Hydrocarbons**

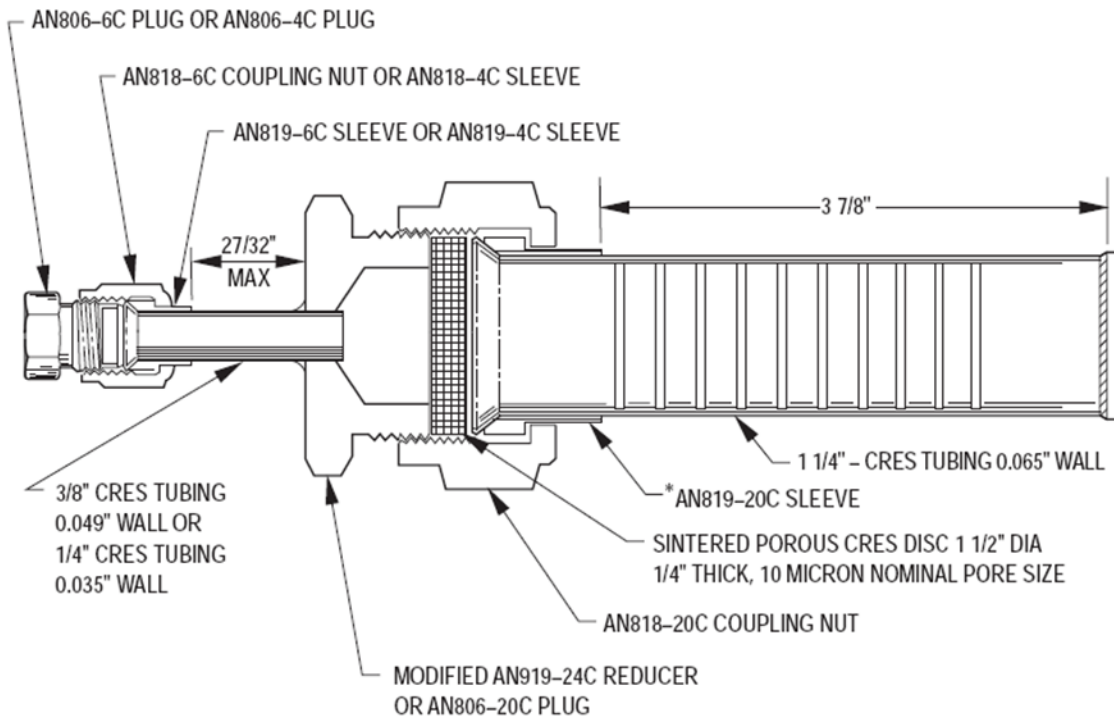
A sample can be obtained at the source by passing the sampling gas through a precision cleaned gas sampling device as shown in Figure 1. Volume requirements are met when sampling collection per Table III is followed.

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**Figure 1. Gas Sampler for Condensable Hydrocarbons**

**TABLE III. SAMPLING TIME**

<b>Nitrogen<sup>1</sup></b>	
Pressure Drop kPa (psi) <sup>3</sup>	Sampling time (min)
1379 – 3446 (200 – 499)	2.5
> 3447 (500)	1.2
<b>Helium<sup>2</sup></b>	
Pressure Drop kPa (psi) <sup>3</sup>	Sampling time (min)
1379 – 2061 (200 – 299)	2.5
> 2062 (300)	1.2

Notes:

1. N<sub>2</sub> is used as the sampling gas for oxidizer and nitrogen systems
2. Helium is used as the sampling gas for helium and fuel systems and can be used for sampling oxidizers
3. Pressure drop is defined as the pressure reading on the gauge at the sampling location when the sampler is connected and the valve is open.

a. Remove the sampler from the sample port and reinstall the AN plug in the inlet of the sampler and insert sampler into Teflon or Aclar bag that complies with MSFC-SPEC-164, "Cleanliness of Components for Use in Oxygen, Fuel, and Pneumatic Systems" or with NASA RTPSTD-8070-

CHECK THE MASTER LIST - VERIFY THAT THIS IS THE CORRECT VERSION BEFORE USE

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0001, "Surface Cleanliness Standard of Fluid Systems for Rocket Engine Test Facilities of the NASA Rocket Propulsion Test Program". Pass the sampling gas through the sampling device under the conditions listed in Table III.

b. Submit to the analysis lab, disassemble the sampler and place the sintered stainless steel disc in Gooch crucible or a 47mm diameter filter holder/filtering apparatus.

c. Flush 200 ml (6.8 oz) of a halogenated solvent through the sintered disc. Repeat twice using the same solvent.

d. Determine the condensable hydrocarbons using the infrared procedure described in Section C.5.

#### **C.4 Infrared Spectrometric Transmission Method for Condensable Hydrocarbons Analysis**

##### **C.4.1 General**

This method involves evaporating an aliquot of solvent (AK225g, etc.), picking up the residue in tetrachloroethylene and analyzing the solution with an infrared spectrometer.

##### **C.4.2 Equipment**

- a. Fourier Transform Infrared (FTIR) Spectrometer
- b. Quartz cell, 20 mm path length (10 mm optional)
- c. Pharmaceutical grade mineral oil
- d. Tetrachloroethylene, spectrometric grade
- e. Temperature controlled oven (optional)

##### **C.4.3 Calibration**

- a. Before using this method, the FTIR spectrometer shall be initially calibrated.
- b. Follow manufacturer's instructions for internal optical alignment if required, and optimal energy throughput. Instrument parameters (number of scans, wave number resolution, gain ranging radius, etc.) should be adjusted as needed to optimize results.
- c. Using tetrachloroethylene in a 20 mm quartz cell, perform a background (single beam) scan daily or before use between  $3200\text{ cm}^{-1}$  and  $2600\text{ cm}^{-1}$ . The background spectrum will be used to develop absorbance spectra for the calibration standards in (d.)

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d. Calibrate the FTIR spectrometer by preparing mineral oil standards in tetrachloroethylene. A minimum of five calibration points shall be obtained. The final calibration concentrations shall range from 0.01 mg/ml to an upper limit of 0.3 mg/ml. Progressing from the lowest calibration standard to the highest standard, scan the various hydrocarbon blends in the 3200  $\text{cm}^{-1}$  and 2600  $\text{cm}^{-1}$  range using a 20 mm quartz cell. The quartz cell should be flushed twice with tetrachloroethylene and a minimum of two times using a standard of the next highest concentration level when changing standards. Intermediate standards should bracket the analytical range of interest.

e. Use the spectra from (d.) above to compute the least squares regression calibration coefficients. The correlation coefficient for the linear regression or quadratic model should be 0.990 or better. Either peak intensity or area can be modeled. The calibration curve should not be forced to go through zero.

### C.5 Infrared Analysis Procedure

a. Perform a background scan daily with tetrachloroethylene or before use between 3200  $\text{cm}^{-1}$  and 2600  $\text{cm}^{-1}$ . The single beam background spectra will be used to develop absorbance spectra for analyzing samples.

b. Following the initial calibration and the background scan (a), analyze check standards daily or before sample analysis. The check standards shall represent 0.01 mg/ml and 0.2 mg/ml. The check standards (mineral oil in tetrachloroethylene) should read within  $\pm 10\%$ .

c. Filter the solvent sample (e.g., AK225g,) through a 10 micron pore size or less filter paper or membrane, compatible with the fluids being tested. Collect the filtered solvent into a cleaned container.

d. Evaporate the sample or a representative aliquot of the solvent sample to near dryness (3 to 5 ml) using a steam bath or rotary evaporator or a thermostatically controlled hot plate. Remove the beaker before dryness and place in an oven at a maximum temperature 110 °C and allow it to dry or with forced air (draft from the fume hood) flowing over the sample or with a nitrogen purge.

e. Allow the beaker or flask to cool and add 7 ml of tetrachloroethylene to the residue.

f. Briefly swirl the solvent over the residue and transfer the solution to a 20 mm quartz cell. Use care during transfer to avoid loss of the sample due to spilling or overfilling of the cell.

g. Record a sample infrared absorption spectrum with a frequency of 3200 to 2600  $\text{cm}^{-1}$  using the same cell path length that was used to develop the calibration curve.

h. Determine the number of milligrams of hydrocarbon in the sample by using the least squares regression calibration curve. If the sample concentration is greater than the highest calibration

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standard, a dilution of the sample with tetrachloroethylene shall be made and the appropriate dilution factor applied.

$$\text{NVR} = (\text{MS}) (\text{Vs})$$

Where:

MS = FTIR measured sample concentration (e.g., mg/ml)

Vs = Tetrachloroethylene volume used to reconstitute the sample residue (e.g., 7 ml)

NVR = mgs of hydrocarbon

i. Perform a hydrocarbon blank on the filtered control solvent (steps c. through h.) and subtract the results from the value obtained for the sample (h).

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## **APPENDIX D. GENERAL GUIDELINES FOR PRESSURE-PURGE INERTING**

The intention of this appendix is to give general guidance when pressure-purging gaseous systems. Engineering evaluation and procedural documentation is required before any inerting process can be performed.

Considerations when generating procedures:

1. The geometry of the piping system or vessel needs to be evaluated. This includes the location of the inlet and outlet ports, etc.
2. The physical properties of the commodity and purge gas need to be evaluated; this includes the densities of the individual gases. Will the purge gas “sweep” the commodity out of the system, or will the two gasses be required to mix before the system can be inerted. If mixing is desired, the velocity of the purge gas should be considered. Turbulent flow will lend to better mixing.

Below is a formula that can be used to calculate the approximate number of purge cycles required to inert a system.

Calculate the number of cycles necessary to obtain the desired maximum concentration of hydrogen using the formula below. Note that NASA/SSC is continuing analysis of the pressure-purge inerting process and the following analysis is believed to be safety conservative for pressure-purge cycle determination.

1. Vent the system to a minimum pressure level (preferably 14-21 kPa (2-3 psig)) if the system is at a higher pressure. Selection of the minimum pressure limit is to minimize purge gas requirements and ensure positive system pressure is maintained throughout the pressure-purge inerting process. For large high pressure gas distribution systems, venting to 50 psi or lower has been found effective.
2. Pressurize the system to a chosen maximum pressure with purge gas. Nominally, use three times the minimum purge pressure. Low maximum pressure minimizes the purge gas requirements for pressure-purge inerting. A maximum pressure (Pmax) of 3x the minimum pressure (Pmin) level provides an effective balance between inert gas usage and the operations associated with the required number of pressure cycles.

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3. Repeat steps 1 and 2 for the calculated number of cycles and verify desired gas concentration by gas sampling.

Expression to Calculate the Number of Cycles:

$$\left(\frac{P_{\min}}{P_{\max}}\right)^n = \phi$$

Where: n = # of pressure cycles assuming isothermal Pressure-Purge process

P<sub>min</sub> = Minimum pressure of cycle, psia

P<sub>max</sub> = Maximum pressure of cycle, psia (using inert gas)

φ = Desired final concentration of gas purged from system (%/100)

The above equation is a satisfactory cycle estimate if isothermal conditions are maintained during the pressure-purge inerting process. However, it underestimates the required number of pressure-purge cycles under isentropic conditions (e.g., if rapid pressure-purge cycling is employed such that the system gas temperature is not allowed to stabilize between the P<sub>min</sub>-P<sub>max</sub>-P<sub>min</sub> pressure levels). For isentropic conditions, it is recommended additional cycles be added to the isothermal pressure-purge cycle relation above.

For an isentropic pressure-purge process:

$$\begin{aligned} \# \text{ cycles} &= n + 2 && \text{for } 0.01 < \phi < 0.1 && (1\% - 10\% \text{ concentrations}) \\ &= n + 3 && \text{for } \phi < 0.01 && (< 1\% \text{ concentration}) \end{aligned}$$

#### **EXAMPLE: (CALCULATION USING FORMULAS)**

An empty, ambient temperature LH<sub>2</sub> tank needs to be inerted to 0.8% H<sub>2</sub> concentration to enable tank entry. The tank initial pressure is 3 psi (100% GH<sub>2</sub>).

Assume:

1. The tank will be vented to 3 psig [17.7 pounds per square inch absolute (psia)] for the pressure-purge cycling process.
2. A maximum tank purge pressure of 38.4 psig (53.1 psia).
3. The pressure-purge process is isothermal.

(Note: The average ambient pressure at SSC is 14.7 psia.)

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Calculation:

$$\frac{(P_{\min})^n}{(P_{\max})} = \phi$$

$$\frac{(17.7)^n}{(53.1)} = (.00800)$$

n = 4.4 or 5 cycles required

if the pressure-purge cycling were performed rapidly, such that the tank pressurant temperature varied between Pmin and Pmax and the process assumed to be isentropic, the number of cycles needed would be 8 (= n+3 since 0.008 = φ < 0.01).