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Space systems — Fluid characteristics, sampling and test methods —

Part 3:
Nitrogen

Systèmes spatiaux — Caractéristiques, échantillonnage et méthodes d'essai des fluides —

Partie 3: Azote

Reference number
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Foreword

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 15859-3 was prepared by Technical Committee ISO/TC 20, *Aircraft and space vehicles*, Subcommittee SC 14, *Space systems and operations*.

ISO 15859 consists of the following parts, under the general title *Space systems — Fluid characteristics, sampling and test methods*:

- *Part 1: Oxygen*
- *Part 2: Hydrogen*
- *Part 3: Nitrogen*
- *Part 4: Helium*
- *Part 5: Nitrogen tetroxide propellants*
- *Part 6: Monomethylhydrazine propellant*
- *Part 7: Hydrazine propellant*
- *Part 8: Kerosine propellant*
- *Part 9: Argon*
- *Part 10: Water*
- *Part 11: Ammonia*
- *Part 12: Carbon dioxide*
- *Part 13: Breathing air*

Introduction

Fluid operations at a spaceport or launch site may involve a number of operators and supplier/customer interfaces, from the fluid production plant to the delivery to the launch vehicle or spacecraft. The purpose of ISO 15859 is to establish uniform requirements for the components, sampling and test methods of fluids used in the servicing of launch vehicles, spacecraft and ground support equipment. The fluid composition limits specified are intended to define the purity and impurity limits of the fluid for loading into the launch vehicle or spacecraft. The fluid sampling and test methods are intended to be applied by any operator. The fluid sampling and test methods are acceptable methods for verification of the fluid composition limits.

INTERNATIONAL STANDARD ISO 15859-3:2004(E)

Space systems — Fluid characteristics, sampling and test methods —

Part 3: Nitrogen

1 Scope

This part of ISO 15859 specifies limits for the composition of nitrogen and establishes the sampling and test requirements applicable for the verification of nitrogen composition.

This part of ISO 15859 is applicable to nitrogen, used in both flight hardware and ground facilities, systems and equipment, of the following types and grades of nitrogen.

- Type I: gaseous
    - Grade A: purging/pressurizing,
    - Grade B: crew breathing,
    - Grade C,
    - Grade F,
    - Grade J;
- Type II: liquid
    - Grade A: purging/pressurizing,
    - Grade B: crew breathing,
    - Grade C,
    - Grade F.

This part of ISO 15859 is applicable to influents only within the specified limits herein.

This part of ISO 15859 is applicable to any sampling operation required to ensure that, when the fluid enters the launch vehicle or spacecraft, the fluid composition complies with the limits provided hereafter or with any technical specification agreed to for a particular use.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 9000, *Quality management systems — Fundamentals and vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 9000 and the following apply.

3.1

direct method

method of measuring fluid purity by direct means as opposed to the indirect method

NOTE A measurement device or analyser can be used as a direct means for measuring the fluid purity.

3.2

indirect method

method of measuring fluid purity by indirect means, which consists in measuring the total volume fraction or mass fraction (in %) of aggregate impurities and subtracting this total from 100 %

3.3

total hydrocarbon content (as methane)

single carbon atom equivalent

3.4

verification test

analysis performed on the fluid in the container, or a sample thereof, which is representative of the supply, permitting the verification of fluid composition limits

4 Chemical composition

Unless otherwise provided in an applicable technical specification, the composition of nitrogen delivered to the flight vehicle interface shall be in accordance with the limits given in Table 1 when tested in accordance with the applicable test methods.

Table 1 — Composition limits

Component		Limits				
		Type I and Type II (gaseous and liquid)				Type I
		Grade A	Grade B	Grade C	Grade F	Grade J
Nitrogen	Volume fraction, %, min. [a]	99,5	[f]	99,995	99,9	99,999
Water	µl/l, max.	26,3	11,5	5,7	15	10
Total hydrocarbons as methane (CH_4)	µl/l, max.	58,3	5,0[g]	5,0	25	—
Oxygen	µl/l, max.	5 000	50	20	1 000	5
Hydrogen	µl/l, max.	[e]	[e]	0,5	[e]	—
Argon[b]	µl/l, max.	[e]	20	2,0	[e]	—
Carbon dioxide[b]	µl/l, max.	[e]	5,0	5,0	[e]	—
Carbon monoxide[b]	µl/l, max.	[e]	5,0	5,0	[e]	—
Aromatic hydrocarbons as benzene[b]	µl/l, max.	—	0,5	—	—	—
Halogenated hydrocarbons[b]	µl/l, max.	—	1,0	—	—	—
Chlorinated hydrocarbons[b]	µl/l, max.	—	0,1	—	—	—
Nitrous oxide[b]	µl/l, max.	—	1,0	—	—	—
Odour		—	None	—	—	—
Particulate matter[c]	mg/l, max.	—	—	1,0	1,0	—
Total impurities[d]	µl/l, max.	5 000	100	50	1 000	10

[a] Nitrogen (in percent) includes trace quantities of neon, helium, and small amounts of argon.

[b] If required by use.

[c] Applies only to Type II (liquid) nitrogen.

[d] Sum of all percentages of water, hydrocarbons, oxygen, hydrogen, and if applicable, carbon monoxide, carbon dioxide, and argon.

[e] No limit for this grade.

[f] By indirect method 99,99 % or by direct method 95,0 %.

[g] For environmental-control-and-life-support-system (ECLSS) ground test only.

5 Procurement

Nitrogen types and grades specified in Clause 1 should be procured in accordance with an applicable national standard.

6 Fluid sampling

CAUTION — Gaseous nitrogen is an asphyxiant. Human contact with liquid nitrogen will result in severe injury. Care should be taken in the handling and storage of liquid nitrogen to prevent contact with the human body. Care should also be taken to prevent high concentrations of gaseous nitrogen in confined spaces.

6.1 Plan

In order to ensure that the fluid composition complies with the limits specified in this part of ISO 15859, a fluid sampling plan should be established by all the involved operators, from the production to the space vehicle interface, and approved by the final user. Sampling activities and test methods shall comply with all safety regulations and rules applicable to that task. This plan shall specify

- the sampling points,
- the sampling procedures,
- the sampling frequency,
- the sample size,
- the number of samples,
- the test methods, and
- the responsibilities of any involved operator.

6.2 Responsibility for sampling

Unless otherwise provided in an applicable technical specification, the nitrogen delivered to the flight vehicle interface shall be sampled and verified by the supplier responsible for providing the nitrogen to the flight vehicle. The supplier may use his/her or any other resources suitable for the performance of the verification tests specified herein unless otherwise directed by the customer.

6.3 Sampling points

Unless otherwise specified, sampling shall be conducted at the fluid storage site or the flight vehicle interface.

6.4 Sampling frequency

Sampling shall be performed annually or in accordance with a time agreed upon by the supplier and the customer.

6.5 Sample size

The quantity in a single sample container shall be sufficient to perform the analysis for the limiting characteristics. If a single sample does not contain a sufficient quantity to perform all of the analyses for the required quality verification test, additional samples shall be taken under similar conditions.

6.6 Number of samples

The number of samples shall be in accordance with one of the following:

a) one sample per storage container;

b) any number of samples agreed upon by the supplier and the customer.

6.7 Storage container

Unless otherwise provided by the applicable sampling plan, the fluid storage container shall not be refilled after the sample is taken.

6.8 Gaseous samples

Gaseous samples shall be a typical specimen from the gaseous nitrogen supply. Samples shall be obtained in accordance with one of the following.

a) By filling the sample container and storage containers at the same time, on the same manifold, and under the same conditions and with the same procedure.

b) By withdrawing a sample from the supply container through a suitable connection into the sample container. No pressure regulator shall be used between the supply and the sample containers. (Suitable valves are permissible.) For safety reasons, the sample container and sampling system shall have a rated service pressure at least equal to the pressure in the supply container.

c) By connecting the container being sampled directly to the analytical equipment using suitable pressure regulation to prevent over-pressurizing this equipment.

6.9 Liquid samples (vaporized)

Vaporized liquid samples shall be a typical specimen from the liquid nitrogen supply. Samples shall be obtained by flowing liquid from the supply container into or through a suitable container in which a representative liquid sample is collected and then completely vaporized.

6.10 Rejection

When any sample of the fluid tested in accordance with Clause 7 of this part of ISO 15859 fails to conform to the requirements specified herein, the fluid represented by the sample shall be rejected. Disposal of the rejected fluid shall be specified by the customer.

7 Test methods

7.1 General

The supplier will ensure, by standard practice, the quality level of nitrogen. If required, alternate test methods are described in 7.3 to 7.14. Other test methods not listed in this part of ISO 15859 are acceptable if agreed upon between the supplier and the customer.

These tests are a single analysis or a series of analyses performed on the fluid to ensure the reliability of the storage facility to supply the required quality level. This can be verified by analysis of representative samples of the fluid from the facility at appropriate intervals as agreed upon between supplier and the customer. Tests may be performed by the supplier or by a laboratory agreed upon between the supplier and the customer.

The analytical requirements for the tests shall include the determination of all limiting characteristics of nitrogen.

7.2 Parameters of analysis

The parameters for analytical techniques contained in 7.3 to 7.14 are the following:

a) purity and impurity contents shall be expressed as a percentage by volume (volume fraction, %) unless otherwise noted;

b) calibration gas standards containing the applicable gaseous components may be required to calibrate the analytical instruments used to determine the limiting characteristic levels of the fluid;

c) if required by the customer, the accuracy of the measuring equipment used in preparing these standards shall be traceable to an established institute for standards;

d) analytical equipment shall be operated in accordance with the manufacturer's instructions.

7.3 Nitrogen purity

The nitrogen purity shall be determined by one of the following procedures.

a) By determining the quantity of the aggregate impurities using the methods in 7.4 to 7.14. The purity of nitrogen is the value obtained when this quantity of aggregate impurities, expressed as a volume fraction (%), is subtracted from 100.

b) By determining the aggregate of all impurities by mass spectrometer. The percentage of nitrogen is the value obtained when the aggregate, expressed as a volume fraction (%), is subtracted from 100.

c) By a gas chromatography method which uses a 5Å zeolite column and measures the peak height versus retention time.

d) By a mass spectrometer method which measures nitrogen at an atomic mass unit (amu) of 28. Carbon monoxide also has an amu of 28 but it can be determined by the method described under 7.10.

7.4 Water content

For liquid nitrogen, the water content is determined by sampling (see 6.9). Online measurement is only possible for gaseous nitrogen. For gaseous or vaporized nitrogen, the water content shall be determined by one of the following procedures.

a) By a dew-point analyser in which the temperature of a viewed surface is measured at the time water first begins to form.

b) By a piezoelectric sorption hygrometer, of which the accuracy of analysis shall be ± 0,1 cm^3/m^3 or 5 % of the reading, whichever is greater.

c) By a metal-oxide-capacitor-equipped analyser within a range which is no greater than 10 times the specific maximum water content.

d) By an electrolytic hygrometer having an indicator graduated in cubic centimetres per cubic metre within a range which is not greater than 10 times the specified maximum water content.

7.5 Total hydrocarbon content (THC)

The total (volatile) hydrocarbon content (as methane) shall be determined by one of the following procedures.

a) By a flame-ionization-type analyser. The analyser shall be calibrated at appropriate intervals by the use of calibration gas standards. The range used shall be no greater than 10 times the specified maximum total hydrocarbon content expressed as methane.

b) By a gas-cell-equipped infrared analyser. The analyser shall be calibrated at appropriate intervals by use of calibration gas standards at a wavelength of approximately 3,5 µm (the characteristic absorption wavelength for C-H stretching). The analyser shall be operated so that its sensitivity for methane is at least 10 % of the specified maximum total hydrocarbon contents.

c) By a gas chromatograph in accordance with 7.8 a).

7.6 Oxygen content

The oxygen content shall be determined by one of the following procedures.

a) By an electrochemical-type oxygen analyser containing a solid or an aqueous electrolyte. The analyser shall be calibrated at appropriate intervals by use of calibration gas standards or integrally in accordance with Faraday's Law. The range used should be no greater than 10 times the specified maximum oxygen content.

b) By a tungsten filament lamp-type analyser. The analyser shall be checked for proper operation at appropriate intervals by the use of a calibration gas standard.

c) By an analyser in which oxygen reacts to form a compound which is subsequently measured. The analyser shall be calibrated at appropriate intervals by the use of calibration standards. The range used shall be no greater than 10 times the specified maximum oxygen content.

d) By a gas chromatography method such as that described under 7.8 a).

e) By a mass spectrometer. The mass spectrometer shall be operated so that its sensitivity is at least 10 % of the specified oxygen content.

f) By an apparatus employing a detector tube filled with a colour-reactive chemical. The degree of accuracy is dependent on the precision of the measurements and the analytical bias of the tube.

g) By a heated zirconium-oxide-type analyser or by a paramagnetic-type analyser. The analyser shall be calibrated at appropriate intervals by the use of calibration gas standards. The range used shall be no greater than 10 times the specified maximum oxygen content.

7.7 Hydrogen content

The hydrogen content shall be determined by one of the following procedures.

a) By a gas chromatograph in accordance with 7.8 a).

b) By a mass spectrometer. The mass spectrometer shall be operated so that its sensitivity is at least 10 % of the specified maximum amount of the component.

c) By an analyser in which hydrogen reacts to form a compound which is subsequently measured. The analyser shall be calibrated at appropriate intervals by the use of calibration gas standards. The range used shall be no greater than 10 times the specified maximum hydrogen content.

7.8 Argon content

The argon content shall be determined by one of the following procedures.

a) By a gas chromatograph. This method may be used not only for argon, helium, and neon determination but also for the determination of any other limiting characteristic gaseous components. See Annex A. The analyser shall be capable of separating and detecting the component with a sensitivity of 10 % of the specified maximum amount of the component. Appropriate impurity concentrating techniques may be used to attain the sensitivity. The analyser shall be calibrated at appropriate intervals by the use of calibration gas standards.

b) By a mass spectrometer. The mass spectrometer shall be operated so that its sensitivity is at least 10 % of the specified maximum amount of the component.

7.9 Carbon dioxide content

The carbon dioxide content shall be determined by one of the following procedures.

a) By a gas-cell-equipped infrared analyser. The analyser shall be calibrated at appropriate intervals by the use of calibration gas standards at a wavelength of approximately 4,3 µm. The analyser shall be operated so its sensitivity for carbon dioxide is at least 10 % of the specified maximum carbon dioxide content.

b) By a gas chromatography method such as that described in 7.8 a). The technique utilized shall be specific for the separation and analysis of carbon dioxide.

c) By an apparatus employing a detector tube filled with a colour-reactive chemical. The degree of accuracy is dependent on the precision of the measurements and the analytical bias of the tube.

d) By a mass spectrometer. The mass spectrometer shall be operated so that its sensitivity is at least 10 % of the specified maximum amount of the component.

e) By a catalytic methanizer gas chromatography method such as that described under 7.8 a).

7.10 Carbon monoxide content

The carbon monoxide content shall be determined by one of the following procedures.

a) By an apparatus employing a detector tube filled with a colour-reactive chemical. The degree of accuracy is dependent on the precision of the measurements and analytical bias of the tube.

b) By a gas-cell-equipped infrared analyser. The analyser shall be calibrated at appropriate intervals by the use of calibration gas standards at approximately 4,6 µm. The analyser shall be operated so that its sensitivity for carbon monoxide is at least 10 % of the specified maximum carbon monoxide content.

c) By an analyser in which carbon monoxide reacts to form a compound which is subsequently measured. The analyser shall be calibrated at appropriate intervals by the use of calibration standards. The range used shall be no greater than 10 times the specified maximum carbon monoxide content.

d) By a gas chromatography method such as that described under 7.8 a). The technique utilized shall be specific for separation and analysis of carbon monoxide.

e) By a catalytic methanizer gas chromatography method such as that described under 7.8 a).

7.11 Aromatic, chlorinated, or halogenated hydrocarbon content

The aromatic, chlorinated, or halogenated hydrocarbon content shall be determined by one of the following procedures.

a) By a gas chromatography method such as described under 7.8 a).

b) By a mass spectrometer. The mass spectrometer shall be operated so that its sensitivity is at least 10 % of the specified maximum amount of the component.

c) By a gas chromatography/mass spectroscopy analyser. The analyser shall be calibrated at appropriate intervals by use of calibration gas standards. The analyser shall be operated so that its sensitivity for methane is at least 10 % of the specified maximum hydrocarbon contents.

7.12 Nitrous oxide content

The nitrous oxide content shall be determined by one of the following procedures.

a) By a gas cell-equipped infrared analyser. The analyser shall be calibrated at appropriate intervals by the use of calibration standards at approximately 4,5 µm (the characteristic absorption wavelength for nitrous oxide). The analyser shall be operated so that its sensitivity for nitrous oxide is at least the specified maximum amount.

b) By a gas chromatograph in accordance with 7.8 a).

7.13 Odour

The odour shall be determined by the following procedure.

Odour in liquid is checked by evaporating to dryness 200 ml of liquid in a loosely covered 400-ml beaker or similar container with a fresh filter paper in the bottom. The cover is removed at the point of complete evaporation and the beaker is odour tested several times until it has warmed to above the freezing point of condensed water on the outside.

CAUTION — In the above procedure, do not place face directly in front of the valve or beaker. Instead, cup the hand and bring some of the gas being vented toward the nose.

7.14 Particulate matter content

A particulate matter test method is not required for gaseous nitrogen. The particulate matter of liquid nitrogen shall be determined by the following method: pass a liquid nitrogen sample through a specified low micrometre-rated, tared, analytical filter disk contained in a suitable holder. The filtered liquid is collected and measured in an open container. The assembly is warmed and the disk is reweighed. The size of any particulate matter can be evaluated by the examination of the filter disk with a suitable optical magnifier.

Annex A
(informative)

Gas chromatography (GC) and mass spectrometer (MS) applications

Gas chromatography (GC) should be used as the reference or preferred method to analyse nitrogen impurities, except for odour, water, and particulate content.

A mass spectrometer coupled with a gas chromatography (GC-MS) may be used as an alternative to simple gas chromatography so as to avoid possible interferences (especially for the hydrocarbons).

Table A.1 summarizes the applications of these methods for nitrogen.

Table A.1 — Application of GC and MS

Component	**GC with DID detector on molecular sieve column**	**GC with FID detector on Porapak[a] column (or equivalent)**	**GC with methanizer and FID detector on Porapak[a] column (or equivalent)**	**GC-MS**	**MS**
Water	—	—	—	—	—
Total hydrocarbon content	—	X	X	X	X
Oxygen	—	X	—	—	—
Hydrogen	—	—	X	X	—
Argon	X	—	—	X	X
Carbon dioxide	—	—	X	X	—
Carbon monoxide	X	—	X	X	—
Aromatic, chlorinated, halogenated hydrocarbons	—	X[b]	—	X	—
Nitrous oxide	X	—	—	X	—
Odour	—	—	—	—	—
Particulate matter	—	—	—	—	—

DID = Discharge ionization detector

FID = Flame ionization detector

"X" indicates that the method can be used.

“—" indicates that the method is not used.

[a] Porapak® packing is an example of a suitable product available commercially. This information is given for the convenience of users of this part of ISO 15859 and does not constitute an endorsement by ISO of this product.

[b] An electron capture detector (ECD) could also be used for halogenated hydrocarbons detection.

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