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Space systems — Fluid characteristics, sampling and test methods —

Part 2: **Hydrogen**

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

Partie 2: Hydrogène



Reference number ISO 15859-2:2004(E)

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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 15859-2 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

ISO 15859-2:2004(E)

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.

Space systems — Fluid characteristics, sampling and test methods —

Part 2: Hydrogen

1 Scope

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

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

 Type I: gaseous					
— Grade A: fuel,					
— Grade F: fuel,					
 Type II: liquid					
— Grade A: fuel,					
— Grade F: fuel.					

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

ISO 14687, Hydrogen fuel — Product specification

Terms and definitions

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

3.1

total hydrocarbon content (as methane)

single carbon atom equivalent

3.2

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

Chemical composition 4

Unless otherwise provided in an applicable technical specification, the composition of hydrogen 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.

Limits Type I Type II Component (gaseous) (liquid) Grade Grade Grade Grade Α F Α F Volume fraction, 99,994 99,995 99,994 99,995 Hydrogen (H₂) % by difference, min. Purity Para-hydrogen (balance Volume fraction, Not Not Not 95,0 ortho-hydrogen) %, min. specified specified specified Total gaseous impurities µl/l, max. 60 50 60 50 Nitrogen, water, and volatile µl/l, max. 9,0 10 9,0 10 hydrocarbons combined **Impurities** Oxygen plus argon μl/l, max. 5,0 1 5,0 1 Helium 45,0 40 45,0 40 µl/l, max. Carbon monoxide plus 1,0 1 1 µl/l, max. 1,0 carbon dioxide

Table 1 — Composition limits

Procurement

The types and grades of hydrogen specified in Clause 1 should be procured in accordance with ISO 14687 or an applicable national standard.

Fluid sampling 6

CAUTION — Hydrogen is highly flammable and explosive, is an asphyxiant, and is volatile. Human contact with liquid hydrogen will result in severe injury. Care should be taken in the handling and storage of liquid hydrogen to prevent contact with the human body and with materials that are not compatible with hydrogen.

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. The plan shall specify

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

6.2 Responsibility for sampling

Unless otherwise provided in an applicable technical specification, the hydrogen delivered to the flight vehicle interface shall be sampled and verified by the supplier responsible for providing the hydrogen 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.

ISO 15859-2:2004(E)

6.8 Gaseous samples

Gaseous samples shall be a typical specimen from the gaseous hydrogen 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 Rejection

When any sample of the fluid tested in accordance with Clause 7 fails to conform to the requirements specified in this part of ISO 15859, 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 hydrogen. If required, alternate test methods are described in 7.3 to 7.10. 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 hydrogen.

7.2 Parameters of analysis

The parameters for analytical techniques contained in 7.3 to 7.10 are the following:

- a) purity and impurity contents shall be mole fraction expressed as a percentage by volume (volume fraction, %) unless otherwise noted;
- calibration gas standards containing the applicable gaseous components may be required to calibrate the analytical instruments used to determine the limiting characteristic levels of 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;
- analytical equipment shall be operated in accordance with the manufacturer's instructions.

7.3 Hydrogen purity

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

- a) By a thermal conductivity analyser measuring the aggregate impurities which have different thermal conductivities from hydrogen. The analyser is to be calibrated at appropriate intervals using calibration gas standards. The range of the analyser shall be no greater than 10 times the difference between the specified minimum value of hydrogen purity, expressed as a volume fraction (%), and 100 %. Thus for a 99,5 % minimum volume fraction of hydrogen, the analyser should have a maximum range of 5 % impurity or from 95 % to 100 % hydrogen.
- b) By a volumetric or manometric gas analysis apparatus.
- c) By determining the quantity of aggregate impurities using the methods given in 7.4 to 7.10. The purity of hydrogen is the value obtained when the quantity of aggregate impurities, expressed as a mole fraction (%), is subtracted from 100 %.
- d) By any suitable chromatographic system which effectively measures specific impurities [see 7.8 a)].

7.4 Hydrogen assay for para-hydrogen (liquid samples only)

Para-hydrogen (percent minimum balance ortho-hydrogen) shall be determined by thermal conductivity type in-stream analysers installed in the supplier's system and shall be calibrated integrally by the appropriate use of temperature-controlled catalyst beds.

The test may be performed only when agreed upon between the supplier and the customer.

7.5 Water content

For liquid hydrogen, the water content cannot be determined by sampling. For gaseous hydrogen, the water content shall be determined by one of the following procedures.

- a) By an electrolytic hygrometer having an indicator graduated in cubic centimetres per cubic metre within a range no greater than 10 times the specified maximum water content. Recombination of oxygen with hydrogen can occur, producing a false high reading. Refer to instrument manufacturer's instructions for proper analytical technique.
- b) By a dew-point analyser in which the temperature of a viewed surface is measured at the time water first begins to form.
- c) By a piezoelectric sorption hygrometer, of which the accuracy of analysis shall be \pm 0,1 cm³/m³ or 5 % of the reading, whichever is greater.
- d) By a metal-oxide-capacitor-equipped analyser within a range no greater than 10 times the specific maximum water content.

7.6 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 content.

Oxygen content

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

- 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 heat-of-reaction-type analyser. The analyser shall be calibrated at appropriate intervals by the 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.
- 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.
- By a gas chromatography method such as that described under 7.8 a).
- By a mass spectrometer. The mass spectrometer shall be operated so that its sensitivity is at least 10 % of the specified oxygen content.

Argon, nitrogen, and helium content

The argon, nitrogen, and helium contents shall be determined by one of the following procedures.

- By a gas chromatograph. This method may be used not only for argon, nitrogen, neon and helium 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 20 % 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 chromatography method such as that described in 7.8 a). The technique utilized shall be specific for the separation and analysis of carbon dioxide.
- By a catalytic methanizer gas chromatograph method such as that described in 7.8 a).
- By an analyser in which carbon dioxide 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 dioxide content.
- 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.

7.10 Carbon monoxide content

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

- a) 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.
- b) 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.
- 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 analytical bias of the tube.
- d) By a catalytic methanizer gas chromatograph method such as that described under 7.8 a).

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 hydrogen impurities, except for water and para-hydrogen.

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

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

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
Para-hydrogen	_	_	_	_	_
Water	_	_	_	_	_
Total hydrocarbon content	_	Х	_	_	_
Oxygen	_	Х	_	_	Х
Argon, nitrogen, helium	X	_	_	Х	Х
Carbon dioxide	_	_	Х	Х	_
Carbon monoxide	Х	_	Х	Х	_

DID = Discharge ionization detector

FID = Flame ionization detector

[&]quot;X" indicates that the method can be used.

^{-&}quot; indicates that the method is not used.

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