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Space systems — Fluid characteristics, sampling and test methods —

Part 1: Oxygen

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

Partie 1: Oxygène

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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 15859-1 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.

Space systems — Fluid characteristics, sampling and test methods —

Part 1: Oxygen

1 Scope

This part of ISO 15859 specifies the limit values for the composition of oxygen and establishes the sampling and test requirements applicable for the verification of the oxygen composition.

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

— Type I: gaseous

  — Grade A: standard, purging/pressurization,

  — Grade CB: crew breathing,

  — Grade F: fuel-cell;

— Type II: liquid

  — Grade A: oxidizer,

  — Grade B: oxidizer,

  — Grade F: fuel-cell.

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
particulate matter
undissolved solids retained on a filter paper with a 10 µm absolute rating

3.2
total hydrocarbon content (as methane)
single carbon atom equivalent

3.3
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 chemical composition of oxygen 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 (gaseous)			Type II (liquid)		
			Grade A	Grade CB	Grade F	Grade A	Grade B	Grade F
Purity	Oxygen (O_2)	Volume fraction, %, min.	99,6	99,5	99,989	99,2	99,5	99,989
Impurities	Total hydrocarbons (as methane)	µl/l, max.	50	50	23	75	67,7	23
	Alkynes (as acetylene)	µl/l, max.	—	—	0,05	1,55	0,5	0,05
	Water	µl/l, max.	8	10	3	26,3	26,3	3
	Particulate matter	mg/l, max.	—	—	—	—	1,0	—
	Methane	µl/l, max.	—	—	16	—	—	16
	Ethane	µl/l, max.	—	—	2	—	—	2
	Propane and higher hydrocarbons (as propane)	µl/l, max.	—	—	1	—	—	1
	Nitrous oxide	µl/l, max.	—	4	1	—	—	1
	Halogenated hydrocarbons	µl/l, max.	—	2	1	—	—	1
	Chlorinated hydrocarbons	µl/l, max.	—	0,2	0,01	—	—	0,01
	Odour	µl/l, max.	—	None	—	—	—	—
	Carbon monoxide (CO) and carbon dioxide (CO_2)	µl/l, max.	—	a	1	b	b	1
	Other [nitrogen (N_2), argon (Ar), krypton (Kr), etc.]	µl/l, max.	—	c	75	—	—	75

a 10 µl/l for CO and 10 µl/l for CO_2.

b 0,1 µl/l for CO and 3 µl/l for CO_2 when required to meet hardware needs.

c Other discernible impurities shall be identified, measured and recorded.

5 Procurement

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

6 Fluid sampling

CAUTION — Oxygen is an oxidant. Human contact with liquid oxygen will result in severe injury. Care should be taken in the handling and storage of liquid oxygen to prevent contact with the human body and with materials that are not compatible with oxygen. Care should also be taken to prevent high concentrations of gaseous oxygen in confined spaces where ignition of combustible materials may occur.

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 oxygen delivered to the flight vehicle interface shall be sampled and verified by the supplier responsible for providing the oxygen 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 oxygen 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 oxygen 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 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 oxygen. If required, alternate test methods are described in 7.3 to 7.16. 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 oxygen.

7.2 Parameters of analysis

The parameters for analytical techniques contained in 7.3 to 7.16 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 Oxygen purity

The oxygen concentration shall be determined by one of the following methods.

a) By a volumetric or manometric gas absorption (Orsat type) analysis apparatus using a suitable oxygen absorbing reagent.

b) By a paramagnetic-type analyser, which shall be calibrated at appropriate intervals by use of calibration gas standards. For nondigital instruments, the range used should be no greater than 10 times the difference between the specified minimum value of oxygen purity, expressed as a volume fraction (%), and 100 %. Thus, for 99,5 % minimum volume fraction of oxygen, the analyser should have a maximum range of 5 % impurity, or from 95 % to 100 % oxygen.

c) By a thermal-conductivity-type analyser, which shall be calibrated at appropriate intervals by use of calibration gas standards. The range used should be no greater than 10 times the difference between the specified minimum value of oxygen purity, expressed as a volume fraction (%), and 100 %. Thus, for 99,5 % minimum volume fraction of oxygen, the analyser should have a maximum range of 5 % impurity, or from 95 % to 100 % oxygen;

NOTE An argon-in-oxygen gas standard is normally used.

CAUTION — The principle of operation for the thermal-conductivity analyser is bipolar in nature. It is normally used only to measure composition of binary mixtures. Where the possibility of a contaminant other than argon (i.e. nitrogen) can exist in the oxygen, the use of a thermal-conductivity analyser may not be appropriate.

d) By determining the amount of aggregate impurities using the methods in 7.4 to 7.16. The percent of oxygen is the value obtained when this amount, expressed as percent, is subtracted from 100.

7.4 Total hydrocarbon content (THC)

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

a) By a gas chromatograph in accordance with 7.16 a).

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.

7.5 Acetylene content

The acetylene content shall be determined by one of the following methods.

a) By a gas chromatograph in accordance with 7.16 a).

b) By a wet chemical method in which the sensitivity for acetylene is at least the specified maximum amount.

7.6 Water content

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

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 the 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 cm^3/m^3 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.

7.7 Particulate matter content

The particulate matter of Type II shall be determined by the following methods.

a) Pass a liquid oxygen 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 re-weighed. The size of particulate matter can be evaluated by examination of the filter disk with a suitable optical magnifier.

b) In order to reduce the amount of permanent particulate matter in liquid oxygen, the liquid may be filtered during transfer. The equipment used is a 10-µm to 40-µm (10 µm nominal, 40 µm absolute) filter assembly installed in the transfer system.

7.8 Methane content

The methane content shall be determined by a gas chromatography method in accordance with 7.16 a).

7.9 Ethane and other hydrocarbon (as ethane) content

The ethane and other hydrocarbon (as ethane) content shall be determined by a gas chromatograph in accordance with 7.16 a).

7.10 Propane and higher hydrocarbons

The propane and higher hydrocarbons, as propane, shall be determined by one of the following methods.

a) By a gas chromatograph in accordance with 7.16 a).

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

7.11 Nitrous oxide content

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

a) By a chemioluminescence method.

b) By a gas chromatograph, in accordance with 7.16 a), equipped with a discharge ionization-type detector (DID).

7.12 Halogenated hydrocarbons and chlorinated hydrocarbons

The halogenated hydrocarbons and chlorinated hydrocarbons shall be determined by one of the following methods.

a) By a gas chromatograph in accordance with 7.16 a).

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

7.13 Odour

The odour shall be determined by one of the following methods.

a) By sniffing a moderate flow of gaseous oxygen flowing from the container being tested.

b) 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 either of the above procedures, 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 Carbon dioxide content

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

a) By a gas chromatography method such as that described in 7.16 a). The technique utilized shall be specific for the separation and analysis of carbon dioxide.

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

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 catalytic methanizer gas chromatographic device in accordance with 7.16 a).

7.15 Carbon monoxide content

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

a) By a gas chromatography method method in accordance with 7.16 a). The technique utilized shall be specific for separation and analysis of carbon monoxide.

b) By a mass spectrometer, which 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 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 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.

e) By a catalytic methanizer gas chromatograph device in accordance with 7.16 a).

7.16 Argon, krypton and nitrogen content

The argon, krypton and nitrogen contents shall be determined by one of the following methods.

a) By a gas chromatograph. This method may be used not only for argon, krypton and nitrogen 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, which shall be operated so that its sensitivity is at least 10 % of the specified maximum amount of the component.

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 oxygen impurities, except for 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 oxygen.

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
Total hydrocarbon content	—	X	—	—	—
Acetylene	—	X	—	X	—
Water	—	—	—	—	—
Particulate matter	—	—	—	—	—
Methane	X	X	—	X	—
Ethane	—	X	—	X	—
Propane	—	X	—	X	—
Nitrous oxide	X	—	—	X	—
Halogenated hydrocarbons	—	X[b]	—	X	—
Odour	—	—	—	—	—
Carbon dioxide	—	—	X	X	—
Carbon monoxide	X	—	X	X	—
Argon, krypton, nitrogen	X	—	—	X	X

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) can also be used for halogenated hydrocarbons detection.

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