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Space systems — Fluid characteristics, sampling and test methods —

Part 5:
Nitrogen tetroxide propellants

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

Partie 5: Ergols à base de tétraoxyde d'azote

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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 15859-5 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 5: Nitrogen tetroxide propellants

1 Scope

This part of ISO 15859 specifies limits for the chemical composition and physical properties of propellants based on nitrogen tetroxide (N_2O_4)-based propellants and establishes the sampling and test requirements applicable for the verification of the nitrogen tetroxide composition.

This part of ISO 15859 is applicable to N_2O_4-based propellants, used as oxidizers in propellant systems of space systems as well as in both flight hardware and ground facilities, systems and equipment, of the following types and grades.

— Types

  — NTO: N_2O_4 with red-brown colour,

  — MON-1: N_2O_4 and NO with green colour,

  — MON-3: N_2O_4 and NO with green colour,

  — MON-10: N_2O_4 and NO with green colour,

  — MON-25: N_2O_4 and NO with green colour;

— Grades

  — standard: no iron requirement,

  — low-iron: 0,5 µg/g or 1,0 µg/g iron maximum.

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.

NOTE NTO is the acronym for nitrogen tetroxide and MON is the acronym for mixed oxides of nitrogen.

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 nominal and 40 µm absolute rating

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

4 Chemical composition and physical properties

4.1 Chemical composition

Unless otherwise provided in an applicable technical specification, the chemical composition and physical properties of N_2O_4-based propellants 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				
			NTO (red-brown)	MON-1 (green)	MON-3 (green)	MON-10 (green)	MON-25 (green)
N_2O_4	Mass fraction, %, min.		99,5	—	97,0	88,8	—
Nitric oxide (NO)	Mass fraction, %,	max.	[a]	1,0	3,0	11,0	26,0
		min.	[a]	0,6	1,5	10,0	25,0
N_2O_4 + NO	Mass fraction, %, min.		—	99,5	99,5	—	99,5
Water equivalent	Mass fraction, %, max.		0,17	0,17	0,20	0,20	0,17
Chlorides	Mass fraction, %, max.		0,040	0,040	0,040	0,040	0,040
Nonvolatile residue[b]	mg/l, max.		—	10,0	10,0	10,0	10,0
Iron[b]	µg/g, max.		—	0,5	1,0	1,0	0,5
Particulate matter	mg/l, max.		10,0	10,0	—	—	10,0

[a] The NO content shall be limited to that which does not change the specified red-brown colour of the propellant.

[b] This requirement applies to the low-iron grade of the propellant only.

4.2 Physical properties

The propellant shall be a homogeneous liquid when examined visually by transmitted light. The NTO shall be red-brown in colour; the MON shall be green in colour.

5 Procurement

The nitrogen tetroxide propellant types and grades specified in Clause 1 should be procured in accordance with an applicable national standard.

6 Fluid sampling

CAUTION — Nitrogen-tetroxide-based propellants, in the liquid or vapour form, are strong oxidizers and are toxic and volatile. Care should be taken in the handling and storage of nitrogen tetroxide to prevent contact with the human body and with materials that are not compatible with nitrogen tetroxide.

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 tetroxide delivered to the flight vehicle interface shall be sampled and verified by the supplier responsible for providing the nitrogen tetroxide 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 Liquid samples

Liquid samples shall be a typical specimen from the liquid nitrogen tetroxide 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. 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.

6.9 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 tetroxide. 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 nitrogen tetroxide.

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 expressed as a percentage by mass (mass fraction, %) unless otherwise specified;

b) calibration standards containing the applicable liquid 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;

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

7.3 Nitrogen tetroxide purity

The nitrogen tetroxide purity shall be determined by the following methods.

a) A sample of the nitrogen tetroxide is placed in a preweighed ampoule, sealed, and weighed. The ampoule is transferred to a sturdy glass bottle or flask containing 100 ml of demineralized water and 20 ml of a 30 % hydrogen peroxide solution and the container is sealed. The container is chilled and shaken to break the ampoule and until all the fumes disappear. An air condenser is installed in the mouth of the container and the contents are heated (approximately 120 °C) for approximately 45 min, using a moderate heat source. The container is cooled to room temperature and the contents are titrated to the yellow end point using a bromothymol blue indicator and a 0,5 mol/l sodium hydroxide solution. The mass fraction of nitrogen tetroxide is calculated from the data. This method provides the total quantity of N_2O_4 and nitric acid, not that of N_2O_4 alone.

b) An indirect method where the amount of aggregate impurities is determined using the methods in 7.4 to 7.10. The mass fraction (%) of nitrogen tetroxide, $w_{N_2O_4}$, is the value obtained when the quantity of aggregate impurities, expressed as a mass fraction (%), is subtracted from 100. See the following.

$$w_{N_2O_4} = 100 - \left[w_{H_2O(free)} + w_{HNO_3} + w_{NO} \right]$$

where w_i is the mass fraction of component i, in %.

The quantities of free H_2O and HNO_3 are determined in accordance with 7.6. These determinations allow the calculation of the "water equivalent" [$w_{H_2O(equiv)}$] content as follows:

$$w_{H_2O(equiv)} = w_{H_2O(free)} + \frac{w_{HNO_3}}{7}$$

Nitric oxide (NO) content is determined in accordance with 7.4.

7.4 Nitric oxide (NO) content

The nitric oxide content shall be determined by the following methods.

a) By a gravimetric method consisting of the measurement of the amount of O_2 needed to oxidize the NO into N_2O_4.

b) By a visible spectrometric absorption method.

7.5 Free water and nitric acid (HNO_3) (water equivalent) content

The free water and nitric acid (water equivalent) content shall be determined by the following method.

a) By a near-infrared absorption method. The technique utilized shall be specific for the detection and analyses of free water and nitric acid in an N_2O_4 medium.

b) By a gas chromatographic method (for water equivalent only).

c) By a coulometric method (for water equivalent only).

7.6 Nitrogen tetroxide and nitric oxide content

The nitrogen tetroxide and nitric oxide content shall be determined by adding the nitrogen tetroxide concentration and the nitric oxide content together.

7.7 Chloride content

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

a) By a silver nitrate titration potentiometric method.

b) By a colorimetric method.

c) By an ion chromatographic method.

d) By a potentiometric method using a chloride-specific electrode.

All these methods shall not be used directly on the liquid N_2O_4 sample but after dissolving it in an aqueous solution or from a nonvolatile residue after dissolving it in an aqueous acid solution.

7.8 Nonvolatile residue (NVR) content

The nonvolatile residue contents shall be determined by a gravimetric measurement method in accordance with the following procedure. A measured sample is gradually evaporated using a suitable heat source in an exhaust hood. The difference in mass before and after evaporation is calculated as the nonvolatile residue.

7.9 Iron content

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

a) By an atomic absorption spectrophotometric method.

b) By an inductively coupled argon plasma emission spectrometric method.

This iron content cannot be measured directly on the liquid N_2O_4 sample but from a nonvolatile residue after dissolving it in an aqueous acid solution.

7.10 Particulate content

The particulate matter content shall be determined by a gravimetric measurement method. A known volume of oxidizer is filtered through a preweighed test membrane filter and the increase in membrane filter mass is determined after washing and drying. The change in mass of a control membrane filter located immediately below the test membrane filter is also determined. The particulate matter contaminant is determined from the increase in mass of the test membrane filter relative to the control membrane filter.

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