INTERNATIONAL STANDARD

ISO 14624-5

First edition
2006-04-15

Space systems — Safety and compatibility of materials —

Part 5: Determination of reactivity of system/component materials with aerospace propellants

Systèmes spatiaux — Sécurité et compatibilité des matériaux —

Partie 5: Détermination de la réactivité des matériaux des systèmes/composants avec les ergols spatiaux

Reference number
ISO 14624-5:2006(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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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 14624-5 was prepared by Technical Committee ISO/TC 20, *Aircraft and space vehicles*, Subcommittee SC 14, *Space systems and operations*.

ISO 14624 consists of the following parts, under the general title *Space systems — Safety and compatibility of materials*:

— *Part 1: Determination of upward flammability of materials*

— *Part 2: Determination of flammability of electrical-wire insulation and accessory materials*

— *Part 3: Determination of offgassed products from materials and assembled articles*

— *Part 4: Determination of upward flammability of materials in pressurized gaseous oxygen or oxygen-enriched environments*

— *Part 5: Determination of reactivity of system/component materials with aerospace propellants*

— *Part 6: Determination of reactivity of processing materials with aerospace fluids*

— *Part 7: Determination of permeability and penetration of materials to aerospace fluids*

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Introduction

This purpose of this part of ISO 14624 is to identify changes resulting from exposure of a material to an aerospace fluid that renders either the material or the fluid unsuitable for use.

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Space systems — Safety and compatibility of materials —

Part 5: Determination of reactivity of system/component materials with aerospace propellants

1 Scope

This part of ISO 14624 specifies test equipment and techniques used to identify interactions resulting from exposure of a material to an aerospace fluid.

This part of ISO 14624 may be used to determine the reactivity of system and component materials with aerospace fluids. It is applicable for determining interactive reactions between propellants and materials used in the design, construction, and operation of propellant storage, transfer, and flight systems. While this procedure is an excellent quick screen test for long-term propellant compatibility, it is semi-qualitative, and (if exposures exceed 12 months) long-term tests need to be used to quantify degradation as a function of time under use conditions.

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 4954:1993, *Steels for cold heading and cold extruding*

ISO 14951-3, *Space systems — Fluid characteristics — Part 3: Nitrogen*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
degradation
adverse physical or chemical change in a substance

3.2
immersion test
test in which the fluid covers the entire sample for the duration of the test

3.3
propellants
fluids, such as hydrazine and monomethylhydrazine, and oxidizers usually used for space projects

3.4
reaction
chemical change in which a substance decomposes, combines with other substances, or interchanges constituents with other substances

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4 Fluid transfer, storage, and flight systems

4.1 General

4.1.1 Procedure

This procedure is applicable for determining interactive reactions between propellants and materials used in the design, construction, and operation of propellant storage, transfer, and flight systems. The sample is immersed in the test fluid for 48 h at the maximum system temperature or 71 °C (160 °F), whichever is higher. This accelerated test provides semi-qualitative information. Tests used to evaluate the long-term interaction of materials with reactive fluids shall be conducted for a period of time no less than that of the anticipated time of use.

4.1.2 Safety

The proper safety equipment must be worn by the technician performing the test. A face shield, gloves, and a laboratory coat or apron shall be worn when handling the test fluids. The laboratory conducting the tests shall have a detailed emergency plan in the event of a runaway reaction.

4.2 Test criteria

4.2.1 Screening test

Exposure of the material (screening test) to the fluid for 2 h at ambient temperature and pressure shall not visibly change either the material or the fluid.

4.2.2 Immersion test

The sample immersed in the test fluid for 48 h at test temperature shall not cause a pressure rate increase that is 1,5 times more than the pressure rate increase that is caused by ISO 4954 stainless steel when exposed to the identical fluid and conditions. The standard test temperature for the hydrazine propellants (see ISO 14951-6 and ISO 14951-7) is 71 °C. This temperature shall be used when the intent of the test is ranking of materials or comparison to literature information. Other temperatures may be used to test materials for specific applications. For other fluids, the standard test temperature will depend upon the vapour pressure of that fluid; for example, the standard temperature for nitrogen tetroxide (see ISO 14951-5) is 21 °C.

For fluids that do not decompose into gaseous products at the test temperature (for example, nitrogen tetroxide), the pressure increase shall not be greater than the vapour pressure of the fluid after exposure to polytetrafluoroethylene (for nonmetals) or ISO 4954 stainless steel (for metals).

4.2.3 Post-test analysis

After the sample has been exposed, decontaminated, and dried, no visible change in colour or texture of the material or test fluid shall be apparent. In addition, the sample mass change shall not be greater than $\pm$ 2 %.

The following also apply:

— the mass of impurities in the fluid after exposure to the material shall not be greater than twice the mass of impurities in the identical fluid after exposure to polytetrafluoroethylene (for nonmetals) or ISO 4954 stainless steel (for metals);

— halide (F^-, Cl^-, Br^-) concentrations in the fluid after exposure to the material shall not exceed the appropriate ISO specification for the fluid purity.

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4.3 Sample

4.3.1 Receiving inspection

When received, the test material must be accompanied by proper identification. The minimum information required is the manufacturer, trade name, composition, specification, generic name, and batch/lot number (if known). A visual inspection shall be performed and any anomalies shall be noted. A suitable material identification form is shown in A.1.

4.3.2 Sample preparation

The sample shall be tested in the intended use form (such as sheets or foams) and in the as-received thickness. Samples for the screening test shall weigh $\leqslant$ 0,25 g. Samples for the immersion test shall have a surface area of 25 $\pm$ 10 mm^2.

4.3.3 Sample cleaning

Samples shall be cleaned and dried to the end-use specifications. Contamination on the surfaces of solid, nonporous samples shall be removed by washing with de-ionized water and mild detergent, rinsing with de-ionized water, and drying with filtered, gaseous nitrogen. Particulate on the surfaces of solid, porous samples shall be removed with filtered, gaseous nitrogen meeting the requirements of ISO 14951-3.

4.3.4 Sample inspection

The cleaned sample shall be inspected to ensure it is at the specified worst-case thickness. Flaws and any residual contamination shall be noted. If the flaws result from sample preparation at the test facility, new samples shall be prepared. Samples with flaws that inordinately increase the surface area to bulk mass ratios shall not be tested. Samples shall be weighed and individually identified.

4.4 Test system

4.4.1 Screening test

The test system for the screening test shall consist of a glass beaker.

4.4.2 Immersion test

The test system for the immersion test shall consist of one reference and one sample chamber and temperature- and pressure-measuring devices (see Figure 1). Differential pressure transducers may be used for fluids, such as hydrazine and monomethylhydrazine, that decompose into gases at the test temperature. Absolute pressure transducers (on the sample and reference sides of the test system) may be used for those fluids that do not decompose into gases or undergo wide pressure fluctuations. Recommended analytical instruments for the post-test analyses include a differential scanning calorimeter, gas chromatography, gas chromatography/mass spectrograph, atomic absorption spectrophotometer, inductively coupled plasma optical spectrometer, inductively coupled plasma/mass spectrometer, ion chromatography, and high-performance liquid chromatography.

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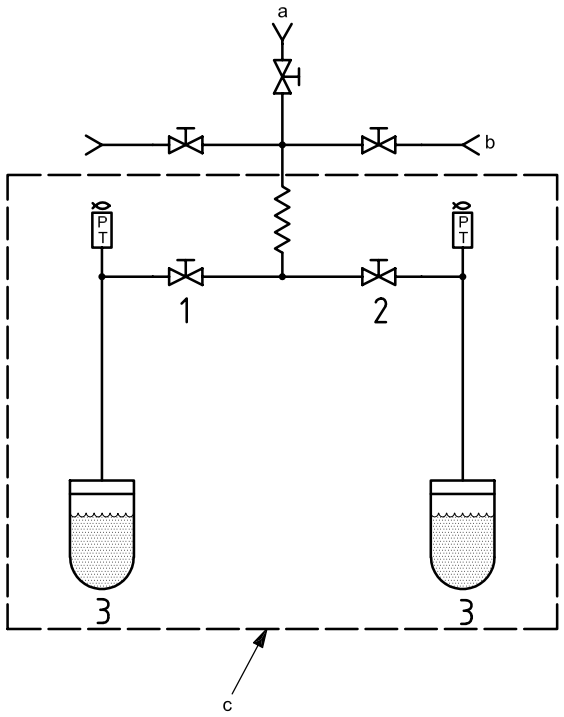

Key

1 solution valve 1
2 solution valve 2
3 test sample

a Vent to hood.
b Gaseous nitrogen source.
c Heated volume.

Figure 1 — Immersion test system

4.5 Procedure

4.5.1 Before testing

The test system shall be clean, and all measuring devices shall be in current calibration. The pretest procedure shall be as follows.

a) Analyse the fluid to be used in testing for impurities.

b) Verify the fluid meets the required use specifications before being exposed to the samples.

c) Record all pertinent information for the test, such as sample identification and pretest information about the sample and fluid.

d) Clean and dry the test and reference samples.

e) Photograph the samples.

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4.5.2 Test

4.5.2.1 Screening test

The screening test shall be as follows.

a) Place a 0,25 g test sample in the glass beaker.

b) Apply 10 mm^3 of fluid one drop at a time to the test sample at ambient temperature and pressure.

c) Wait 2 h, then examine the sample and fluid visually for obvious changes caused by the exposure.

d) Complete a suitable screening test report form (see A.2).

4.5.2.2 Immersion test

The immersion test shall be as follows.

a) Place the test sample in the sample chamber and add sufficient liquid, approximately 25 ml, to completely cover the test sample for the duration of the test. The addition of the test fluid must be performed to rigorously exclude water and carbon dioxide in the case of the propellant hydrazines and water in the case of nitrogen tetroxide. Contamination of the test fluid by these chemicals may give false indications of reactivity.

b) Seal the sample chamber to the sample side of the test apparatus.

c) Add sufficient test fluid to the reference chamber to obtain the same ullage as in the sample chamber.

d) Seal the reference chamber to the reference side of the test apparatus.

e) Activate the temperature- and pressure-monitoring devices.

f) Heat both chambers at a rate of less than 2 °C per minute until the test temperature, 71 °C minimum, is reached.

g) Continue the test for 48 h or until the pressure difference between the sample and reference transducers (PT) has exceeded the vapour pressure of the fluid plus 140 kPa.

h) Allow the temperature to lower to ambient.

4.5.3 Post-test analysis

The post-test analysis shall be as follows.

a) Perform a post-test analysis of the material and fluid to determine the extent of changes in the physical and chemical characteristics. The removal of the test fluid from the test system must be done in such a way as to exclude water and carbon dioxide contamination from the air.

b) Measure changes in weight, dimension, texture, and colour.

c) Perform a comparison of the thermal properties of the material by differential scanning calorimetry for nonmetals.

d) Determine changes in the purity in the test fluid or residue by liquid chromatography, ion chromatography, atomic absorption spectrophotometer, inductively coupled plasma emission spectrometer, inductively coupled plasma/mass spectrometer, or gas chromatography/mass spectroscopy analyses.

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e) Compare the post-test fluid analysis with the appropriate fluid specification. A suitable report form is shown in A.3 for a stainless-steel sample. The post-test fluid analysis shall be derived from the composition of the material being tested and have specific analytes appropriate to the composition.

f) If required, a graph of the volume of gas evolved versus time, for both the test sample and reference sample, may be provided.

4.6 Precision

Measurements shall be made to the following precision:

a) absolute pressure, ± 1 % of reading;

b) temperature, ± 3 °C;

c) sample dimensions, ± 5 % of the measurements;

d) time, ± 5 min.

4.7 Good laboratory practices

At least every two years, the test facility shall successfully demonstrate the ability to obtain accurate and repeatable data when testing selected reference materials.

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Annex A
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Example forms

A.1 Material identification form

Test material

Manufacturer: _____

Trade name: _____

Composition: _____

Specification: _____

Generic name: _____

Batch/lot number: _____

Use temperature (minimum): _____

Use temperature (maximum): _____

Aerospace fluid exposure time (field use): _____

Manufacturer

Name: _____

Address: _____

City: _____

State: _____

Country: _____

Supplier

Name: _____

Address: _____

City: _____

State: _____

Country: _____

Remarks: _____

_____

_____

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A.2 Screening test report form

Test sample material description

Test conditions

Test environment: _____

Test temperature: _____

Test duration: _____

Test results, observations, and comments

Pretest mass: _____

Photograph pretest samples

Post-test mass: _____

Material characteristics

Component	Pretest observations	Post-test changes

Note(s): Pass _____ Fail _____

Post-test photograph(s):

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A.3 Post-test analysis report form

Test sample material description

Geometric surface area: _____

Test conditions

Test environment: _____

Test temperature: _____

Test duration: _____

Reference material: ISO 4954:1993

Geometric surface area: _____

Test results, observations, and comments

Pretest mass: _____

Post-test mass: _____

Average gas pressure

Sample	Reference

Material characteristics

Component	Pretest observations	Post-test changes

Note(s):

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Post-test fluid analysis

Analysis	Unit	Limit	Sample	Reference
Nonvolatile residue	mg	0,1		
Bromide	mg	1,2		
Chloride	mg	0,5		
Chromium	µg	2,3		
Iron	µg	2,3		
Nickel	µg	2,3		

Notes: ND indicates that the results were less than the reporting limit.

Post-test photograph(s):

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[2] ISO 14951-5, *Space systems — Fluid characteristics — Part 5: Nitrogen tetroxide propellant*

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[6] ISO 15859-5, *Space systems — Fluid characteristics, sampling and test methods — Part 5: Nitrogen tetroxide propellants*

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[8] ISO 15859-7, *Space systems — Fluid characteristics, sampling and test methods — Part 7: Hydrazine propellant*

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