
**Space systems — Safety and
compatibility of materials —**

Part 6:

**Determination of reactivity of processing
materials with aerospace fluids**

Systèmes spatiaux — Sécurité et compatibilité des matériaux —

*Partie 6: Détermination de la réactivité des matériaux de traitement
avec les fluides aérospatiaux*

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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.

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.

ISO 14624-6 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*

Introduction

The 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 unsuitable for use or produces an exothermic reaction that may result in a fire.

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Space systems — Safety and compatibility of materials —

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

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 can be used to determine the reactivity of processing materials with aerospace fluids either through intent or casual exposure. It provides a means to determine the effects of minor amounts of fluids, such as a splash or spill, on materials used in ground support processing operations.

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 4599, *Plastics — Determination of resistance to environmental stress cracking (ESC) — Bent strip method*

ISO 4954, *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

aerospace fluid

fluid that is commonly used in the fabrication, development and processing of materials and in the production of aerospace and ground support equipment and propellants

EXAMPLES Cleaning agents, lubricants and solvents.

3.2

aerospace material

material used in the fabrication and/or production of ground support and flight components and systems

3.3

degradation

adverse physical or chemical change in a substance

- 3.4 exothermic reaction**
chemical reaction that generates heat
- 3.5 ground support equipment**
equipment used in the processing and preparation of flight hardware
- 3.6 immersion test**
test in which the fluid covers the entire sample for the duration of the test
- 3.7 reaction**
chemical change in which a substance decomposes, combines with other substances, or interchanges constituents with other substances
- 3.8 test conclusions**
those results that are reported on the reactivity test report form

4 Safety precautions

4.1 Laboratory facilities

Many aerospace fluids are considered to be toxic chemicals. These chemicals shall only be opened and used inside an approved laboratory hood.

4.2 Protective clothing

Personal protective clothing shall be worn by personnel when performing these tests. The minimum protections required are fluid-compatible gloves, laboratory apron, and face shield or goggles.

5 Test procedure

5.1 Inspection of sample on receipt

When received, the test material shall be accompanied by proper identification. The minimum information required is the manufacturer, trade name, composition, specification, generic name and batch/lot number. A visual inspection shall be performed and any anomalies shall be noted. Table A.1 shows a suitable material identification form.

5.2 Preparation of sample

5.2.1 General

The sample shall be tested in the intended use form (such as sheets or foams) and in the as-received thickness. Samples for the immersion test shall have a surface area of (250 ± 5) mm².

5.2.2 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 deionized water and mild detergent, rinsing with deionized water, and drying with filtered, gaseous nitrogen. Particulates on the surfaces of solid, porous samples shall be removed with filtered, gaseous nitrogen meeting the requirements of ISO 14951-3.

5.2.3 Sample inspection

The 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.

5.2.4 Test sample configuration

5.2.4.1 Sheets, film, and fabrics

Material being tested for chemical reactivity shall be cut in the form of a 100-mm square sample in the use thickness (see Figure 1). To determine changes in the mechanical properties of a material, the sample shall be cut in the form of a 100 mm by 150 mm rectangle (see Figure 1). For non-isotropic materials, the 100 mm by 150 mm samples shall be cut from both the machine (warp) and transverse (fill) directions.

5.2.4.2 Adhesives and coatings

Adhesives and coatings shall be applied in a thickness equivalent to normal use on aluminium foil and cured, if necessary, in accordance with the manufacturer's instructions.

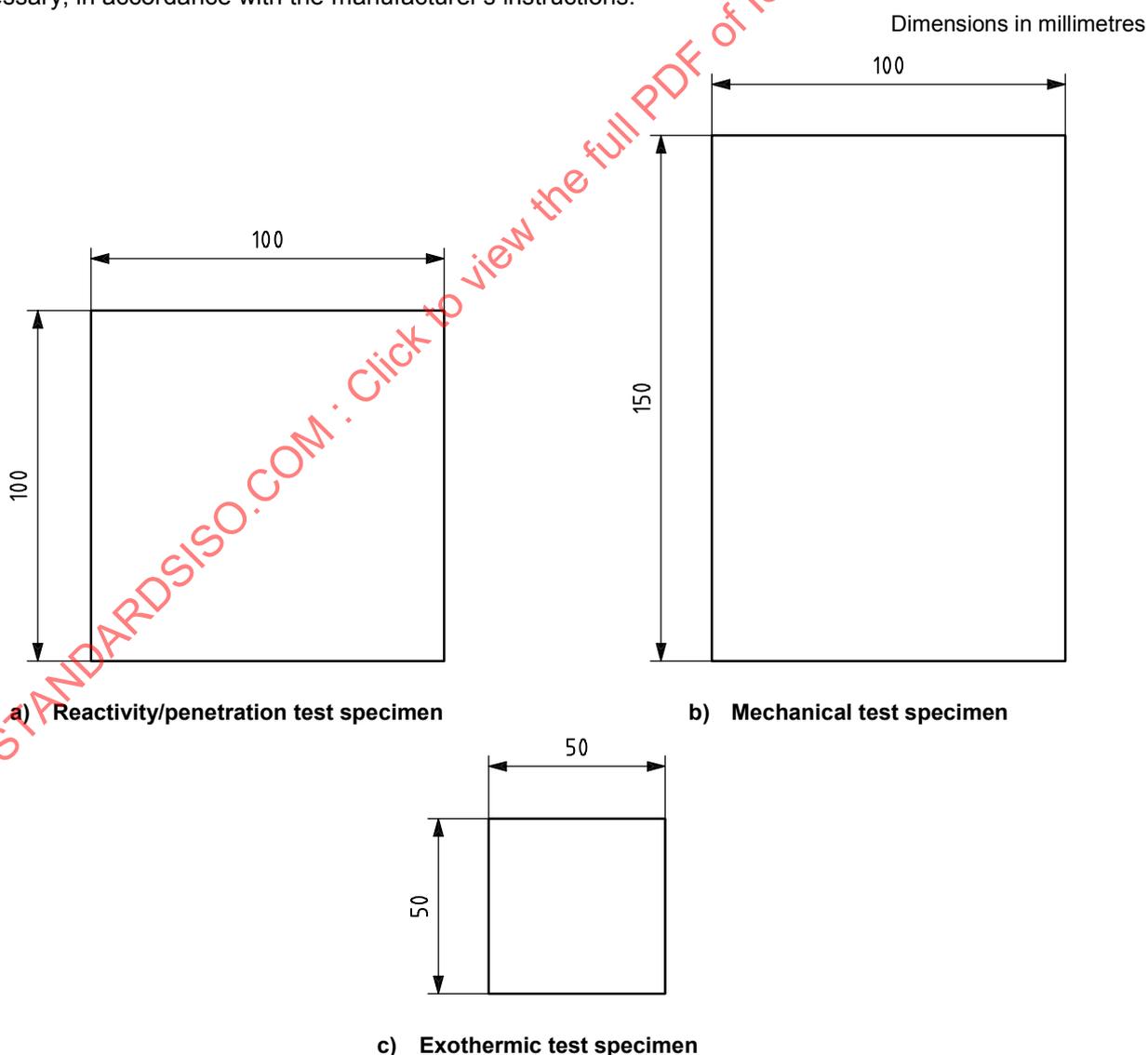


Figure 1 — Test specimen dimensions

5.2.4.3 Tapes

Tapes shall be applied on aluminium foil, a watch glass or Petri dish in the as-received condition and thickness.

5.2.4.4 Greases and gels

Greases and gels shall be applied on aluminium foil, watch glass or Petri dish in a thickness equivalent to normal use. They shall be cured, if required, in accordance with the manufacturer's instructions.

5.2.4.5 Liquids

Liquids shall be tested by placing 1 ml in the bottom of a 20 ml glass laboratory beaker.

5.2.4.6 Non-standard configurations

Complex shapes such as O-rings, cables, pipes, shall be tested in a configuration consistent with the intended use. Samples shall be cleaned as specified in 5.2.2.

5.3 Test methods

5.3.1 Reactivity test

5.3.1.1 General

This test is used to determine a possible material reaction and/or degradation when exposed to propellants or other liquids of interest.

5.3.1.2 Test procedure

The test procedure shall be as follows.

- a) Place a test specimen sample of the test material (see Figure 1) on a watch glass or in a Petri dish and place a thermocouple in contact with the middle of the specimen. The thermocouple shall be jacketed with glass or other inert material to minimize any reaction with the test fluid or the material being tested. Since the evaporation of the test fluid can mask a reaction, a differential thermocouple scheme using one thermocouple in contact with the sample and another thermocouple in contact with the test fluid may be more definitive.
- b) Add the test fluid, approximately 1 ml of the specified test fluid, to the centre of the sample, taking care not to expose the edges of the sample to the fluid to prevent wicking.
- c) Allow the test fluid to remain in contact with the sample for the specified exposure time. (The test exposure time of the fluid on the test material shall be determined by the expected use-time of the material.)
- d) Add test fluid as required to maintain a liquid film on the test sample during the specified exposure time.
- e) Carefully observe the test sample and thermocouple readout throughout the duration of the test.
- f) At the end of the specified exposure time, carefully blot the liquid from the sample and rinse the sample with running water for 60 s. Blotting and rinsing shall be performed with knowledge of the blotting material compatibility of the test fluid. Rinsing shall be performed in accordance with the environmental regulations governing disposal of the test fluid.
- g) Allow the test sample to dry under flowing air or nitrogen for 24 h prior to final evaluation.

5.3.1.3 Report

The report shall consist of the following as a minimum (an example of a suitable form for reporting the results of this test is shown in Table A.2):

- a) name of the test material, supplier, and manufacturer;
- b) test temperature, any temperature change, duration, and sample thickness before and after the test;
- c) any reactivity observed during the exposure, such as burning, smoking, bubbling, frothing, charring, solubility, swelling or fracture of the sample;
- d) any changes in the condition of the sample after the exposure, such as colour, flexibility, rigidity, surface condition, transparency, pitting, hardness, tackiness, friability or powder formation;
- e) test conclusions.

5.3.2 Penetration test

5.3.2.1 General

This test is used to determine both the possible liquid penetration and chemical reactivity of materials when exposed to aerospace fluids or other chemicals of interest. This test shall not be performed if the material failed the reactivity test.

5.3.2.2 Test procedure

The test procedure shall be as follows.

- a) Place an appropriately sized sample of the test material (see Figure 1) over a beaker.
- b) Add approximately 1 ml of the specified test fluid to the centre of the sample, taking care not to expose the edges of the sample to the fluid and start the stopwatch.
- c) Allow the test fluid to remain in contact with the sample for the specified exposure time.
- d) Add test fluid as required to maintain a liquid film on the test sample during the specified exposure time.
- e) Carefully observe for the first fallen droplet at the bottom of the beaker and note the time of occurrence.
- f) For materials used for protective garments, observe for initial wetness underneath the test sample and note the time of occurrence.

NOTE Atmospheric condensation could occasionally form underneath a sample during a test, giving a false indication of penetration. In such an event, verification can be made by applying a hypergol-compatible blotter that is known to discolour when in contact with a hypergolic fluid.

- g) Carefully blot the liquid from the sample at the end of the specified exposure time.
- h) Rinse the sample with running water for 60 s. Rinsing shall be performed in accordance with the environmental regulations governing disposal of the test fluid.
- i) Allow the test sample to dry under flowing air or nitrogen for 24 h prior to final evaluation.

5.3.2.3 Report

The report shall consist of the following as a minimum (an example of a suitable form for reporting the results of this test is shown in Table A.2):

- a) name of the test material, supplier and manufacturer;
- b) test temperature, pressure, duration, and sample thickness before and after the test;
- c) any penetration observed during the exposure and the elapsed time of occurrence;
- d) for materials used for protective garments, any wetness observed underneath the test sample during the exposure and the elapsed time of occurrence;
- e) any reactivity observed during the exposure, such as burning, smoking, bubbling, frothing, charring, solubility, swelling or fracture of the sample;
- f) any changes in the condition of the sample after the exposure, such as colour, flexibility, rigidity, surface condition, transparency, pitting, hardness, tackiness, friability or powder formation;
- g) test conclusions.

5.3.3 Mechanical evaluation test

5.3.3.1 General

This test is used to determine possible changes in the mechanical properties of a material due to exposure to aerospace fluids or other chemicals of interest.

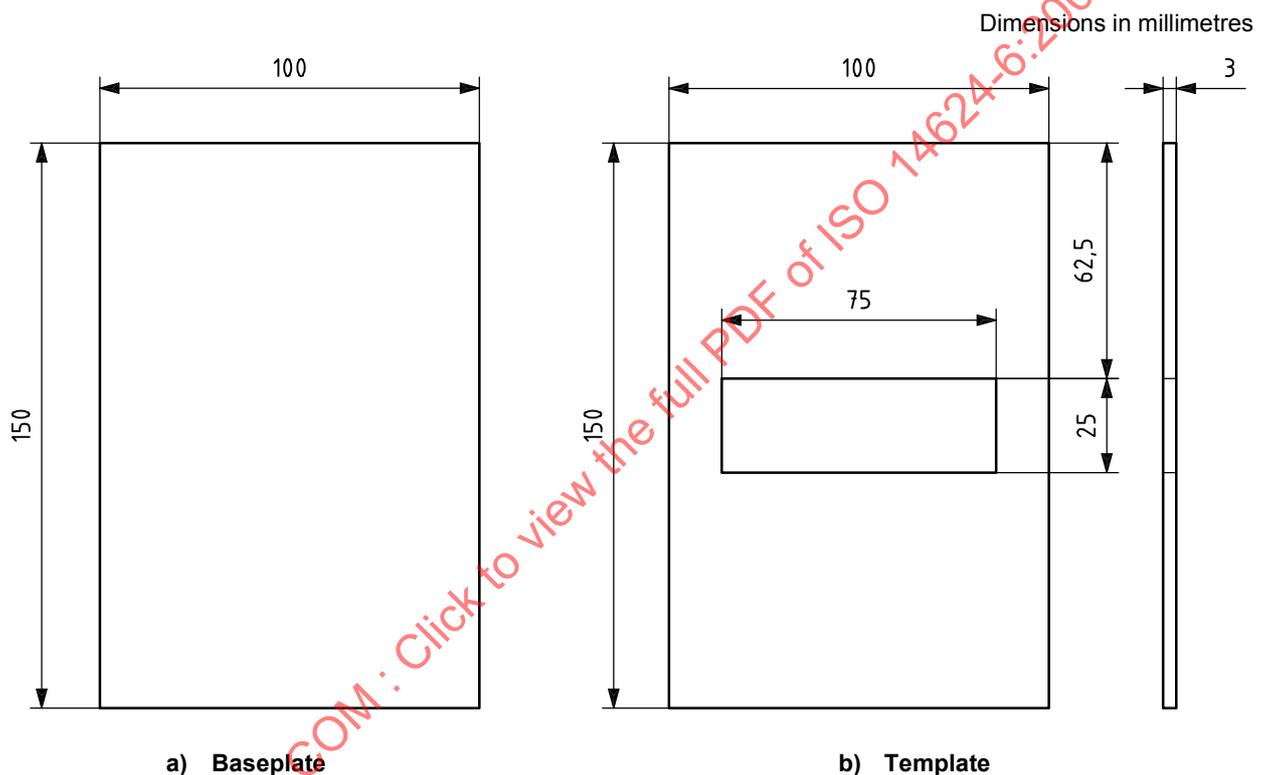
The reactivity test (see 5.3.1) shall be used as a screening test prior to the performance of this test.

5.3.3.2 Test procedure

The test procedure shall be as follows.

- a) Select the desired mechanical property to be evaluated and determine the value of that property on unexposed samples as a control.
- b) Place a 100 mm by 150 mm test specimen on a flat polytetrafluoroethylene or stainless-steel base plate meeting the requirements of ISO 4954 (see Figure 2).
- c) Take a template, shown in Figure 2, and apply a bead of fluid-compatible grease (e.g. perfluoroether) around the 75 mm by 25 mm opening (to prevent wicking under the template). An alternative is to have a compatible template constructed with a continuous ridge around the opening on the side facing the sample that may be clamped against the sample effecting a chevron seal.
- d) Then clamp both the template (with the grease against the test specimen) and test specimen to the base plate. The test specimen shall be sandwiched between the template and the base plate.
- e) Place sufficient test fluid in the slot to wet the full 75 mm by 25 mm surface of the exposed strip of test specimen.
- f) Allow the test fluid to remain in contact with the test specimen for the specified exposure time.
- g) Add test fluid as necessary to maintain a uniform liquid film across the width of the test specimen during the specified exposure time.

- h) At the end of the specified exposure time, carefully remove the test fluid and rinse with running water for 60 s. Rinsing shall be performed in accordance with the environmental regulations governing disposal of the test fluid.
- i) Disassemble the test fixture.
- j) If used, wipe the grease off the test specimen.
- k) Allow the test sample to dry under flowing air or nitrogen for 24 h prior to final evaluation.
- l) Determine the value of the mechanical property to be evaluated using a test procedure appropriate for the material being evaluated.



NOTE Items are constructed of stainless steel or Teflon.

Figure 2 — Exposure fixture for mechanical test

5.3.3.3 Report

The report shall consist of the following as a minimum:

- a) name of the test material, supplier and manufacturer;
- b) name of the test chemical;
- c) test temperature, pressure, duration, and sample thickness before and after the test;
- d) any reactivity observed on the test specimen in accordance with 5.3.1;
- e) the value of the mechanical property obtained for the exposed and unexposed test material and the method used to measure that property.

5.3.4 Shock sensitivity test

The shock sensitivity of a material exposed to these fluids shall be assessed using mechanical impact test procedures similar to the liquid and gaseous oxygen impact test, drop-weight impact of solid-phase hazardous materials, or the drop-weight impact on monopropellant test.

5.3.5 Exothermic reaction test

5.3.5.1 General

This test measures the temperature rise of a material when exposed to aerospace fluids or other chemicals of interest. An excessive temperature rise could suggest an impending, spontaneous ignition of the material.

NOTE Since nitrogen tetroxide has a boiling point of 20 °C, this method is not applicable for nitrogen tetroxide as its evaporative cooling effect would make any results obtained inconclusive.

5.3.5.2 Test procedure

The test procedure shall be as follows.

- a) Take an appropriately sized sample of the test material (see Figure 1) and place it on a watch glass or Petri dish.
- b) Position a sheathed thermocouple or resistance thermometer so it touches the centre of the sample.
- c) Add 1 ml of the specified test fluid to the centre of the sample, making sure that the sheath is in the liquid.
- d) Allow the test fluid to remain in contact with the sample for the specified exposure time while monitoring the temperature.
- e) At the end of the specified exposure time, carefully blot the sample dry, rinse with running water for 60 s, and air dry for 24 h. Rinsing shall be performed in accordance with the environmental regulations governing disposal of the test fluid.

5.3.5.3 Report

The report shall consist of the following as a minimum (an example of a suitable form for reporting the results of the test is shown in Table A.2):

- a) name of the test material, supplier and manufacturer;
- b) name of the test chemical;
- c) starting temperature of the test fluid, the maximum fluid temperature observed during the test and the time of occurrence, and the test fluid temperature at the end of the test;
- d) duration of the test and sample dimensions;
- e) any reactivity observed in the test specimen.

5.3.6 Environmental stress cracking test

5.3.6.1 General

The method specified in ISO 4599 or other appropriate procedures shall be used to determine the susceptibility of a material to environmental stress cracking (ESC) when placed in contact with a test fluid. This procedure utilizes a two-point buckling load technique, which generates a bending stress at the midsection of the test specimen.

Preliminary qualitative tests at relatively high stress levels may be performed to identify materials with a tendency to undergo rapid ESC or crazing, and to reduce the total number of tests required to characterize a material. The following tests shall be performed.

- a) Perform a series of tests to determine the time to crack or craze at different stress levels.
- b) Plot the stress level versus the time to crack or craze. An L-shaped curve shall be obtained.
- c) Draw straight lines through the points that make up the vertical and horizontal legs of the L. The intersection of the lines will determine the critical stress level for that particular solvent/material combination.
- d) Repeat the procedure for each different solvent/material combination.

5.3.6.2 Report

The report shall contain the following information as a minimum:

- a) name of the test material, supplier and manufacturer;
- b) name of the test chemical;
- c) test temperature, pressure, duration, and sample thickness (at the exposed area) before and after the test;
- d) length, thickness and width of the overall test sample;
- e) critical stress level as obtained from the plot.

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Annex A
(informative)
Examples of forms

Table 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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