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**Space systems — Surface cleanliness of  
fluid systems —**

Part 3:

**Analytical procedures for the  
determination of nonvolatile residues and  
particulate contamination**

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*Systèmes spatiaux — Propreté des surfaces en contact avec des  
fluides —*

*Partie 3: Modes opératoires analytiques pour la détermination des  
résidus non volatils et de la contamination particulaire*

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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.

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 14952-3 was prepared by Technical Committee ISO/TC 20, *Aircraft and space vehicles*, Subcommittee SC 14, *Space systems and operations*.

ISO 14952 consists of the following parts, under the general title *Space systems — Surface cleanliness of fluid systems*:

- *Part 1: Vocabulary*
- *Part 2: Cleanliness levels*
- *Part 3: Analytical procedures for the determination of nonvolatile residues and particulate contamination*
- *Part 4: Rough-cleaning processes*
- *Part 5: Drying processes*
- *Part 6: Precision-cleaning processes*

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## Introduction

This part of ISO 14952 defines sampling and analytical methods to verify cleanliness levels for parts and components used in space fluid systems. It can be used to determine the cleanliness level of precision-cleaned parts and components used in ground support equipment, launch vehicles and spacecraft.

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# Space systems — Surface cleanliness of fluid systems —

## Part 3:

# Analytical procedures for the determination of nonvolatile residues and particulate contamination

## 1 Scope

This part of ISO 14952 provides sampling and analytical test methods to validate the cleanliness levels of parts and components that have been precision cleaned, and identifies processes that may be used for the verification of cleanliness in ground support equipment, launch vehicles and spacecraft. This part of ISO 14952 is used to sample and verify the level of cleanliness of parts and components that have been precision cleaned and applies equally to parts, components and systems in ground support equipment, launch vehicles and spacecraft.

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## 2 Normative references

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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 2210:1972, *Liquid halogenated hydrocarbons for industrial use — Determination of residue on evaporation*

ISO 5789:1979, *Fluorinated hydrocarbons for industrial use — Determination of non-volatile residue*

ISO 5884:1987, *Aerospace — Fluid systems and components, Methods for system sampling and measuring the solid particle contamination of hydraulic fluids*

ISO 14951-3:1999, *Space systems — Fluids characteristics — Part 3: Nitrogen*

ISO 14951-4:1999, *Space systems — Fluids characteristics — Part 4: Helium*

ISO 14951-10:1999, *Space systems — Fluids characteristics — Part 10: Water*

ISO 14952-1:2003, *Space systems — Surface cleanliness of fluid systems — Part 1: Vocabulary*

ISO 14952-5:2003, *Space systems — Surface cleanliness of fluid systems — Part 5: Drying processes*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 14952-1 apply.

## 4 Acceptance inspection

### 4.1 General

Unless otherwise specified by the customer, acceptance inspection shall be performed as specified in 4.2 to 4.7.

### 4.2 Visual inspection

**4.2.1** The surfaces of all items that will contact the service medium shall be visually inspected for the presence of moisture, corrosion, scale, dirt, grease and other foreign matter. An external light source or borescope may be required to examine internal surfaces.

**4.2.2** Items having limited accessibility for visual inspection shall be accepted or rejected on the basis of the quality assurance inspections of 4.3, 4.4 and 4.5. The presence of visible contamination that discloses a particle population greater than the level specified shall be cause for rejection. Discoloration of a surface due to welding and passivation shall be permitted provided no weld scale or other contaminants remain.

### 4.3 Acidity and alkalinity test

All surfaces that have been cleaned shall be tested for acidity and alkalinity with pH paper while the surfaces are wet from the final water rinse. Dry surfaces of completed items shall be wetted with a few drops of high purity water, with a pH range of 5,0 to 8,0, meeting the requirements of ISO 14951-10, to permit testing as required. When tested, the pH shall range from 5,0 to 8,0.

### 4.4 Acceptance inspection of items cleaned in a controlled environment

#### 4.4.1 General

Items cleaned in a controlled environment, except those processed to level visually clean (VC) and/or level visually clean and inspected with the aid of an ultraviolet (UV) light, shall be tested for conformance to the applicable cleanliness level by the test fluid-flush procedure given in 4.4.2 to 4.5.4.

#### 4.4.2 Test fluids

The test fluids shall not react with, combine with, etch, or otherwise cause immediate or latent degradation of the item being tested, and shall be selected from those specified in Table 1, unless otherwise approved by the customer. The test fluid shall meet the following requirements.

- a) The test fluid shall be filtered to remove particulates greater than 1 µm and shall not exceed 10 mg/l of nonvolatile residue (NVR) (see ISO 5789). For particle analysis where NVR analysis is not required, the maximum allowable NVR level of the test solvent shall not exceed 50 mg/l.
- b) Isopropanol and ethanol shall not be used as the test fluid for oxidizer systems and hardware unless the softgoods are removed or are applied with a suitable pretreatment process. Softgoods for oxidizer systems should be cleaned with a waterbased process and blown dry with Type A nitrogen conforming to ISO 14951-3. If the softgoods are cleaned with isopropanol or ethanol purge, the softgoods shall be purged with Type A nitrogen conforming to ISO 14951-3, until the methane hydrocarbon equivalent of the effluent gas does not exceed that of the source gas. Alternatively, the softgoods may be vacuum dried as described in ISO 14952-5:2003, 5.2.
- c) Subtraction of the test fluid blank particle count from the test sample particle count shall not be allowed.
- d) The quality of the test fluids shall be verified at least once a day prior to use.
- e) Some test fluids may have low threshold limit values; therefore, chemical hygiene tables should be consulted prior to use.



- f) The NVR of the flushing solvent should be less than or equal to the NVR of the cleanliness level being verified.
- g) The test fluids shall be compatible with the fluid used in the system or components being tested.

**WARNING — Halogenated solvents shall not be used on titanium alloys.**

#### 4.4.3 Test fluid volume for analysis

The test fluid volume required for analysis shall be dependent upon the analytical method employed. The standard test sample shall be 500 ml of test fluid to ensure that all critical surfaces are flushed. The 500-ml sample of test fluid shall represent a minimum surface area of 0,1 m<sup>2</sup> to a maximum of 0,5 m<sup>2</sup>. For very large items, the volume of flushing solvent should correlate to a minimum sampling area. In cases where all critical surfaces can be sampled with 100 ml of test fluid and the analytical method requires 100 ml or less of test fluid, a 100 ml sample of test fluid shall be allowed to represent 0,1 m<sup>2</sup> of critical surface area, if approved by the customer.

**Table 1 — Solvents, maximum allowable nonvolatile residue**

Solvent	Maximum allowable NVR	
	Test fluid	
	General use mg/l	Oxidizer mg/l
Perfluoro- <i>n</i> -butyl methyl ether (C <sub>4</sub> F <sub>9</sub> OCH <sub>3</sub> )	10	10
Tetrachloroethylene <sup>a</sup>	10	10
Perfluoro- <i>n</i> -butyl methyl ether (20 % to 80 % by mass) and perfluoroisobutyl methyl ether (20 % to 80 % by mass)	10	10
Isopropanol	10	not allowed
Ethanol	10	not allowed
Azeotrope of HFC 43-10 MEE (62 % by mass ) and <i>trans</i> -1,2-dichloroethylene (38 % by mass) <sup>a</sup>	10	10
HCFC - 225 ca/cb <sup>a</sup>	10	10
HCFC-141b <sup>a</sup>	10	10
HFC 43-10 MEE <sup>a</sup>	10	10
HFE 7100	10	10
HFE 71DE	10	10

<sup>a</sup> These solvents have low threshold limit values and may pose a hazard in controlled areas or cleanrooms.

#### 4.4.4 Test fluid-flush procedure (solvent)

The fluid-flush procedure shall be as follows.

- a) Ascertain the test procedure and total volume of test fluid necessary to flush the cleaned item or items in accordance with Method I (see 5.1.1).
- b) Flush all critical surfaces uniformly with the test fluid. Tubing, piping and hoses shall be flushed in accordance with either Method I or Method II (see 5.1.1 and 5.1.2). Where flushing does not reach all interior surfaces, the test fluid shall be introduced and the item shall be manually shaken or rolled until all interior surfaces are wetted. Large, difficult-to-flush items may be positioned so that the vessel can be filled from the bottom and overflowed from the top.

- c) Catch the test fluid in a precision-cleaned container.
- d) Immediately upon the completion of step c), dry the tested items in accordance with the applicable drying procedure.

Some analytical methods specify other test procedures (see Clause 5).

#### 4.4.5 Analysis of test fluid-flush sample (solvent)

##### 4.4.5.1 General

When a solvent is used as the test fluid, the test sample shall be analysed for particle population and NVR by the following recognized analytical methods. Other analytical methods may be used which have demonstrated accuracy and repeatability, provided that their use is approved by the customer.

##### 4.4.5.2 Particle population analysis (solvent-flush)

The solvent-flush sample shall be analysed for particle population as follows.

###### a) Microscopic particle population

The particle analysis shall be performed in accordance with 5.2.

###### b) Particle population analysis (automatic particle counters)

Automatic liquidborne particle counters may be used for final verification of cleanliness of the end product, provided the individual counters have demonstrated accuracy and repeatability, which correlates with accepted analytical methods, and provided that their use is approved by the customer.

##### 4.4.5.3 NVR analysis (solvent-flush)

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###### 4.4.5.3.1 General

The solvent-flush samples that have been filtered in accordance with 4.4.5.2 shall be analysed for NVR by one of the following methods.

###### 4.4.5.3.2 Gravimetric NVR analysis method

The filtered solvent sample shall be evaporated to determine the NVR content in accordance with 5.3.

###### 4.4.5.3.3 Solvent purity meter

A solvent purity meter may be used for final verification of cleanliness of the end product, provided the individual meter has demonstrated accuracy and repeatability, which correlates with accepted analytical methods, and provided that its use is approved by the customer.

###### 4.4.5.3.4 Infrared spectrometric NVR analysis method

Infrared (IR) spectrometric NVR analysis of solvent samples may be used if the following apply:

- a) the method quantifies hydrocarbons and other contaminants which are reactive with liquid oxygen or hypergolic fluids;
- b) the analysis method has demonstrated accuracy and repeatability and the method is approved by the customer.

#### 4.4.5.3.5 Mass spectroscopy (MS) NVR analysis method

MS NVR analysis of solvent samples may be used if the following apply:

- a) the method quantifies hydrocarbons and other contaminants which are reactive with liquid oxygen or hypergolic fluids;
- b) the analysis method has demonstrated accuracy and repeatability and the method is approved by the customer.

#### 4.4.5.3.6 Gas chromatography/mass spectroscopy NVR analysis method

Gas chromatography/mass spectroscopy (GC/MS) NVR analysis of solvent samples may be used if the following apply:

- a) the method quantifies hydrocarbons and other contaminants which are reactive with liquid oxygen or hypergolic fluids;
- b) the analysis method has demonstrated accuracy and repeatability and the method is approved by the customer.

#### 4.4.6 Analysis of aqueous-based, fluid-flush sample

The aqueous-based, fluid-flush samples shall be analysed for particle population and NVR as follows.

- a) Particle population analysis (aqueous)

The particle analyses of 4.4.5.2 may be used for final verification of cleanliness of the end product, provided the sampling and analysis methods have demonstrated accuracy and repeatability, which correlate with accepted analytical methods, and provided that their use is approved by the customer.

- b) NVR analysis (aqueous)

Aqueous NVR sampling and analysis methods may be used for the final verification of cleanliness of the end product, provided the methods have demonstrated accuracy and repeatability (which correlate with accepted analytical methods) and provided that their use is approved by the customer. An accepted method is given in 5.4.

#### 4.4.7 Drying

##### 4.4.7.1 General

After testing for particle population and NVR, all components and parts shall be thoroughly dried to remove residual cleaning, rinsing, and/or verification media.

##### 4.4.7.2 Purge drying

All components and parts rinsed shall be dried by a purge of gaseous nitrogen, filtered to remove particulates greater than 1  $\mu\text{m}$  (in accordance with ISO 14951-3, Type A), or helium, filtered to remove particulates greater than 1  $\mu\text{m}$  (in accordance with ISO 14951-4, Type 1, Grade A). The critical internal surfaces of small vessels, hoses and tube assemblies shall also be purge dried. If the critical internal surfaces cannot be inspected visually, perform analyses in accordance with 5.1.3. All items rinsed with reagent water which cannot be visually inspected (100 %) shall be tested by Method III (see 5.1.3) for surface moisture. Component parts may be dried with heated air dryers equipped with HEPA filters. Recommended purge drying practices are discussed in ISO 14952-5.