INTERNATIONAL STANDARD

ISO 14952-3

First edition
2003-11-15

Space systems — Surface cleanliness of fluid systems —

Part 3:
Analytical procedures for the determination of nonvolatile residues and particulate contamination

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

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

Reference number
ISO 14952-3:2003(E)

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Contents

Page

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*

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.

INTERNATIONAL STANDARD **ISO 14952-3:2003(E)**

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.

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 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 isoproanol 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^2 to a maximum of 0,5 m^2. 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^2 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-*n*-butyl methyl ether ($C_4F_9OCH_3$)	10	10
Tetrachloroethylene[a]	10	10
Perfluoro-*n*-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 *trans*-1,2-dichloroethylene (38 % by mass)[a]	10	10
HCFC - 225 ca/cb[a]	10	10
HCFC-141b[a]	10	10
HFC 43-10 MEE[a]	10	10
HFE 7100	10	10
HFE 71DE	10	10

[a] 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)

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 µm (in accordance with ISO 14951-3, Type A), or helium, filtered to remove particulates greater than 1 µm (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.

4.4.7.3 Vacuum drying

Intricate parts with features (such as wire mesh filter elements and fine threaded holes) shall be placed in a clean vacuum oven, purged with test gas, heated, and then evacuated until dry. Recommended vacuum drying practices are discussed in ISO 14952-5.

4.5 Acceptance inspection of items cleaned in the field

4.5.1 General

Test items such as tubing, piping and vessels cleaned in the field shall be tested for conformance to the applicable cleanliness level using Method I, Method II or Method V (as described in 5.1.1, 5.1.2 and 5.1.5). Testing procedures are determined by the configuration of the item being cleaned and by the method of dispensing the test fluid.

4.5.2 Test fluids

The test fluids shall be in accordance with 4.4.2.

WARNING — The compatibility of nonmetallic materials with the applicable test fluid shall be determined prior to testing. Permission to remove nonmetallic materials prior to testing shall be granted at the discretion of the customer.

4.5.3 Ratio of testing fluid volume to critical surface area

All critical surface areas shall be sampled with test fluid unless specified otherwise. The 500-ml sample of test fluid shall be representative of the total critical surface area; however, for analytical purposes, the 500-ml sample shall represent a minimum surface area of 0,1 m^2 to a maximum of 0,5 m^2.

4.5.4 Particle population analysis

Each sample shall be tested for particle population in accordance with the procedure of 4.4.5.2. The particle population per 0,1 m^2 shall be determined on the basis of the proportional critical surface area.

4.5.5 NVR analysis

The NVR shall be determined in accordance with 4.4.6 b). NVR per 0,1 m^2 shall be determined on the basis of the proportional critical surface area.

4.5.6 Drying

All items cleaned in the field shall be dried in accordance with 4.4.7.

4.5.7 Maintaining cleanliness

4.5.7.1 General

The cleaning contractor is responsible for maintaining the cleanliness of items and systems cleaned in the field until it is received or accepted by the customer.

4.5.7.2 Test gas purge

Items such as vessels, pipe and tubing systems, and pipe, tubing and flex hose assemblies shall be maintained under the test gas purge of 13,8 kPa to 34,5 kPa until all ports, orifices and fittings are sealed. The test gas shall be nitrogen, filtered to remove particulates greater than 5 µm (in accordance with ISO 14951-3, Type A), or helium, filtered to remove particulates greater than 5 µm (in accordance with ISO 14951-4, Type 1, Grade A).

4.5.7.3 Temporary hardware replacement

Temporary hardware installed in subsystems, systems, and related field equipment for cleaning shall be replaced with clean functional components after the subsystem, system or related field equipment has been verified clean. Procedures and practices shall be established to maintain system cleanliness. Adjacent, external system and structural surfaces shall be cleaned to level GC prior to replacement. Where practical, the hardware replacement shall be performed in a controlled environment, which can be provided by a portable cleanroom (tent) or similar structure.

4.5.7.4 Component replacement

Replacement of functional components in clean systems shall be in accordance with 4.5.7.3.

4.6 Acceptance inspection of packaging materials

4.6.1 Environmental control

All quality assurance operations shall be accomplished within a cleanroom that is consistent with or cleaner than the packaging material being inspected. Care shall be taken not to contaminate the packaging materials, which should be stored in an area with proper cleanliness ratings.

4.6.2 Sampling

Packaging materials shall be examined and tested to determine compliance with the cleanliness requirements of 4.6.1. All the plastic film of one type, one size and one configuration, such as tubing, flat roll stock, sheet and fabricated bags offered by one manufacturer at one time, shall be considered to be one lot.

4.6.3 Visual inspection

No evidence of oil, solvents, paints, grease, dirt, soap, ink, metal chips or other foreign matter shall be permitted on either the external surfaces or the internal surfaces of packaging materials when inspection is made with the unaided eye.

4.6.4 Thickness of packaging film

The thickness of plastic films used for precision packaging shall conform to the limits specified in Table 2. Thickness measurements shall be made with a micrometre caliper having a flat anvil and capable of being read to the nearest 2,5 µm.

WARNING — Handling plastic films under low-humidity conditions (i.e. less than 40 %) may result in the buildup of static charges on the film.

Table 2 — Packaging materials thickness and service requirements

Plastic film	Thickness range µm	Use
Polyethylene	100 to 170	Overwrap, except may be used for inner wrap of items cleaned for non-oxygen service to level VC
Nylon 6 or equivalent polyamide	40 to 60	Precision packaging, not for liquid and gaseous oxygen and hypergol service
Polychlorotrifluoroethylene	40 to 60	Precision packaging, suitable for liquid and gaseous oxygen and hypergol service
Polyfluoroethylenepropylene	10 to 500	Precision packaging, suitable for liquid and gaseous oxygen and hypergol service

4.6.5 Verification of cleanliness level

4.6.5.1 General

All plastic films of one lot shall have the cleanliness level verified prior to use.

4.6.5.2 Minimum critical surface area for test

The minimum interior critical surface area for verification of cleanliness level shall be 0,1 m^2. Sampling shall be according to 4.6.2, except that additional sample material from the offered lot shall be used when necessary to make 0,1 m^2.

4.6.5.3 Sample preparation

Fabricated bags shall be sealed across the open end. Tubular packaging material shall be fabricated into a bag by cutting off, with properly cleaned tools, a length conforming to the requirements of 4.6.5.2 and sealing both ends. Flat roll sheet and stock shall be fabricated into a bag by cutting out a section with an area conforming to the requirements of 4.6.5.2, folding the section and sealing the section as necessary. The cutting, purging and sealing techniques shall be as follows.

a) Cutting

When clean plastic film is to be cut, stainless steel, chrome-plated or nickel-plated scissors shall be used. The scissors shall be carefully pushed through the film. Avoid sawing and hacking actions, resulting from opening and closing the scissors, to prevent the generation of particles. Razor blades or other single-blade-type instruments shall not be used to cut plastic film.

b) Purging

Prior to final sealing of the plastic film bag containing the clean component, the plastic film bag shall be purged with filtered gaseous nitrogen, filtered to remove particulates greater than 5 µm (in accordance with ISO 14951-3, Type A).

c) Sealing

An all-purpose impulse sealer shall be used to produce effective seals with plastic films. If specific sealing procedures are not available, the recommendations of the manufacturer shall be followed for temperature setting and dwell time. Polychlorofluorocarbon shall be sealed on all sides when fabricating bags and shall not be centrefolded. Centrefolding may generate particles since fluorohalocarbon films tend to be brittle.

All items shall be handled in a manner that minimizes exposure of the interior critical surfaces to airborne particles. One corner of the completely sealed test bag shall be cut off so that an opening of a maximum of 19 mm in length is created.

4.6.6 Rinsing procedures

Fluids that conform to the cleanliness level of 4.4.2 shall be used as the test fluid in the ratio of 100 ml of fluid per 0,1 m^2 of critical surface area. The following rinsing procedure shall be used:

a) introduce test fluid into the sealed bag through the previously cut opening;

b) close the bag by folding over the cut corner;

c) gently agitate the test fluid within the bag for a minimum of 15 s, wetting all surfaces;

d) pour the used test fluid into a precision-cleaned beaker, taking care to exclude airborne contamination;

e) analyse the test fluid for particulate population and NVR in conformance with 4.4.5.

4.7 Acceptance inspection of items by functional test

4.7.1 General

Functional items, such as valves, regulators, cylinders, flexhoses, tube assemblies, vessels and installed systems, shall be functionally tested as specified in 4.7.2 to 4.7.4 after final cleaning and assembly.

4.7.2 Calibrated instrumentation

Test parameters, such as pressure, temperature, time, voltage, current and resistance, shall be monitored using calibrated instrumentation capable of measuring the specified parameters.

4.7.3 Hydrostatic testing

Functional items, such as flexhoses, tube assemblies, vessels and systems that require hydrostatic test, shall be tested prior to the final or precision-cleaning operation using an appropriate test fluid at the specified temperature and pressure.

4.7.4 Component testing

Functional components, such as valves, solenoid valves, regulators, actuators and cylinders, shall be assembled using clean parts and shall be then functionally tested with gaseous nitrogen, filtered to remove particulates greater than 5 µm (conforming to ISO 14951-3, Type A), unless otherwise specified. The component shall be tested under the specified conditions.

5 Sampling and analytical practices

5.1 Cleanliness level test methods

5.1.1 Method I

The liquid flush test for particle population and NVR remaining on critical surfaces of items cleaned in a controlled environment shall be performed in the following manner.

a) All items, except those processed to level VC, level UV, and/or rough clean requirements, shall be sampled.

b) Parts having a surface area greater than 0,5 m^2 shall be flushed with 100 ml (maximum) per 0,1 m^2 of critical surface area. However, the test sample volume shall be 500 ml and any excess shall be discarded.

For parts having an area less than 0,5 m^2 but greater than 0,1 m^2, a 500-ml sample is considered representative of the actual surface area of the part being sampled.

c) Small components, fittings, soft goods, etc. (items small enough to fit inside an 800-ml beaker), shall be individually dipped and agitated in 500 ml of test fluid. Small components should be combined into batches having a total surface area of at least 0,1 m^2.

d) Critical areas of large components, flanges, valves, etc. (items too large to dip), shall be flushed and sampled.

e) Individual components, such as drums, having an area greater than 0,5 m^2 shall be flushed with 100 ml (maximum) of test fluid for each 0,1 m^2 of critical surface area. The test fluid shall be collected in or transferred to a single container, agitated then sampled to obtain 500 ml (total sample) from the top, centre and bottom of the original test fluid sample for analysis. Discard excess test fluid (sample is representative of 0,5 m^2).

f) Small containers with less than 0,5 m^2 internal surface area, such as cylinders, dewars, etc., shall be sampled by using 500 ml of test fluid. Agitate the container to cover all critical surfaces with sample medium, then drain the test fluid from the container for analysis.

5.1.2 Method II

Liquid flow test for monitoring particle population and NVR remaining on critical surfaces of items cleaned in the field shall be performed in the following manner.

a) Flow test fluid in accordance with 4.4.2 through the item at a minimum average velocity of 1,25 m/s.

b) Catch a test fluid sample in a precision-cleaned container.

5.1.3 Method III

The gas flow test for moisture remaining on a critical surface after cleaning shall be performed in the following manner.

a) The cleaned item shall be purged with test gas. The test gas shall be nitrogen, filtered to remove particulates greater than 5 µm (conforming to ISO 14951-3, Type A).

b) The item shall be purged for a sufficient time to ensure all of the residual air and vapour have been expelled from the item. Hold (stop) the purge flow for sufficient time to allow entrapped moisture to diffuse or permeate into the static purge gas. Begin monitoring for moisture in the purge gas at the item's discharge port. Resume purge gas flow.

c) The dewpoint of the effluent test gas shall be monitored by a suitable instrument. The method or instrument used in the dewpoint method shall be capable of detecting to a level of less than 20 µl/l of moisture. The manufacturer's directions shall be followed for the operation of a specific instrument. The effluent gas moisture content shall be 24 µl/l (dewpoint = – 55 °C) for individual components, or 128 µl/l (dewpoint = – 40 °C) for systems, unless specified otherwise. The dewpoint of the gas at the outlet should not exceed that at the inlet.

d) If the effluent gas moisture content exceeds the allowable limit, continue purging the item periodically repeating steps b) and c) until the test requirement is met.

5.1.4 Method IV

The liquid flow test to evaluate a system's capability to deliver fluid that meets specified requirements shall be performed in the following manner.

a) Sampling of the system shall be performed at the system's designed point of delivery under normal system operating conditions. Fluid samples shall be drawn under the system's design operating conditions from the flowing stream, not from a dead space in the system.

b) If the fluid is a hazardous material or liquid, route the fluid discharge to the fluid deservicing or return system.

c) The following fluid sample size is recommended:

- liquid system: 500 ml to 1,0 l.

5.1.5 Method V

The gas flow test method for monitoring particle population and NVR remaining on critical surfaces of items normally cleaned in the field shall be performed as follows.

a) Sampling of the component, subsystem, or system shall be performed at the specified point under the following conditions and the sampling gas shall be drawn through an absolute 1-µm filter membrane for 2 min:

Item	Operating pressure	Sampling pressure	Sampling gas	Remarks
GHe, GH$_2$ LH$_2$ system	$\geqslant$ 1 380 kPa	1 380 kPa	GHe or GN$_2$	See ISO 14951-4 or ISO 14951-3
	$<$ 1 380 kPa	Operating pressure		
Other	$\geqslant$ 1 380 kPa	1 380 kPa	GN$_2$	See ISO 14951-3
	$<$ 1 380 kPa	Operating pressure		

b) The particles on the filter membrane shall be counted using procedures described in 5.2.

c) An NVR sample may also be obtained by passing the sampling gas through a precision-cleaned collection tube under the following conditions:

Item	Sampling gas	Flow rate	Duration	Sampling volume
GHe, GH$_2$ LH$_2$ system	GHe	100 l/min	35 min	3 500 l
	GN$_2$	100 l/min	5 min	500 l
Other	GN$_2$	100 l/min	5 min	500 l

d) Extract the deposited NVR sample from the sample tube using an approved solvent (see Table 1).

e) Determine the NVR content of the solvent using the gravimetric procedure as described in 5.3 or, if suitable, the infrared spectrometer procedure as directed in 4.4.5.3.4.

5.1.6 Method VI

The flow test method to evaluate the cleanliness of a pipeline and associated parts after cleaning as described in ISO 14952-6:—, 4.11, shall be performed as follows.

a) Cover a slug (mole or tampon) having a diameter slightly smaller than that of the pipeline with an absorbent material wet with a suitable solvent chosen from Table 1. Pressurize the pipeline with nitrogen, filtered to remove particulates greater than 5 µm (conforming to ISO 14951-3, Type A), to force the slug through the pipeline. Catch the slug at the end of the pipeline and visually examine the cover for evidence of contamination.

b) Repeat the sampling process with a clean, damp cover each time until the cover shows no visual sign of contamination.

c) Purge the pipeline as described in 5.1.3, Method III.

5.2 Microscopic particle population

Proceed as follows.

a) Assemble a precision-cleaned filtration apparatus.

b) Using clean forceps with non-serrated tips, place a test fluid compatible filter membrane (47-mm diameter with 0,4-µm to 1,0-µm pores) in position in the filter holder. Prior to insertion, the filter membrane may be rinsed with filtered test fluid to remove any adherent contamination.

c) Fill the filter funnel approximately three-quarters full of test fluid and turn on the vacuum pump.

d) Add the remaining test fluid to the filter funnel at a rate necessary to maintain the funnel more than half full until all of the test fluid has been added. Do not allow the test fluid to pour directly onto the filter membrane after filtration has started.

e) When filtration is completed, remove the filter membrane from the holder and place it in a disposable Petri dish, or equivalent, until the particles are counted.

f) Retain the filtrate for analysis of the NVR in accordance with 4.4.5.2.

g) Place the filter membrane under the microscope.

h) Direct a high-intensity light source of 5 000 cd to 6 000 cd into the filter membrane from an oblique position to obtain maximum definition for sizing and counting. High-intensity illumination is a critical requirement.

i) Use a magnification of approximately 40 to 50 power for counting particles for conformance to level 150 and greater, and approximately 100 power for level 100 and less.

j) The particles may be counted using procedures described in ISO 5884 except that when the total number of particles of a given particle size range is to be between 1 and 154, the number of particles over the entire effective filtering area of the membrane shall be counted.

5.3 Gravimetric NVR analysis method

Perform the gravimetric NVR analysis in accordance with ISO 2210 or a similar method that will allow the evaporated test fluid to be recovered and recycled. If the test fluid used is perchlorethylene, a silicone-based oil bath shall be used with the rotary evaporator because to the high boiling point of perchlorethylene. The gravimetric NVR analysis method shall be performed as follows.

a) Degrease an evaporation flask by washing it three times with alcohol and three times with the test fluid. Transfer the 500 ml of filtrate described in 5.2 into the clean, degreased flask.

b) Evaporate the 500-ml sample to 10 ml to 20 ml.

c) After cooling, transfer the sample to a clean, constant mass (within 0,1 mg), tared weighing dish. Wash the flask three times with a total volume of 5 ml of clean, filtered fluid and transfer the wash fluid to the weighing dish.

d) Continue evaporation by placing the weighing dish inside a constant-temperature oven at 105 °C to 110 °C. Allow the weighing dish to remain inside the oven until the fluid has just evaporated to dryness. A thermostatically controlled hot plate may be substituted for the oven.

e) Remove the weighing dish from the oven and place in a desiccator to cool for 30 min.

f) After cooling, remove the weighing dish from the desiccator, weigh the dish to the nearest 0,1 mg, and record the mass.

g) Carry out a blank determination on the filtered fluid, and subtract the results from the NVR value obtained for the sample.

5.4 Aqueous ultrasonic sampling and total carbon (TC) NVR analysis

5.4.1 General

This procedure defines the method of performing aqueous ultrasonic sampling and total carbon (TC) NVR analysis of small parts.

5.4.2 Apparatus

5.4.2.1 Ultrasonic bath, 15 W/l to 25 W/l (25 kHz to 27 kHz).

5.4.2.2 Sampling pans for parts, made of stainless steel, of volumes 1 l, 2 l, 3 l and 4 l.

5.4.2.3 Bracket, to suspend parts sampling pan in ultrasonic bath.

5.4.3.4 Total carbon analyser (TCA).

An analyser with a high-temperature (minimum 880 °C) combustion chamber with a sensitivity of ± 0,2 µg per gram of carbon (C) (milligrams of C per litre) and direct sample injection into combustion furnace has been found to be satisfactory.

NOTE Tests have shown that fluorinated materials, such as the perfluoroether lubricants, require a temperature of at least 850 ^{o}C to decompose on a typical catalytic bed used in combustion analysers.

5.4.2.5 Syringe (TCA sample), of capacity 200 µl or variable 500 µl.

5.4.3 Procedure

5.4.3.1 Preliminary steps

Proceed as follows.

a) Set the ultrasonic bath temperature at 70 °C ± 5 °C and de-gas the bath for 10 min before use.

b) Set the TCA to the optimum parameters with the furnace temperature at least 880 °C. Calibrate the TCA in accordance with the manufacturer's instructions.

c) Clean the sampling pans. Conduct the sampling procedure without parts to verify the cleanliness of the pans. The TCA results should be less than 1,0 µg/g. If the total carbon reading is greater than 1,0 µg/g, check the quality of the reagent water and/or the cleanliness of the parts sampling pan.

d) Record the TCA results on the sampling pan as blank sample (C_B).

5.4.3.2 Sampling and determination

Proceed as follows.

a) Place the parts with a surface area of 0,1 m^2 to 0,2 m^2 in a clean sampling pan.

b) Measure the quantity of reagent water (conforming to ISO 14951-10, Type HP) required to cover the parts in the sampling pan.

c) Cover the sampling pan with aluminium foil or a stainless-steel cover, and place it on a bracket in the ultrasonic bath.

d) The ratio reagent water to parts surface area shall not exceed 1 000 ml per 0,1 m^2; the ideal ratio is 500 ml per 0,1 m^2.

e) Set the level of water in the ultrasonic bath so it is above the water level in the sampling pan.

f) Sonicate parts in the ultrasonic bath for 10 min. Perform steps f), g) and h) as quickly as possible within the maximum time limit of 120 min.

g) Remove the parts sampling pan from the ultrasonic bath and remove the cover. Swirl the parts sampling pan to mix the water.

h) Draw a 200-µl sample of water from the parts sampling pan with a syringe.

i) Inject the 200-µl sample of water into the TCA following the instrument operating instructions and record the TCA results.

j) Record the sample total carbon reading (C_S).

5.4.4 Calculation

Calculate the equivalent nonvolatile residue (aqueous ultrasonic sampling) as follows:

$$\mathrm{NVR} = (C_\mathrm{S} - C_\mathrm{B})\, V_\mathrm{W} / F \cdot A$$

where

NVR is the equivalent NVR (mg/m^2 or mg/0,1 m^2);

C_S is the total carbon value of sample (mg of C per litre);

C_B is the total carbon value of blank (mg of C per litre);

V_W is the volume of water (litre);

A is the surface area of parts (m^2);

F is the sensitivity factor (mg of C per mg of contaminant); this is an empirical constant derived from test of known contaminants.

See Annex A for the determination of the sensitivity factor (F).

Annex A
(informative)

Sensitivity factor calculation

A.1 For samples taken from parts for the purpose of cleanliness verification, an NVR value may be calculated from the TC value. In order to calculate the NVR, a sensitivity factor must be determined. This requires some knowledge of the composition of the contaminant. For use of this technique in a manufacturing facility, the problem is easily resolved because the manufacturer knows the identity of the materials used in the processes; i.e. cutting oils, adhesives, solder flux, etc. For an independent cleaning facility, the problem becomes much more difficult.

NOTE The contaminant may be a single compound or a mixture of several compounds. The majority of materials used in processes are mixtures and the actual contaminant should be used to determine the SF.

A.2 To determine the sensitivity factor based on the mass of a known contaminant, disperse 1,0 mg of the contaminant in 1,0 l of water. Perform the carbon analysis 20 times and average the results. The sensitivity factor may then be derived from

$$F = T_{\mathrm{C}}/S$$

where

F is the sensitivity factor (mg of C per mg of contaminant);

T_{C} is the average total carbon value of the sample (mg of C per litre);

S is the contaminant solution concentration (mg/l)

EXAMPLE Assume that the contaminant is methanol and the solution concentration is 1 mg/l. The molecular mass of methanol is 32 and contains 1 atom of carbon. Dividing the atomic mass of carbon (12) by 32 equals 0,375 mg of carbon per litre. Substituting into the formula gives 0,375 mg/l/1 mg/l, which gives F = 0,375.

Many contaminants are not soluble in water. Heating the water and ultrasonic agitation may be required to adequately emulsify the contaminant.

A.3 Some contaminants are very difficult to emulsify directly. Some success has been achieved by applying a known amount of contaminant to a small, thin, lightweight, preweighed coupon such as shim stock. The coupon is then ultrasonically agitated in a known amount of heated water. The coupon is dried and reweighed. The difference in coupon mass is the amount of contaminant extracted into the water. The water sample is analysed for TC and an sensitivity factor can then be calculated based on the known contaminant concentration and the measured TC value.

A.4 If a sample of the known contaminant is not available but the percentage of each component in the mixture is known, determine the sensitivity factor for each component in the mixture as described in A.2. Then multiply the value of F determined for each component of the mixture by the mass fraction of that component in the mixture. Sum the values and record the sum as the value of F for the mixture.

A.5 Another method to determine the sensitivity factor of a mixture with unknown composition is to determine the value of F of each suspected component and then adjust the value of F for that component by estimating the probability of that component being in the mixture. Then sum the probability adjusted sensitivity factors for each component and record the sum as the value of F for the mixture.

ICS 49.080; 49.140

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